APPENDIX C
FIELD INSTRUMENT MANUALS AND METHODS

Manual Change Notice
2100 Series Laboratory Turbidimeters

Introduction

Hach Company is now supplying specially packaged StablCal® Sealed Vial sets with all 2100 Series turbidimeters. Because of this, the accessory set supplied with these instruments has been modified. This change affects the manual supplied with the instrument. Please refer to the following table and to the information that follows.

Turbidimeter Model and Part Numbers	Manual Part Number	Manual Sections Affected	Corresponding Sections in this Memo
2100N 47000-00 (115 V) and 47000-02 (230V)	47000-88	Standard Accessories (Section 1.2) Unpacking (Section 1.4.1) Calibration Check (Section 2.11) Calibration (Section 3.2) Replacement Parts and Accessories	*Section 1 Standard Accessories* *Section 2 Unpacking the Instrument* *Section 3 Calibration Check* *Section 6 Calibrating the Turbidimeter (Using StablCal® Sealed Vial Standards)* *Section 7 Replacement Parts and Accessories*
2100AN 47001-00 (115 V) and 47001-02 (230 V)	47001-88	Standard Accessories (Section 1.2) Unpacking (Section 1.4.1) Calibration Check (Section 2.3.9) Calibration (Section 3.2) Replacement Parts and Accessories	*Section 1 Standard Accessories* *Section 2 Unpacking the Instrument* *Section 3 Calibration Check* *Section 6 Calibrating the Turbidimeter (Using StablCal® Sealed Vial Standards)* *Section 7 Replacement Parts and Accessories*
2100N IS 47900-00 (115 V) and 47900-02 (230 V)	47900-18	Standard Accessories (Section 1.2) Unpacking (Section 1.4.1) Calibration Check (Section 2.3.10) Calibration (Section 3.2) Replacement Parts and Accessories	*Section 1 Standard Accessories* *Section 2 Unpacking the Instrument* *Section 3 Calibration Check* *Section 6 Calibrating the Turbidimeter (Using StablCal® Sealed Vial Standards)* *Section 7 Replacement Parts and Accessories*
2100AN IS 47901-00 (115 V) and 47901-02 (230 V)	47901-88	Standard Accessories (Section 1.2) Unpacking (Section 1.4.1) Calibration Check (Section 2.3.9) Calibration (Section 3.2) Replacement Parts and Accessories	*Section 1 Standard Accessories* *Section 2 Unpacking the Instrument* *Section 3 Calibration Check* *Section 6 Calibrating the Turbidimeter (Using StablCal® Sealed Vial Standards)* *Section 7 Replacement Parts and Accessories*

1. Standard Accessories

Accessory items supplied with the turbidimeter include six sample cells, a set of StablCal® Sealed Vial Primary Turbidity Standards, a power cord, silicone oil, sample cell oiling cloth, a dust cover, 2 rolls of printer paper (supplied with the 2100AN and 2100AN IS only) and an instrument manual.

2. Unpacking the Instrument

Remove the instrument and accessories from their shipping box and inspect them for damage that may have occurred during shipment due to rough handling or extreme weather conditions.

Verify the following items are present:

- 2100 Series Laboratory Turbidimeter
- Instrument Manual with Quick Reference Guide
- A set of StablCal Primary Standards in sealed vials with instructions:
  For 2100N —Cat. No. 26621-05;
  For 2100AN—Cat. No. 26595-05;
  For 2100N IS—Cat. No. 26621-05;
  For 2100AN IS—Cat. No. 26595-05
- USEPA Filter Assembly (installed in sample cell compartment)
  (Cat. No. 30312-00)
- Oiling Cloth—Cat. No. 47076-00
- Six Sample Cells—Cat. No. 20849-00
- Silicone Oil, 15 mL (0.5 oz.) dropping bottle—Cat. No. 1269-36
- Power Cord—Cat. No. 18010-00 (115 V North American use)
  Cat. No. 46836-00 (230 V European use)
- Dust Cover—Cat. No. 47030-00
- 2 Rolls of Printer Paper—Cat. No. 47090-00 (2100AN and 2100AN IS only)
- 455 nm Filter Assembly—Cat. No. 19998-00 (2100AN only)

If any of the items are missing or damaged, please contact the Customer Service Department, Hach Company, Loveland, Colorado. Do not return the instrument without prior authorization. In the United States, the toll-free number is 1-800-227-4224. Outside the United States, contact your nearest Hach dealer.

3. Calibration Check

Quickly and easily verify the calibration of your 2100 Series Turbidimeter using the included StablCal Sealed Vials. Simply select the vial closest to the range being measured (do not use the <0.1 NTU vial—it does not have a precisely defined NTU value).

Prepare the vial as described in Section *5 Handling StablCal® Sealed Vial Standards*. Insert the vial in the cell holder and read the value. If the value is within ±10% of the stated vial value, the instrument calibration is valid for reporting purposes. If the reading is not within ±10%, re-calibrate the instrument.

4. Calibration

The electronic and optical design of 2100 Series turbidimeters provide long-term stability and minimize the need for frequent calibration. The multi-detector ratio optical system compensates for electronic and optical system variations between calibrations.

Hach recommends calibrating the instrument before it is used for the first time. When data is used for USEPA reporting, recalibrate at least every 90 days, or as stipulated by the regulating authority. Periodically, as experience or regulating authorities indicate, verify the instrument calibration using one of the StablCal® standards supplied with the instrument. If the reading in the range of use is not within 10% of the standard's assigned value, recalibrate the instrument.

Note: *For maximum accuracy and ease-of-use, your Hach turbidimeter is supplied with a StablCal Calibration set. The set contains prepared, stabilized formazin suspensions in specially sealed vials.*

Note: *The calibration is based on a first order linear equation consisting of up to four independent variables. Unpredictable results may occur if standards other than the recommended calibration points are used. The factory suggested calibration points have been determined by Hach Company chemists and engineers to provide the best calibration accuracy. Use of standards other than StablCal, or user-prepared formazin may result in less accurate calibrations.*

5. Handling StablCal® Sealed Vial Standards

Read these steps before handling the StablCal Standards.

Important Note: *Never shake or invert the < 0.1 NTU Standard. If the standard has been mixed or shaken, wait 15 minutes before using.*

If the standards have been used often (daily to weekly), begin with step 5.

If the standards have just arrived from the manufacturer or have been sitting undisturbed for longer than one week, begin with step 1.

1. Remove the < 0.1 NTU Standard from the plastic case and set it aside. Close the case lid.

2. Leave the remaining standards in the case and shake them for 2–3 minutes.

3. Let the standards stand undisturbed for 5 minutes.

4. Skip to Step 7, below.

5. Remove the < 0.1 NTU Standard from the plastic case and set it aside. Close the case lid.

6. Leave the remaining standards in the plastic case and invert the case 10 times.

7. Thoroughly clean, rinse, and dry the outside of the vials.

8. Immediately before using each standard, apply silicone oil (Cat. No. 1269-36) to the outside of the vial.

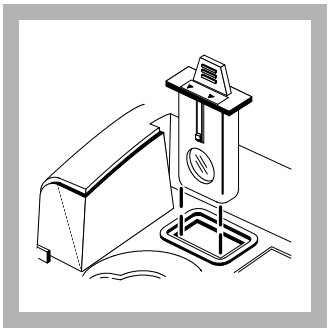
a. Apply a very thin bead of silicone oil from the top to bottom of the vial.

b. Spread the oil uniformly with the oiling cloth. Wipe off the excess so only a thin coat of oil remains. The vial should appear nearly dry with little or no visible oil.

Note: *Store the oiling cloth in a sealed plastic bag to keep it clean.*

9. Proceed with calibration.

6. Calibrating the Turbidimeter (Using StablCal® Sealed Vial Standards)



1. Insert the EPA filter module if measuring for EPA-reporting (2100N and 2100AN only).

Note: *Clean the filter before performing a primary calibration, or at least every 3 months (which is the USEPA-recommended calibration frequency).*

Note: *Clean the filter with glass cleaner, lens cleaner, or isopropyl alcohol, and a cotton-tipped swab.*

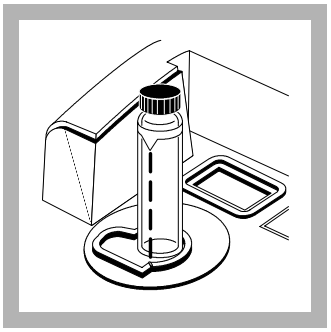
2. Press **CAL/Zero**.

The CAL mode annunciator lights, and the small green LED digits in the mode display flashes **00**. The NTU value of the dilution water used in the previous calibration is displayed.

Note: *Ratio on and Ratio off calibration data are measured and recorded simultaneously.*

Note: *Calibration for EBC and NEPH units of measure is set automatically via the NTU calibration.*

Note: *Upon entering the Calibration Mode, Automatic Range, Signal Averaging On, Ratio on and NTU units are automatically selected. Upon completion of calibration, all operational modes are restored to precalibration settings.*

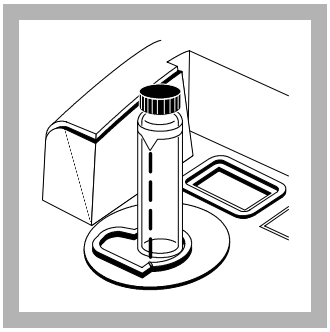


3. Select the StablCal vial labeled < 0.1 NTU. Wipe the cell clean and apply a thin film of silicone oil to its surface. Place it in the cell holder and close the cell cover. Press **ENTER**.

The instrument display counts down from **60** to **0**, and then takes a measurement. The instrument automatically increments to the next standard, the display shows 20.00 NTU, and the standard number **01** is shown in the mode display. Remove the < 0.1 NTU vial from the cell holder.

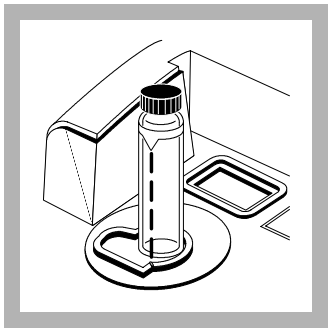
Note: *Install all StablCal vials with the orientation mark aligned with the cell holder reference mark.*

Note: *To exit the calibration procedure at any time without changing any stored value, press* **UNITS/Exit**.



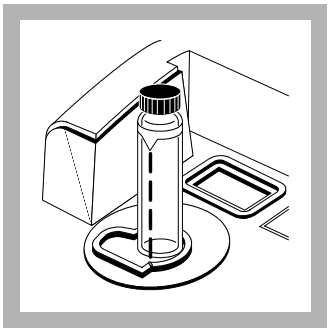
4. Select the StablCal vial labeled 20.00 NTU. Wipe the vial clean and apply a thin film of silicone oil to its surface. Place it in the cell holder and close the cell cover. Press **ENTER**.

Wait for the instrument to count down as before and display the next standard. Remove the vial from the cell holder.



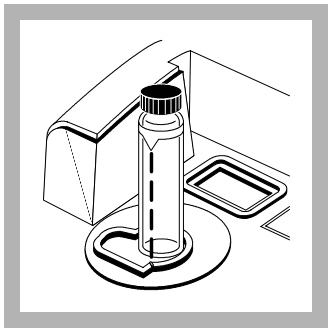
5. Select the StablCal vial labeled 200.0 NTU. Wipe the vial clean and apply a thin film of silicone oil to its surface. Place it in the cell holder and close the cell cover. Press **ENTER**.

Wait for the instrument to count down as before and display the next standard. Remove the vial from the cell holder.



6. Select the StablCal vial labeled 1000 NTU. Wipe the vial clean and apply a thin film of silicone oil to its surface. Place it in the cell holder and close the cell cover. Press **ENTER**.

Wait for the instrument to count down as before and display the next standard. Remove the vial from the cell holder.

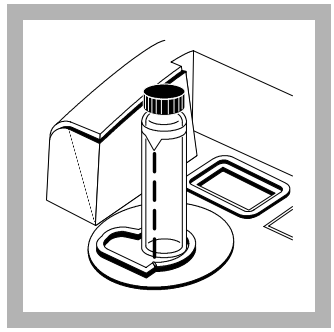


7. Select the StablCal vial labeled 4000 NTU. Wipe the vial clean and apply a thin film of silicone oil to its surface. Place it in the cell holder and close the cell cover. Press **ENTER**.

Wait for the instrument to count down as before and display the next standard. Remove the vial from the cell holder.

2100AN and **2100AN IS** instruments, complete step 8.

2100N and **2100N IS** instruments, skip to step 9.



2100 AN and 2100 AN IS instruments only:

8. Select the StablCal vial labeled 7500 NTU. Wipe the vial clean and apply a thin film of silicone oil to its surface. Place it in the cell holder and close the cell cover. Press **ENTER**.

9. Press **CAL/Zero**. The instrument makes calculations based on the new calibration data, stores the new calibration and returns to measurement mode.

Note: *If power is lost during calibration, new calibration data is lost, and the previous calibration remains in effect. To exit the calibration without saving new values, press* **UNITS/Exit**.

Note: *If* **ERR01** *or* **ERR02** *appears in the display, an error occurred during calibration. Clear the error message and proceed with measurements by pressing* **ENTER**. *The* **Cal?** *annunciator will be on, indicating a questionable calibration. Turn the* **Cal?** *annunciator off by recalibrating the instrument to remove erroneous data.*

7. Replacement Parts and Accessories

Refer to *Unpacking the Instrument* on page *2* for a list of replacement parts.

Catalog Number DOC022.53.00725

DR 2800 Spectrophotometer

PROCEDURES MANUAL

November 2005 Edition 1

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★ **Denotes USEPA accepted or approved for water or wastewater analysis.**

Method Precision Data 1

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Alachlor
Method 10202 Immunoassay Method

Aluminum
Method 8012 Aluminon Method Powder Pillows (0.008 to 0.800 mg/L)

Aluminum
Method 8326 Eriochrome Cyanine R Method Powder Pillows (0.002 to 0.250 mg/L Al^{3+})

Aluminum
Method 10215 Chromazurol S Method TNTplus™ 848 (0.02 to 0.50 mg/L Al)

Arsenic
★Method 8013 Silver Diethyldithiocarbamate Method (0 to 0.200 mg/L As)

Atrazine
Method 10050 Immunoassay Method

Barium
★Method 8014 Turbidimetric Method Powder Pillows (2 to 100 mg/L)

Benzotriazole/Tolyltriazole
Method 8079 UV Photolysis Method Powder Pillows (Benzotriazole 1.0 to 16.0 mg/L, Tolyltriazole 1.0 to 20.0 mg/L)

Boron
Method 10061 Azomethine-H Method Powder Pillows LR (0.02 to 1.50 mg/L as B)

Boron
Method 8015 Carmine Method Powder Pillows (0.2 to 14.0 mg/L)

Bromine
Method 8016 DPD Method Powder Pillows or AccuVac® Ampuls (0.05 to 4.50 mg/L)

Cadmium
Method 8017 Dithizone Method Powder Pillows (0 to 80.0 µg/L)

Cadmium
Method 10217 Cadion Method TNTplus™ 852 (0.02 to 0.30 mg/L Cd)

Chloramine (Mono)
Method 10172 Indophenol Method HR (0.1 to 10.0 mg/L Cl_2)

Chloramine (Mono)
Method 10171 Indophenol Method Powder Pillows LR (0.04 to 4.50 mg/L Cl_2)

Chloramine (Mono); Nitrogen, Free Ammonia
Method 10200 Indophenol Method Powder Pillows (0.01–0.50 mg/L NH_3–N; 0.04–4.50 mg/L Cl_2)

Chloride
Method 8113 Mercuric Thiocyanate Method (0.1 to 25.0 mg/L Cl^-)

Chlorine Dioxide
Method 8138 Direct Reading Method HR (5 to 1000 mg/L)

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Chlorine Dioxide	
Method 8065 Chlorophenol Red Method	LR (0.01 to 1.00 mg/L)
Chlorine Dioxide	
Amaranth Method	(20 to 500 µg/L)
Chlorine Dioxide	
Method 8345 Direct Reading Method	MR (1–50 mg/L)
Chlorine Dioxide	
★Method 10126 DPD Method Powder Pillows and AccuVac® Ampuls	(0.04 to 5.00 mg/L)
Chlorine, Free	
★Method 8021 DPD Method Powder Pillows or AccuVac® Ampuls	(0.02 to 2.00 mg/L)
Chlorine, Free	
Method 10102 DPD Method Test 'N Tube™ Vials	(0.09 to 5.00 mg/L)
Chlorine, Free	
Method 10059 DPD Rapid Liquid Method Pour-Thru™ Cell	(0.02 to 2.00 mg/L)
Chlorine, Free	
Method 10069 DPD Method Powder Pillows	HR (0.1 to 10.0 mg/L as Cl_2)
Chlorine, Total	
★Method 8167 DPD Method Powder Pillows or AccuVac® Ampuls	(0.02 to 2.00 mg/L)
Chlorine, Total	
Method 10070 DPD Method Powder Pillows	HR (0.1 to 10.0 mg/L as Cl_2)
Chlorine, Total	
Method 10101 DPD Method Test 'N Tube™ Vials	(0.09 to 5.00 mg/L)
Chlorine, Total	
Method 10060 DPD Rapid Liquid Method Pour-Thru Cell	(0.02 to 2.00 mg/L)
Chlorine, Total	
★Method 8370 DPD Method Pour-Thru™ Cell	ULR (2 to 500 µg/L as Cl2)
Chlorine, Total	
★Method 10014 DPD Method Pour-Thru Cell and OriFlo™ Filtration	ULR (2 to 500 µg/L as Cl_2)
Chromium, Hexavalent	
★Method 8023 1,5-Diphenylcarbohydrazide Method Powder Pillows or AccuVac® Ampuls	(0.010 to 0.700 mg/L Cr^{6+})
Chromium, Total	
Method 8024 Alkaline Hypobromite Oxidation Method, Powder Pillows	(0.01 to 0.70 mg/L)
Chromium, Total and Hexavalent	
Method 10218 (Chromium, Hexavalent) Method 10219 (Chromium, Total) 1,5-Diphenylcarbohydrazide Method TNTplus™ 854	(0.03 to 1.00 mg/L Cr)
Cobalt	
Method 8078 1-(2-Pyridylazo)-2-Naphthol (PAN) Method Powder Pillows	(0.01 to 2.00 mg/L)
Color, True and Apparent	
Method 8025 Platinum-Cobalt Standard Method,	(15 to 500 units)

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Copper
Method 8143 Porphyrin Method Powder Pillows — LR (1 to 210 µg/L)

Copper
★Method 8506 and Method 8026 Bicinchoninate Method Powder Pillows or AccuVac® Ampuls — (0.04 to 5.00 mg/L)

Cyanide
Method 8027 Pyridine-Pyrazalone Method Powder Pillows — (0.002 to 0.240 mg/L CN^-)

Cyanuric Acid
Method 8139 Turbidimetric Method Powder Pillows — (5 to 50 mg/L)

Fluoride
★Method 8029 SPADNS Method Reagent Solution or AccuVac® Ampuls — (0.02 to 2.00 mg/L F^-)

Formaldehyde
Method 8110 MBTH Method Powder Pillows — (3 to 500 µg/L)

Hardness
Method 8030 Calcium and Magnesium; Calmagite Colorimetric Method — (0.05 to 4.00 mg/L Ca and Mg as $CaCO_3$)

Hardness
Method 8374 Calcium and Magnesium; Chlorophosphonazo Colorimetric Method — (8 to 1000 µg/L Ca and Mg as $CaCO_3$)

Hardness, Total
Method 8374 Calcium and Magnesium; Chlorophosphonazo Rapid Liquid Method Pour-Thru Cell — ULR (4 to 1000 µg/L Ca & Mg as $CaCO_3$)

Hydrazine
Method 8141 p-Dimethylaminobenzaldehyde Method Reagent Solution or AccuVac® Ampuls — (4 to 600 µg/L)

Iodine
Method 8031 DPD Method Powder Pillows or AccuVac® Ampuls — (0.07 to 7.00 mg/L)

Iron
Method 8147 FerroZine® Method FerroZine® Reagent Solution Pillows — (0.009 to 1.400 mg/L)

Iron, Ferrous
Method 8146 1, 10 Phenanthroline Method Powder Pillows or AccuVac® Ampuls — (0.02 to 3.00 mg/L)

Iron
Method 8147 FerroZine® Rapid Liquid Method* Pour-Thru Cell — (0.009 to 1.400 mg/L Fe)

Iron, Total
Method 8112 TPTZ Method Powder Pillows or AccuVac® Ampuls — (0.012 to 1.800 mg/L)

Iron, Total
Method 8365 FerroMo Method Powder Pillows — (0.01 to 1.80 mg/L)

Iron, Total
★Method 8008 FerroVer® Method Powder Pillows or AccuVac® Ampuls — (0.02 to 3.00 mg/L)

Lead
Method 8317 LeadTrak® Fast Column Extraction Method — (5 to 150 µg/L)

Lead
★Method 8033 Dithizone Method Powder Pillows — (3 to 300 µg/L)

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Lead
Method 10216 PAR Method TNTplus™ 850 (0.1 to 2.0 mg/L Pb)

Manganese
Method 8149 1-(2-Pyridylazo)-2-Naphthol PAN Method Powder Pillows LR (0.006 to 0.700 mg/L)

Manganese
★Method 8034 Periodate Oxidation Method Powder Pillows HR (0.1 to 20.0 mg/L)

Mercury
Method 10065 Cold Vapor Mercury Concentration Method (0.1 to 2.5 µg/L)

Molybdenum, Molybdate
Method 8036 Mercaptoacetic Acid Method Powder Pillows or AccuVac® Ampuls HR (0.2 to 40.0 mg/L)

Molybdenum, Molybdate
Method 8169 Ternary Complex Method Powder Pillows (0.02 to 3.00 mg/L)

Nickel
Method 8150 1-(2 Pyridylazo)-2-Napthol (PAN) Method Powder Pillows (0.006 to 1.000 mg/L)

Nickel
★Method 8037 Heptoxime Method Powder Pillows (0.02 to 1.80 mg/L Ni)

Nickel
Method 10220 Dimethylglyoxime Method TNTplus™ 856 (0.1 to 6.0 mg/L Ni)

Nitrate
Method 8039 Cadmium Reduction Method Powder Pillows or AccuVac® Ampuls HR (0.3 to 30.0 mg/L NO_3^-–N)

Nitrate
Method 8192 Cadmium Reduction Method Powder Pillows LR (0.01 to 0.50 mg/L NO_3^-–N)

Nitrate
Method 10020 Chromotropic Acid Method Test ‘N Tube™ Vials HR (0.2 to 30.0 mg/L NO_3^-–N)

Nitrate
Method 10206 Dimethylphenol Method TNTplus 836 HR (5–35 mg/L NO_3^-–N or 22–155 mg/L NO_3)

Nitrate
Method 10206 Dimethylphenol Method TNTplus 835 LR (0.23 to 13.50 mg/L NO_3^-–N or 1.00 to 60.00 mg/L NO_3)

Nitrate
Method 8171 Cadmium Reduction Method Powder Pillows or AccuVac® Ampuls MR (0.1 to 10.0 mg/L NO_3^-–N)

Nitrite
Method 10019 Diazotization Method Test ‘N Tube™ Vials LR (0.003 to 0.500 mg/L NO_2^-–N)

Nitrite
★Method 8507 Diazotization Method Powder Pillows or AccuVac® Ampuls LR (0.002 to 0.300 mg/L NO_2^-–N)

Nitrite
Method 8153 Ferrous Sulfate Method Powder Pillows HR (2 to 250 mg/L NO_2^-)

Nitrite
★Method 10207 Diazotization Method TNTplus™ 839LR (0.015 to 0.600 mg/L NO_2^-–N or 0.05 to 2.00 mg/L NO_2)

Nitrogen, Ammonia
Method 8155 Salicylate Method Powder Pillows (0.01 to 0.50 mg/L NH_3–N)

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Nitrogen, Ammonia	
★Method 8038 Nessler Method	(0.02 to 2.50 mg/L NH_3–N)
Nitrogen, Ammonia	
Method 10031 Salicylate Method Test 'N Tube™ Vials	HR (0.4 to 50.0 mg/L NH_3–N)
Nitrogen, Ammonia	
Method 10023 Salicylate Method Test 'N Tube™ Vials	LR (0.02 to 2.50 mg/L NH_3–N)
Nitrogen, Ammonia	
Method 10205 Salicylate Method TNTplus™ 830	ULR (0.015 to 2.000 mg/L NH_3–N)
Nitrogen, Ammonia	
Method 10205 Salicylate Method TNTplus™ 831	LR (1 to 12 mg/L NH_3–N)
Nitrogen, Ammonia	
Method 10205 Salicylate Method TNTplus™ 832	HR (2 to 47 mg/L NH_3–N)
Nitrogen, Free Ammonia	
Method 10201 Indophenol Method Powder Pillows	(0.01 to 0.50 mg/L NH_3–N)
Nitrogen, Total	
Method 10072 Persulfate Digestion Method Test 'N Tube™ Vials	HR (2 to 150 mg/L N)
Nitrogen, Total	
Method 10071 Persulfate Digestion Method Test 'N Tube™ Vials	LR (0.5 to 25.0 mg/L N)
Nitrogen, Total	
Method 10208 Persulfate Digestion Method TNTplus™ 826	LR (1 to 16 mg/L N)
Nitrogen, Total	
Method 10208 Persulfate Digestion Method TNTplus™ 827	HR (5 to 40 mg/L N)
Nitrogen, Total	
Method 10208 Persulfate Digestion Method TNTplus™ 828	UHR (20 to 100 mg/L N)
Nitrogen, Total Inorganic	
Method 10021 Titanium Trichloride Reduction Method Test 'N Tube™ Vials	(0.2 to 25.0 mg/L N)
Nitrogen, Total Kjeldahl	
Method 8075 Nessler Method (Digestion Required)	(1 to 150 mg/L)
Organic Carbon, Total	
Method 10129 Direct Method	LR (0.3 to 20.0 mg/L C)
Organic Carbon, Total	
Method 10173 Direct Method	MR (15 to 150 mg/L C)
Organic Carbon, Total	
Method 10128 Direct Method	HR (100 to 700 mg/L C)
Oxygen, Dissolved	
★Method 8166 HRDO Method AccuVac® Ampuls	HR (0.3 to 15.0 mg/L O2)
Oxygen, Dissolved	
Method 8333 Ultra High Range Method AccuVac® Ampul	UHR (1.0 to 40.0 mg/L O_2)
Oxygen, Dissolved	
Method 8316 Indigo Carmine Method AccuVac® Ampuls	LR (6 to 800 µg/L O_2)

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★ **Denotes USEPA accepted or approved for water or wastewater analysis.**

Oxygen Demand, Chemical

★Method 8000 Reactor Digestion Method TNTplus™ — LR (TNT821, 3–150 COD); HR (TNT822, 20–1500mg/L COD)

Oxygen Demand, Chemical

★Method 8000 Reactor Digestion Method — (3 to 150, 20 to 1500, and 200 to 15,000 mg/L COD)

Oxygen Demand, Chemical

Method 10067 Manganese III Reactor Digestion Method (with optional chloride removal) — (30 to 1000 mg/L COD Mn)

Oxygen Demand, Chemical

Method 10067 Manganese III Reactor Digestion Method (without chloride removal) — (30 to 1000 mg/L COD Mn)

Oxygen Demand, Chemical

★Method 10211 Reactor Digestion Method TNTplus™ 820 — ULR (1–60 mg/L COD)

Oxygen Demand, Chemical

★Method 10212 Reactor Digestion Method TNTplus™ 823 — UHR (250–15,000 mg/L COD)

Oxygen Scavengers

Method 8140 Iron Reduction Method for Oxygen Scavengers Powder Pillows

Ozone

Method 8311 Indigo Method AccuVac® Ampul

PCB (Polychlorinated Biphenyls)

Method 10050 Immunoassay Method

Phenols

★Method 8047 4-Aminoantipyrine Method — (0.002 to 0.200 mg/L)

Phosphonates

Method 8007 Persulfate UV Oxidation Method Powder Pillows — (0.02 to 2.50 and 1.0 to 125.0 mg/L)

Phosphorus, Acid Hydrolyzable Digestion

★Method 8180 Acid Digestion Method

Phosphorus, Acid Hydrolyzable

Method 8180 PhosVer™ 3 with Acid Hydrolysis Method Test ‘N Tube™ Vials — (0.06 to 3.50 mg/L PO_4^{3-})

Phosphorus, Total, Digestion

★Method 8190 Acid Persulfate Digestion Method

Phosphorus, Reactive (Orthophosphate)

Method 8178 Amino Acid Method — (0.23 to 30.00 mg/L PO_4^{3-})

Phosphorus, Reactive

Method 8114 Molybdovanadate Rapid Liquid Method Pour-Thru Cell — HR (0.3 to 45.0 mg/L PO_4^{3-})

Phosphorus, Reactive (Orthophosphate)

Method 8114 Molybdovanadate Method Reagent Solution or AccuVac® Ampuls — (0.3 to 45.0 mg/L PO_4^{3-})

Phosphorus, Reactive (Orthophosphate)

Method 8114 Molybdovanadate Method Test ‘N Tube™ Vials — HR (1.0 to 100.0 mg/L PO_4^{3-})

★ **Denotes USEPA accepted or approved for water or wastewater analysis.**

Phosphorus, Reactive
Method 10055 Ascorbic Acid Rapid Liquid Method Pour-Thru Cell — LR (19 to 3000 µg/L PO_4^{3-})

Phosphorus, Reactive (Orthophosphate)
★Method 8048 PhosVer 3 (Ascorbic Acid) Method Powder Pillows or AccuVac® Ampuls — (0.02 to 2.50 mg/L PO_4^{3-})

Phosphorus, Reactive (Orthophosphate)
★Method 8048 PhosVer® 3 Method Test 'N Tube™ Vials — (0.06 to 5.00 mg/L PO_4^{3-} or 0.02 to 1.60 mg/L P)

Phosphorus, Reactive (Orthophosphate) and Total
Method 10209 Reactive; Method 10210 Total Ascorbic Acid Method TNTplus 843 — LR (0.15–4.50 mg/L PO_4^{3-} or 0.05–1.50 mg/L PO_4–P)

Phosphorus, Reactive (Orthophosphate) and Total
Method 10209 Reactive; Method 10210 Total Ascorbic Acid Method TNTplus™ 844HR — (1.5 to 15.0 mg/L PO_4^{3-} or 0.5 to 5.0 mg/L PO_4–P)

Phosphorus, Reactive (Orthophosphate) and Total
Method 10209 Reactive; Method 10210 Total Ascorbic Acid Method TNTplus™ 845UHR — (6 to 60 mg/L PO_4^{3-} or 2 to 20 mg/L PO_4–P)

Phosphorus, Reactive (Orthophosphate)
Method 10214 Molybdovanadate Method TNTplus™ 846 — (5.0 to 90.0 mg/L PO_4^{3-} or 1.6 to 30 mg/L PO_4–P)

Phosphorus, Total
★Method 8190 PhosVer® 3 with Acid Persulfate Digestion Method Test 'N Tube™ Vials — (0.06 to 3.50 mg/L PO_4^{3-} or 0.02 to 1.10 mg/L P)

Phosphorus, Total
Method 10127 Molybdovanadate Method with Acid Persulfate Digestion Test 'N Tube™ Vials — HR (1.0 to 100.0 mg/L PO_4^{3-})

Potassium
Method 8049 Tetraphenylborate Method Powder Pillows — (0.1 to 7.0 mg/L)

Quaternary Ammonium Compounds
Method 8337 Direct Binary Complex Method Powder Pillows — (0.2 to 5.0 mg/L as CTAB)

Selenium
Method 8194 Diaminobenzidine Method — (0.01 to 1.00 mg/L)

Silica
Method 8282 Heteropoly Blue Method Pour-Thru Cell — ULR(3 to 1000 µg/L as SiO_2)

Silica
Method 8282 Heteropoly Blue Rapid Liquid Method Pour-Thru Cell — ULR(3 to 1000 µg/L as SiO_2)

Silica
Method 8186 Heteropoly Blue Method Powder Pillows — LR (0.010 to 1.600 mg/L as SiO_2)

Silica
Method 8185 Silicomolybdate Method Powder Pillows — HR (1 to 100 mg/L)

Silver
Method 8120 Colorimetric Method Powder Pillows — (0.005 to 0.700 mg/L)

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★ **Denotes USEPA accepted or approved for water or wastewater analysis.**

Sulfate
★Method 8051 SulfaVer 4 Method Powder Pillows or AccuVac® Ampuls (2 to 70 mg/L)

Sulfide
★Method 8131 Methylene Blue Method (5 to 800 µg/L)

Sulfite
Colorimetric Method (0.10 to 5.00 mg/L)

Surfactants, Anionic (Detergents)
Method 8028 Crystal Violet Method (0.002 to 0.275 mg/L as LAS)

Suspended Solids
Method 8006 Photometric Method (5 to 750 mg/L)

Tannin and Lignin
Method 8193 Tyrosine Method (0.1 to 9.0 mg/L)

Toxicity
Method 10017 ToxTrak™ Method , ' (0 to 100% Inhibition)

TPH (Total Petroleum Hydrocarbons)
Method 10050 Immunoassay Method

Trihalomethanes
Method 10132 THM Plus™ Method Water Bath Method (10 to 600 ppb as Chloroform)

Volatile Acids
Method 8196 Esterification Method Powder Pillows (27 to 2800 mg/L)

Zinc
Method 8009 Zincon Method Powder Pillows (0.01 to 3.00 mg/L)

Expected Precision for DR 2800 Methods

PARAMETER	Method Number	Stored Program	Method	Concentration Range	95% Confidence Interval		
					Lower Limit	Target Conc.	Upper Limit
Aluminum	8012	10	Aluminon	0.008–0.800 mg/L Al	0.385	0.393	0.402
	8326	9	Eriochrome Cyanine R	0.002–0.250 mg/L Al	0.092	0.096	0.101
	10215	barcode	TNTplus 848 Chromazurol S	0.02–0.50 mg/L Al	0.32	0.35	0.38
Barium	8014	20	Turbidimetric	2–100 mg/L Ba	29	30	31
Benzotriazole	8079	30	UV Photolysis	0.2–16.0 mg/L Benzotriazole	9.7	10	10.3
Boron	10061	45	LR, Azomethine H	0.02–1.50 mg/L B	0.87	0.92	0.97
	8015	40	Carmine	0.2–14.0 mg/L B	7.5	7.6	7.7
Bromine	8016	50	DPD	0.05–4.50 mg/L Br_2	2.79	2.81	2.83
	8016	55	DPD, AccuVac	0.05–4.50 mg/L Br_2	2.63	2.77	2.9
Cadmium	8017	60	Dithizone	0.7–80.0 µg/L CD	39.3	40.0	40.7
	10217	barcode	TNTplus 852 Cadion	0.02–0.30 Cd	0.18	0.20	0.22
Chloramine, Mono	10172	67	HR TNT, Indophenol	0.1–10.0 mg/L Cl_2	5.6	5.9	6.2
	10171	66	LR TNT, Indophenol	0.04–4.50 mg/L Cl_2	2.58	2.60	2.62
Chloride	8113	70	Mercuric Thiocyanate	0.1–25.0 mg/L Cl^-	15.7	17.8	19.9
Chlorine Dioxide	n/a	78	Amaranth	20–500 µg/L ClO_2	192	250	308
	10126	76	DPD/Glycine	0.04–5.00 mg/L ClO_2	2.94	3.11	3.28
	10126	77	DPD/Glycine AccuVac	0.04–5.00 mg/L ClO_2	2.76	2.83	3.00
	8138	75	HR, Direct Reading (445 nm)	5–1000 mg/L ClO_2	459	475	492
	8065	72	LR, Chlorophenol Red	0.01–1.00 mg/L ClO_2	0.50	0.53	0.55
	8345	73	MR, Direct Reading (360 nm)	0.0–50 mg/L ClO_2	42	43	45
Chlorine, Free	8021	80	DPD	0.02–2.00 mg/L Cl_2	1.24	1.25	1.26
	8021	85	DPD, AccuVac	0.02–2.00 mg/L Cl_2	1.17	1.23	1.29
	10059	82	DPD Rapid Liquid	0.02–2.00 mg/L Cl_2	1.17	1.18	1.19
	10102	89	DPD TNT	0.09–5.00 mg/L Cl_2	2.63	2.68	2.72
	10069	88	HR, DPD	0.1–10.0 mg/L Cl_2	5.3	5.4	5.5
Chlorine, Total	8167	80	DPD	0.02–2.00 mg/L Cl_2	1.24	1.25	1.26
	8167	85	DPD, AccuVac	0.02–2.00 mg/L Cl_2	1.17	1.29	1.23
	10060	82	DPD, Rapid Liquid	0.02–2.00 mg/L Cl_2	1.17	1.19	1.18
	10101	89	DPD, TNT	0.09–5.00 mg/L Cl_2	2.63	2.68	2.72
	10070	88	HR, DPD	0.1–10.0 mg/L Cl_2	5.3	5.4	5.5
	8370	86	ULR, DPD	2–500 µg/L Cl_2	247	255	263

Expected Precision for DR 2800 Methods (continued)

PARAMETER	Method Number	Stored Program	Method	Concentration Range	95% Confidence Interval		
					Lower Limit	Target Conc.	Upper Limit
Chromium, Hexavalent	8023	90	1,5 Diphenylcarbohydrazide	0.010–0.700 mg/L Cr^{6+}	0.497	0.500	0.503
	8023	95	1,5 Diphenylcarbohydrazide, AccuVac	0.010–0.700 mg/L Cr^{6+}	0.497	0.503	0.5
	10218	barcode	TNTplus 854 1,5 Diphenylcarbohydrazide	0.03–1.00 mg/L Cr^{6+}	0.48	0.50	0.52
Chromium, Total	10219	barcode	TNTplus 854 1,5 Diphenylcarbohydrazide	0.03–1.00 mg/L Cr	0.48	0.50	0.52
	8024	100	Alkaline Hypobromite Oxidation	0.01–0.70 mg/L Cr	0.469	0.50	0.503
Cobalt	8078	110	PAN	0.01–2.00 mg/L Co	0.99	1.00	1.01
Color, True and Apparent	8025	120	Platinum-Cobalt (455 nm)	15–500 units Pt-Co	245	250	255
	8025	125	Platinum-Cobalt (465 nm)	15–500 units Pt-Co	247	250	253
Copper	8506	135	Bicinchoninate	0.04–5.00 mg/L Cu	0.97	1.00	1.03
	8026	140	Bicinchoninate, AccuVac	0.04–5.00 mg/L Cu	0.99	1.00	1.01
	8143	145	Porphyrin	1–210 µg/L Cu	47	50	53
Cyanide	8027	160	Pyridine-Pyrazolone	0.002–0.240 mg/L CN^-	0.090	0.100	0.110
Cyanuric Acid	8139	170	Turbidimetric	5–50 mg/L Cyanuric Acid	7	10	13
Fluoride	8029	190	SPADNS	0.02–2.00 mg/L F^-	0.97	1.00	1.03
	8029	195	SPADNS, AccuVac	0.02–2.00 mg/L F^-	0.92	1.00	1.08
Formaldehyde	8110	200	MBTH	2–500 µg/L CH_2O	312	320	328
Hardness, Calcium	8030	220	Calmagite	0.05–4.00 mg/L Ca as $CaCO_3$	1.90	2.00	2.10
Hardness, Magnesium	8030	225	Calmagite	0.05–4.00 mg/L,Mg as $CaCO_3$	1.92	2.00	2.09
Hardness, Total	8374	227	ULR, Chlorophosphonazo, Rapid Liquid	8–1000 µg/L, Ca and Mg as $CaCO_3$	478	500	522
Hardness, Total	8374	228	ULR, Chlorophosphonazo	8–1000 µg/L, Ca and Mg as $CaCO_3$	478	500	522
Hydrazine	8141	231	p-Dimethylaminobenzaldehyde	4–600 µg/L N_2H_4	242	250	258
	8141	232	AccuVac, p-Dimethylaminobenzaldehyde	4–600 µg/L N_2H_4	240	250	260

Expected Precision for DR 2800 Methods (continued)

PARAMETER	Method Number	Stored Program	Method	Concentration Range	95% Confidence Interval		
					Lower Limit	Target Conc.	Upper Limit
Iodine	8031	245	DPD	0.07–7.00 mg/L I_2	4.43	4.46	4.50
	8031	246	DPD, AccuVac	0.07–7.00 mg/L I_2	4.17	4.39	4.50
Iron	8147	260	FerroZine	0.009–1.400 mg/L Fe	0.985	1.000	1.015
	8147	261	FerroZine, Rapid Liquid	0.009–1.400 mg/L Fe	0.997	1.000	1.003
Iron, Ferrous	8146	255	1,10 Phenanthroline	0.02–3.00 mg/L Fe^{2+}	1.99	2.00	2.01
	8146	257	1,10 Phenanthroline, AccuVac	0.02–3.00 mg/L Fe^{2+}	2.03	2.05	2.07
Iron, Total	8008	265	FerroVer	0.02–3.00 mg/L Fe	1.99	2.00	2.01
	8112	270	TPTZ	0.012–1.800 mg/L Fe	1.032	1.043	1.054
	8365	275	FerroMo	0.01–1.80 mg/L Fe	0.98	1.00	1.02
	8008	267	FerroVer, AccuVac	0.02–3.00 mg/L Fe	2.03	2.05	2.07
	8112	272	TPTZ, AccuVac	0.012–1.800 mg/L Fe	1.008	1.024	1.040
Lead	8317	283	LeadTrak Fast Column Extraction	5–150 µg/L Pb	48	53	58
	8033	280	Dithizone	0–300 µg/L Pb	122	147	172
	10216	barcode	TNTplus 850, PAR	0.1–2.0 mg/L Pb	0.8	1.0	1.2
Manganese	8149	290	PAN	0.006–0.700 mg/L Mn	0.472	0.481	0.490
	8034	295	Periodate Oxidation	0.1–20.0 mg/L Mn	9.9	10.0	10.1
Mercury	10065	312	Cold Vapor	0.1–2.5 µg/L Hg	0.9	1.0	1.1
Molybdenum, Molybdate	8169	315	LR, Ternary Complex	0.02–3.00 mg/L Mo^{6+}	1.94	2.00	2.06
	8036	320	HR, Mercaptoacetic Acid	0.2–40.0 mg/L Mo^{6+}	9.7	10.0	10.3
	8036	322	HR, AccuVac, Mercaptoacetic Acid	0.2–40.0 mg/L Mo^{6+}	9.7	10.0	10.3
Nickel	8150	340	PAN	0.006–1.000 mg/L Ni	0.492	0.500	0.508
	8037	335	Heptoxime	0.02–1.80 mg/L Ni	0.97	1.00	1.03
	10220	barcode	TNTplus 856 Dimethylglyoxime	0.1–6.0 mg/L Ni	4.9	5.0	5.1
Nitrogen, Ammonia	8038	380	Nessler	0.02–2.50 mg/L NH_3–N	1.06	1.07	1.08
	8155	385	Salicylate	0.01–0.50 mg/L NH_3–N	0.45	0.47	0.49
	10031	343	HR, TNT, Salicylate	0.4–50.0 mg/L NH_3–N	32.1	34.0	35.9
	10023	342	LR, TNT, Salicylate	0.02–2.50 mg/L NH_3–N	0.83	0.93	1.03
	10205	barcode	TNTplus 832 ,HR Salicylate	1–47 mg/L NH_3–N	9	10	11
	10205	barcode	TNTplus 831, LR Salicylate	1–12 mg/L NH_3–N	9.6	10.0	10.4
	10205	barcode	TNTplus 830, ULR Salicylate	0.015–2.000 mg/L NH_3–N	0.968	1.000	1.032

Expected Precision for DR 2800 Methods (continued)

PARAMETER	Method Number	Stored Program	Method	Concentration Range	95% Confidence Interval		
					Lower Limit	Target Conc.	Upper Limit
Nitrogen, Nitrate	8039	355	HR, Cadmium Reduction	0.3–30.0 mg/L NO_3^-–N	14	14.7	15.4
	8039	361	HR, AccuVac Cadmium Reduction	0.3–30.0 mg/L NO_3^-–N	14	14.7	15.4
	10020	344	HR, TNT, Chromotropic Acid	0.2–30.0 mg/L NO_3^-–N	9.5	10.0	10.5
	8192	351	LR, Cadmium Reduction	0.01–0.50 mg/L NO_3^-–N	0.31	0.36	0.41
	8171	353	MR, Cadmium Reduction	0.1–10.0 mg/L NO_3^-–N	4.3	4.5	4.8
	8171	359	MR, AccuVac Cadmium Reduction	0.1–10.0 mg/L NO_3^-–N	6	6.4	6.8
	10206	barcode	TNTplus 836, HR Dimethylphenol	5–35 mg/L NO_3–N	9	10	11
	10206	barcode	TNTplus 835, LR Dimethylphenol	0.23–13.5 mg/L NO_3–N	9.37	10.00	10.63
Nitrogen, Nitrite	8153	373	HR, Ferrous Sulfate	2–250 mg/L NO_2^-	191	200	209
	8507	371	LR, Diazotization	0.002–0.300 mg/L NO_2^-–N	0.294	0.300	0.306
	8507	375	LR, AccuVac Diazotization	0.002–0.300 mg/L NO_2^-–N	0.147	0.150	0.153
	10019	345	LR, TNT Diazotization	0.002–0.300 mg/L NO_2^-–N	0.297	0.300	0.303
	10207	barcode	TNTplus 839 Diazotization	0.015–0.600 mg/L NO_2–N	0.290	0.300	0.310
Nitrogen, Total Inorganic	10021	346	TNT, Titanium Trichloride Reduction	0.2–25.0 mg/L N	21.2	21.4	21.6
Nitrogen, Total Kjeldahl	8075	399	Nessler	1–150 mg/L TKN	70	76	82
Nitrogen, Total	10072	394	HR, TNT Persulfate Digestion	10–150 mg/L N	98	100	102
	10071	350	LR, TNT Persulfate Digestion	0.5–25.0 mg/L N	9.6	10.0	10.4
	10208	barcode	TNTplus 827, HR Persulfate Digestion	5–40 mg/L N	29	30	31
	10208	barcode	TNTplus 826, LR Persulfate Digestion	1–16 mg/L N	9.7	10.0	10.3
	10208	barcode	TNTplus 828, UHR Persulfate Digestion	20–100 mg/L N	48	50	52

Expected Precision for DR 2800 Methods (continued)

PARAMETER	Method Number	Stored Program	Method	Concentration Range	95% Confidence Interval		
					Lower Limit	Target Conc.	Upper Limit
Oxygen Demand, Chemical (COD)	8000	435	HR, Reactor Digestion	200–15,000 mg/L COD	793	800	807
	8000	430	LR, Reactor Digestion	3–150 mg/L COD	77	80	83
	10067	432	Manganese III	20–1000 mg/L COD	575	600	625
	8000	431	ULR, Reactor Digestion	0.7–40.0 mg/L COD	28.9	30.0	31.2
	8000	barcode	TNTplus 822, HR Reactor Digestion	20–1500 mg/L COD	736	750	764
	8000	barcode	TNTplus 821, LR Reactor Digestion	3–150 mg/L COD	72	75	78
	10212	barcode	TNTplus 823, UHR Reactor Digestion	250–15,000 mg/L COD	5805	6000	6195
	10211	barcode	TNTplus 820, ULR Reactor Digestion	1–60 mg/L COD	48	50	52
Oxygen, Dissolved	8166	445	HR, AccuVac, HRDO	0.3–15.0 mg/L O_2	6.7	7.0	7.3
	8316	446	LR, AccuVac, Indigo Carmine	6–800 µg/L O_2	N/L	N/L	N/L
	8333	448	UHR, AccuVac	1.0–40.0 mg/L O_2	23.6	26.4	29.2
Oxygen Scavengers	8140	184	Iron Reduction	15–1000 µg/L, MEKO (Methylethylketoxime)	976	983	990
	8140	183	Iron Reduction	13–1500 µg/L Isoascorbic acid	886	893	899
	8140	182	Iron Reduction	9–1000 µg/L Hydroquinone	600	605	609
	8140	181	Iron Reduction	3–450 µg/L DEHA (Diethylhydroxylamine)	223	226	229
	8140	180	Iron Reduction	5–600 µg/L Carbohydrazide	299	302	304
Ozone	8311	456	HR, AccuVac, Indigo	0.01–1.50 mg/L O_3	1.05	1.23	1.41
	8311	454	LR, AccuVac, Indigo	0.01–0.25 mg/L O_3	0.14	0.20	0.26
	8311	455	MR, AccuVac, Indigo	0.01–0.75 mg/L O_3	0.42	0.59	0.76
Phenols	8047	470	4-Aminoantipyrine	0.002–0.200 mg/L phenol	0.093	0.100	0.107
Phosphonates	8007	501	Persulfate UV Digestion	multiple ranges	N/L	N/L	N/L
Phosphorus, Acid Hydolyzable	8180	536	TNT, PhosVer 3 with Acid Hydrolysis	0.06–3.50 mg/L PO_4^{3-}	2.92	2.98	3.04

Expected Precision for DR 2800 Methods (continued)

PARAMETER	Method Number	Stored Program	Method	Concentration Range	95% Confidence Interval		
					Lower Limit	Target Conc.	Upper Limit
Phosphorus, Reactive	8114	480	Molybdovanadate	0.3–45.0 mg/L PO_4^{3-}	30.42	30.80	31.18
	8178	485	Amino Acid	0.23–30.00 mg/L PO_4^{3-}	10.60	10.70	10.80
	10055	488	LR, Ascorbic Acid Rapid Liquid	19–3000 µg/L PO_4^{3-}	575	600	625
	8048	490	PhosVer 3	0.02–2.50 mg/L PO_4^{3-}	1.98	2.00	2.02
	8114	482	AccuVac, Molybdovanadate	0.3–45.0 mg/LPO_4^{3-}	31.0	31.2	31.4
	8048	492	AccuVac, PhosVer 3	0.02–2.50 mg/L PO_4^{3-}	1.99	2.00	2.01
	8114	489	HR, Molybdovanadate, Rapid Liquid	0.3–45.0 mg/L PO_4^{3-}	10.4	10.5	10.7
	8048	535	TNT, PhosVer 3	0.06–5.00 mg/L PO_4^{3-}	2.92	2.98	3.04
	8114	540	TNT, Molybdovanadate	1.0–100.0 mg/L PO_4^{3-}	49.4	50.3	51.2
	10214	barcode	TNTplus 846, Molybdovanadate	5.0–90.0 mg/L PO_4^{3-}	48.4	50.0	51.6
Phosphate, Total and Reactive	10209 10210	barcode	TNTplus 845, Ascorbic Acid	6–60 mg/L PO_4^{3-}	49	50.0	51
	10209 10210	barcode	TNTplus 844, Ascorbic Acid	1.5–15.0 mg/L PO_4^{3-}	9.5	10.0	10.5
	10209 10210	barcode	TNTplus 843, Ascorbic Acid	0.15–1.50 mg/L PO_4^{3-}	3.39	3.50	3.61
Phosphorus, Total & Acid Hydrolyzable	8190	536	TNT, PhosVer 3 with Acid Persulfate	0.06–3.50 mg/L PO_4^{3-}	3.05	3.12	3.19
Phosphorus, Total HR	10127	540	TNT, Molybdovanadate with Acid Persulfate	1.0–100.0 mg/L PO_4^{3-}	52.1	52.8	53.5
Potassium	8049	905	Tetraphenylborate	0.1–7.0 mg/L K	4.8	5.0	5.2
Quaternary Ammonium Compounds	8337	401	Direct Binary Complex	0.2–5.0 mg/L CTAB	2.74	3.00	3.26
Selenium	8194	640	Diaminobenzidine	0.01–1.00 mg/L Se	0.47	0.50	0.53
Silica	8185	656	HR, Silicomolybdate	1–100 mg/L SiO_2	47	49	51
	8186	651	LR, Heteropoly Blue	0.010–1.600 mg/L SiO_2	0.970	0.980	0.990
	8282	645	ULR, Heteropoly Blue	3–1000 µg/L SiO_2	526	530	534
	8282	645	ULR, Heteropoly Blue Rapid Liquid	3–1000 µg/L SiO_2	526	530	534
Silver	8120	660	Colorimetric	0.005–0.700 mg/L Ag	0.468	0.480	0.492
Sulfate	8051	680	SulfaVer 4	2–70 mg/L SO_4^{2-}	30	40	50
	8051	685	AccuVac, SulfaVer 4	2–70 mg/L SO_4^{2-}	32	40	48
Sulfide	8131	690	Methylene Blue	5–800 µg/L S^{2-}	504	520	536
Sulfite	—	692	HPT430	0.10–5.00	N/L	N/L	N/L
Surfactants, Anionic (Detergents)	8028	710	Crystal Violet	0.002–0.275 mg/L LAS	0.172	0.180	0.188

Expected Precision for DR 2800 Methods (continued)

PARAMETER	Method Number	Stored Program	Method	Concentration Range	95% Confidence Interval		
					Lower Limit	Target Conc.	Upper Limit
Suspended Solids	8006	630	Photometric	5–750 mg/L Susp. Solids	N/L	N/L	N/L
Tannin & Lignin	8193	720	Tyrosine	0.1–9.0 mg/L tannin	5.8	6.0	6.2
TOC HR (Total Organic Carbon)	10128	426	Colorimetric	100–700 mg/L C	341	354	367
TOC LR (Total Organic Carbon)	10129	427	Colorimetric	0.3–20.0 mg/L C	7.8	8.7	9.5
TOC MR (Total Organic Carbon)	10173	425	Colorimetric	15–150 mg/L C	68	71	73
Tolyltriazole	8079	730	UV Photolysis	1.0–20.0 mg/L Tolyltriazole	11.6	12.0	12.4
Trihalomethanes	10132	725	THM Plus™	10–600 µg/L THM	53	66	73
Volatile Acids	8196	770	Esterification	27–2800 mg/L HOAC	1211	1343	1475
Zinc	8009	780	Zincon	0.01–3.00 mg/L Zn	0.97	1.0	1.03

This document explains the layout and location of information in a typical written chemical procedure. Layout may vary slightly between procedures, depending on the level of detail and the steps required for a particular test.

Method Identification Number

Approval of Acceptance of method by United States EPA where applicable

Procedure Name

Types of samples analyzed

Name of method used

Reference for method used

Range with units of measure

Sulfide

★**Method 8131**

Methylene Blue Method

(5 to 800 µg/L)

Scope and Application: For testing total sulfides, H_2S, HS^-, and certain metal sulfides in groundwater, wastewater brines, and seawater; USEPA Approved for reporting wastewater analysis[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

[2] Procedure is equivalent to USEPA method 376.2 and Standard Method 4500-S^{2-} D for wastewater.

Reference for USEPA approval or acceptance

Important information to review before starting the procedure

Test Preparation

Before starting the test:

Analyze samples immediately. Do not preserve for later analysis.
Avoid excessive agitation of samples to minimize sulfide loss.
Some sulfide loss may occur if dilution is necessary.
Sulfide 2 reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration regulated as a hazardous waste by Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.

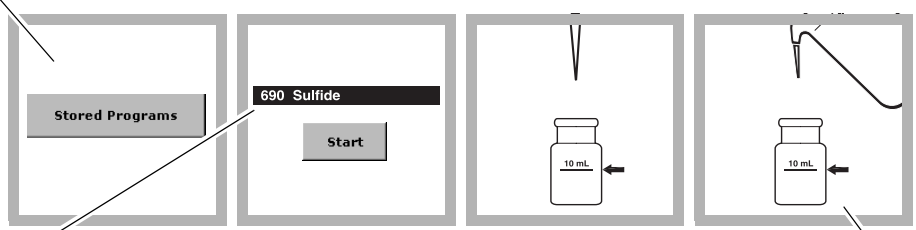
Items to assemble to perform the procedure

Collect the following items:	**Quantity**
Sulfide 1 Reagent	2 mL
Sulfide 2 Reagent	2 mL
Water, deionized	25 mL
Pipet, serological, 10-mL	1
Pipet Filler, safety bulb	1
Sample Cells, 1-inch square, 10 mL, matched pair	2
Stopper for 18-mm tube	2

Note: *Reorder information for consumables and replacement items is on page 4.*

Screen presses to access the program and run the test

Method 8131



1. Press **STORED PROGRAMS**.

2. Select the test.

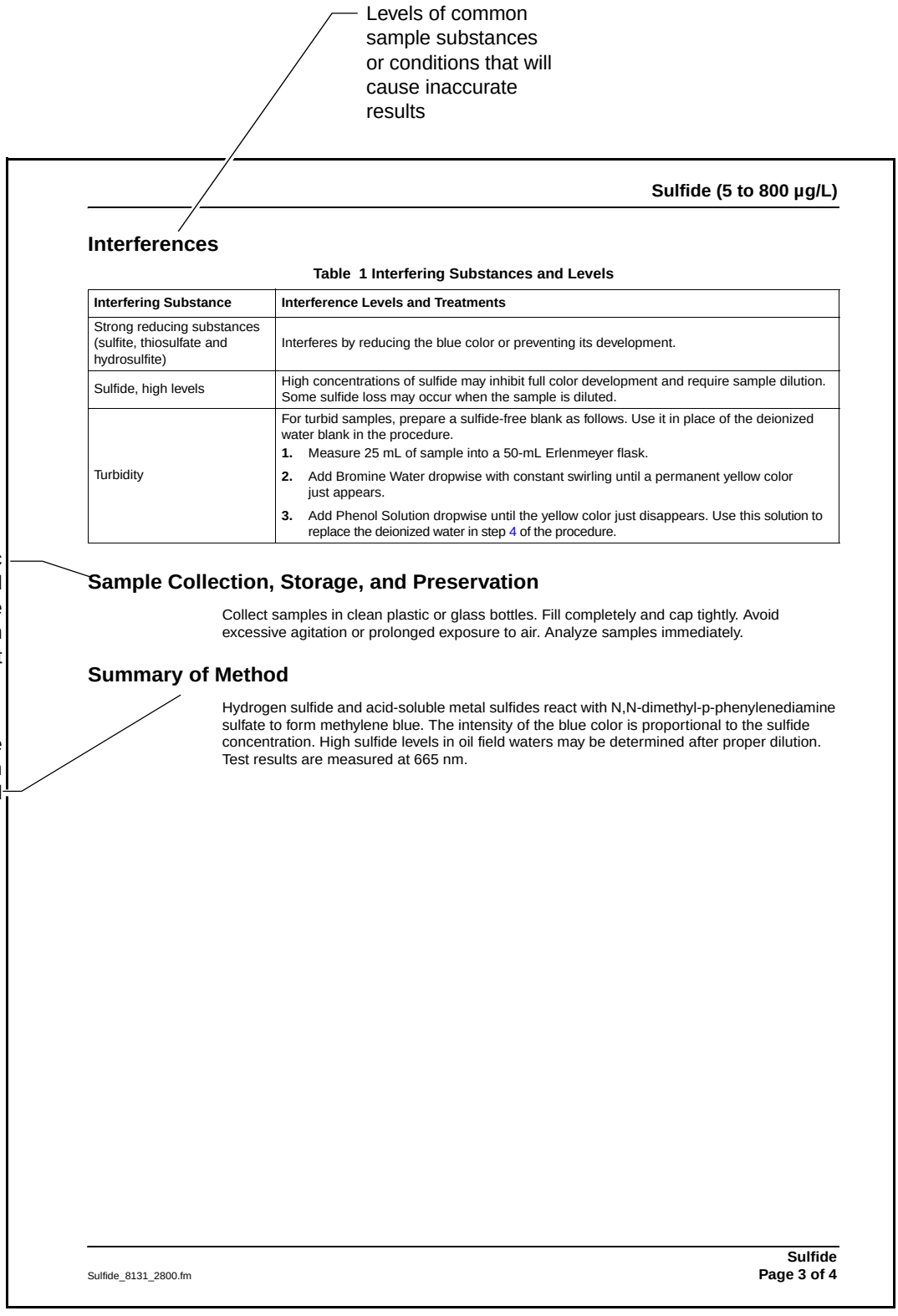
3. Prepared Sample: Avoiding excess agitation of the sample, use a pipet add 10 mL of sample to a square sample cell.

4. Blank Preparation: Measure 10 mL of deionized water into a second square sample cell.

Program number

Illustration of procedure steps

Procedure step



Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Strong reducing substances (sulfite, thiosulfate and hydrosulfite)	Interferes by reducing the blue color or preventing its development.
Sulfide, high levels	High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.
Turbidity	For turbid samples, prepare a sulfide-free blank as follows. Use it in place of the deionized water blank in the procedure. **1.** Measure 25 mL of sample into a 50-mL Erlenmeyer flask. **2.** Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears. **3.** Add Phenol Solution dropwise until the yellow color just disappears. Use this solution to replace the deionized water in step 4 of the procedure.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

Summary of Method

Hydrogen sulfide and acid-soluble metal sulfides react with N,N-dimethyl-p-phenylenediamine sulfate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. High sulfide levels in oil field waters may be determined after proper dilution. Test results are measured at 665 nm.

Lists all reagents and standards required for the procedure

Items recommended for accuracy verification

Quantity of reagent or apparatus needed to perform the procedure

Sulfide (5 to 800 µg/L)

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Sulfide Reagent Set (100 tests), includes:	—	—	22445-00
(2) Sulfide 1 Reagent	2 mL	100 mL MDB	1816-32
(2) Sulfide 2 Reagent	2 mL	100 mL MDB	1817-32
Water, deionized	25 mL	4 liters	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Pipet, serological, 10-mL	1	each	532-38
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper, for 18-mm Tube	2	6/pkg	1731-06

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bromine Water	29 mL	2211-20
Phenol Solution	29 mL	2112-20
Stopper, for 18-mm Tube	25/pkg	1731-25

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Updated June 05 Edition 1

Alachlor

Method 10202

Immunoassay Method[1]

Scope and Application: For water

[1] This test is semi-quantitative. Results are expressed as greater or less than the threshold value used.

Test Preparation

This method analyzes for Alachlor in water. Sample calibrators and reagents are added to cuvettes coated with Alachlor-specific antibodies. The color that develops is then measured and compared with the color measurements of the calibrators. The test requires about 30 minutes for complete analysis. As many as 20 cuvettes (18 samples and 2 calibrators) can be run simultaneously.

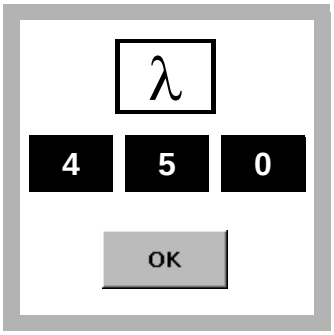
Before starting the test:

Read the entire procedure before starting. Identify and make ready all the necessary reagents, cuvettes, and other apparatus before beginning the analysis.
Timing is critical; follow instructions carefully.
A consistent technique when mixing the cuvettes is critical to this test. The best results come from using the cuvette rack and mixing as described in *Using the 1-cm MicroCuvette Rack on page 4.* Cuvettes can be mixed individually, but test results may not be as consistent.
Handle the cuvettes carefully. Scratches on the inside or outside may cause erroneous results. Carefully clean the outside of the cuvettes with a clean absorbent cloth or tissue before placing them into the instrument.
Antibody cuvettes and enzyme conjugate are made in matched lots. Do not mix reagent lots.
To avoid damaging the Color Developing Solution, do not expose it to direct sunlight.
The cuvette rack is designed to be inverted with the cuvettes in place. This is especially helpful when running many samples at once; the cuvettes can remain in the rack and be processed together until they are read in the spectrophotometer.
Twenty Antibody Cuvettes are provided with each reagent set. One Antibody Cuvette will be used for each calibrator and each sample. Cuvettes are not reusable.
Protective nitrile gloves are recommended for this procedure.

Collect the following items	**Quantity**
Alachlor Reagent Set	1
Caps, flip spout	1
Marker, laboratory	1
Rack, for 1-cm Micro Cuvettes	1
Wipes, disposable	1
Pipet, TenSette®, 0.1–1.0 mL	1
Pipet tip for 19700-01, TenSette Pipet	1

Reorder information for consumables and replacement items is on page 7.

Immunoassay for Water

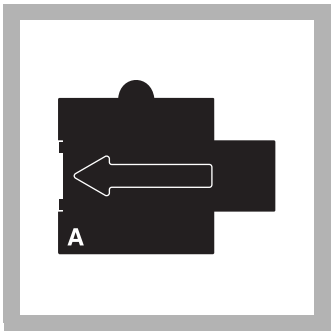


1. Press

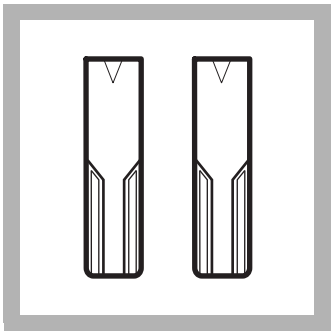
SINGLE WAVELENGTH

Press **OPTIONS** and the λ button. Enter

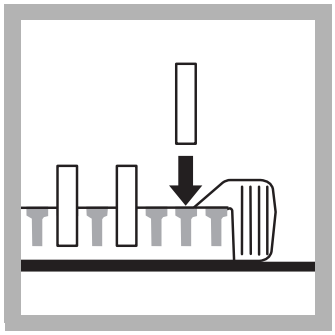
450 nm and press **OK.**



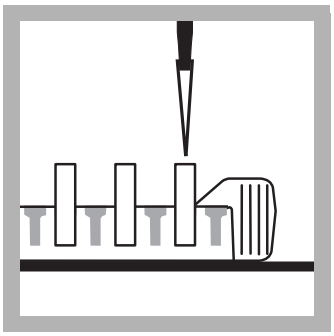
2. Insert Adapter A.



3. Label an Antibody Cuvette for each calibrator and each sample to be tested.

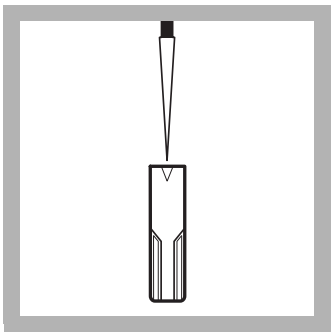


4. Insert the cuvettes into the rack snugly.



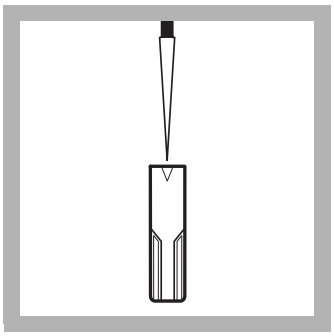
5. Pipet 0.5 mL of each calibrator into the appropriately labeled cuvette.

Use a new pipette tip for each calibrator.

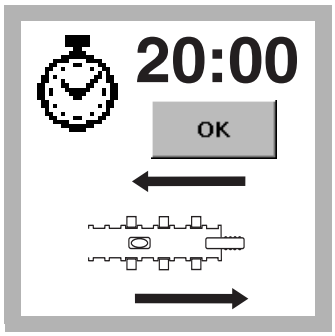


6. Pipet 0.5 mL of each sample to be tested into the appropriately labeled cuvette.

Use a new pipette tip for each sample.



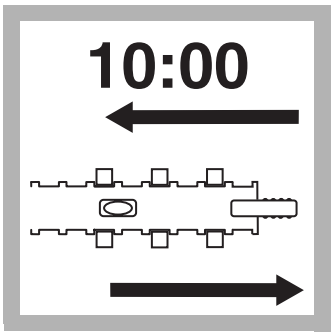
7. Immediately pipet 0.5 mL of Alachlor Enzyme Conjugate into each cuvette.



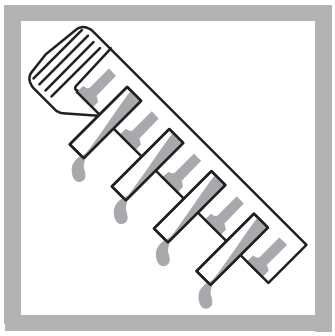
8. Press **OPTIONS**. Press **TIMER**. Enter 20:00 minutes and press **OK.**

A 20-minute reaction time will begin.

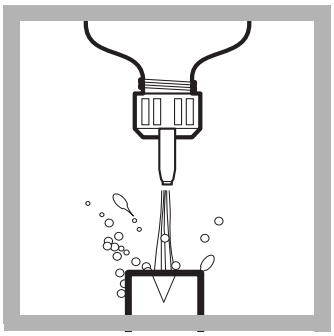
Immediately mix the contents of the cuvettes for 30 seconds using the technique described in Using the 1-cm MicroCuvette Rack on page 4.



9. After 10 minutes mix the contents of the rack for 30 seconds using the technique described in Using the 1-cm MicroCuvette Rack on page 4.



10. At the end of the 20-minute period, discard the contents of all the cuvettes into an appropriate waste container.

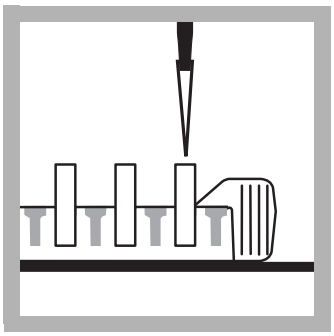


11. Wash each cuvette forcefully and thoroughly four times with deionized water. Empty the rinse water into the waste container.

Ensure that most of the water is drained from the cuvettes by turning the cuvettes upside down and tapping them lightly on a paper towel.

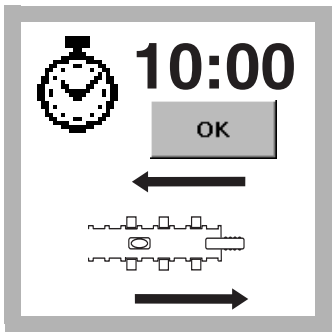
Color Development

Important Note: *Timing is critical. Follow instructions carefully.*



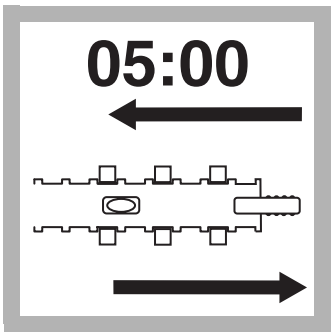
12. With the cuvettes still held snugly in the rack, pipet 0.5 mL of Color Developing Solution into each Antibody Cuvette.

Use a new pipette tip for each cuvette.



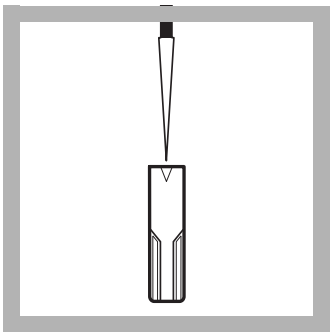
13. Press **OPTIONS**. Press **TIMER**. Enter 10:00 minutes and press **OK.**

A reaction period will begin. Mix, using the instructions on page 4.



14. After 5 minutes, mix the contents of the rack a second time for a period of 30 seconds using the same technique as step 13.

Solutions will turn blue in some or all of the cuvettes.



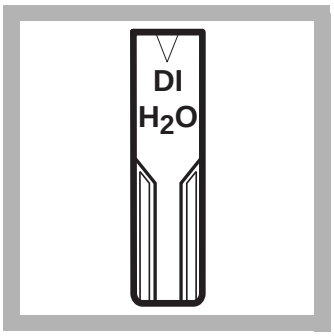
15. At the end of the 10-minute reaction period, pipette 0.5 mL of Stop Solution into each cuvette in the same order as the Color Developing Solution was added in step 12.

Slide the rack for 20 seconds (Using the 1-cm MicroCuvette Rack on page 4.)

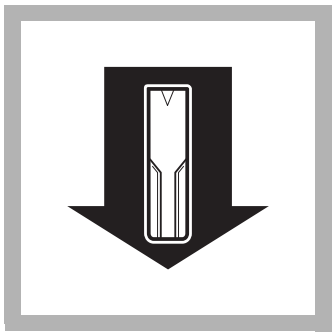
Blue solutions will turn yellow with the addition of the Stop Solution.

Use the same pipette tip repeatedly for this step.

Measuring the Color

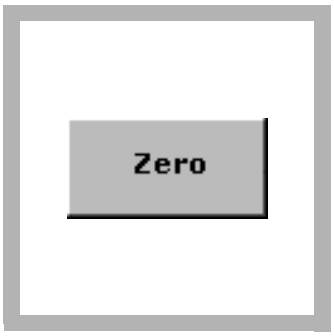


16. Label and fill a Zeroing Cuvette with deionized water. Wipe the outside of all the cuvettes with a tissue to remove water, smudges, and fingerprints.



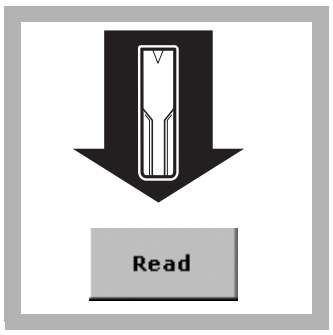
17. Insert the filled Zeroing Cuvette into the cell holder—arrow pointing to the right.

Orient the arrow in the same direction for all cuvettes.



18. Press **ZERO**.

The display will show: 0.000 Abs



19. Insert the first calibrator into the cell holder.

Press **READ**. The display will give an absorbance reading. Record the results for each calibrator and sample.

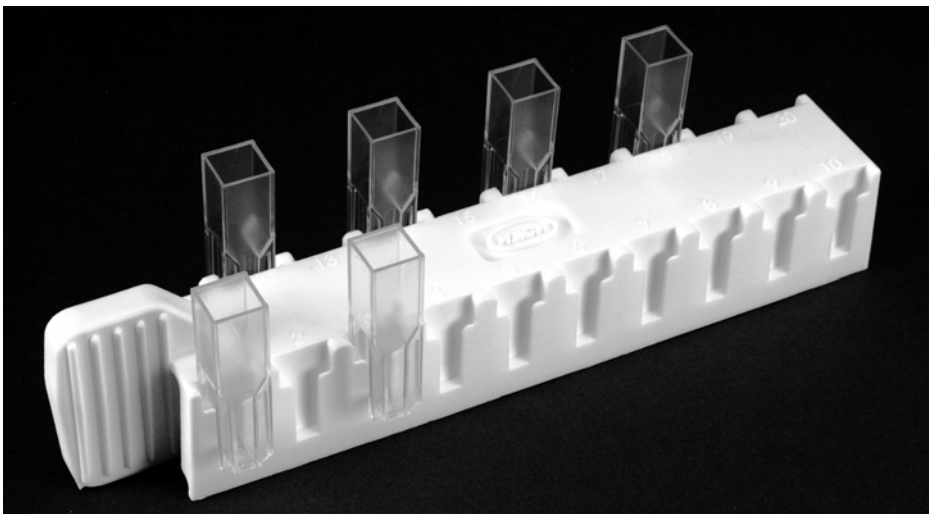
Repeat this step for all remaining calibrators and samples.

See Interpreting and Reporting Results for help with interpretation of results.

Using the 1-cm MicroCuvette Rack

The MicroCuvette rack (Figure 1) has been designed specifically to aid in achieving precise and accurate results when using the immunoassay technique to analyze several samples at the same time.

Figure 1 The 1-cm MicroCuvette Rack



Loading the Rack—The cuvette rack is designed so that it may be inverted with the cuvettes in place. Identify each cuvette with a sample or calibrator number and insert all the cuvettes in the rack before beginning the procedure. Fit the cuvettes snugly into the rack, but do not force them or they may be difficult to remove and their contents may spill. The cuvettes should remain in place when the rack is inverted and tapped lightly.

Mixing—Set the rack on a hard, flat surface that is at least twice the length of the rack. Hold the rack by one end and vigorously slide it back and forth along its long axis for 30 seconds. The rack should move through a distance equal to its own length in each direction.

Interpreting and Reporting Results

There is an inverse relationship between the concentration of Alachlor and the reading. In other words, the higher the reading, the lower the concentration of Alachlor (Table 1.

Table 1 Relative Alachlor Concentration

If the sample reading is...	the sample Alachlor Concentration is...
…less than calibrator reading	…greater than the calibrator concentration
…greater than calibrator reading	…less than the calibrator concentration

Example
Readings:

0.1 ppb Alachlor Calibrator: **0.475 Abs**

0.5 ppb Alachlor Calibrator: **0.245 Abs**

Sample #1: **0.140 Abs**

Sample #2: **0.300 Abs**

Sample #3: **0.550 Abs**

Interpretation
Sample #1—Sample reading is less than the readings for both calibrators. Therefore the sample concentration of Alachlor is greater than both 0.1 ppb and 0.5 ppb Alachlor.

Sample #2—Sample reading is between the readings for the 0.1 ppb and 0.5 ppb Alachlor calibrators. Therefore the sample concentration of Alachlor is between 0.1 ppb and 0.5 ppb.

Sample #3—Sample reading is greater than the readings for both calibrators. Therefore the sample concentration of Alachlor is less than both 0.5 ppb and 0.1 ppb.

Storing and Handling Reagents

- Wear protective gloves and eyewear.
- When storing reagent sets for extended periods of time, keep them out of direct sunlight. Store reagents at a temperature of 4 °C when not in use.
- Keep the foil pouch containing the Antibody Cuvettes sealed when not in use.
- If Stop Solution comes in contact with eyes, wash thoroughly for 15 minutes with cold water and seek immediate medical help.

Sensitivity

The Alachlor immunoassay test cannot differentiate between certain herbicides and metabolites, but it detects their presence to differing degrees. Table 2 shows the required concentration for selected chemicals.

Table 2 Required Concentration for Selected Chemicals

Compound	Concentration to give a positive response of 0.1 ppb Alachlor	Concentration to give a positive response of 0.5 ppb Alachlor
Acetochlor	0.45 ppb	4 ppb
Butachlor	0.09 ppm	1 ppm
2 Chloro-2',6'-Diethylacetaniline	0.030 ppm	2 ppm
Metolachlor	0.085 ppm	2 ppm
2,6-Diethylaniline	0.3 ppm	9 ppm
Propachlor	0.72 ppb	12 ppb

Table 3 Compounds not detectable at 10,000 ppb

Atrazine	2, 4-D
Aldicarb	Chlorpyirfos
Diazoton	Carbendazim
Carbofuran	

Sample Collection and Storage

Collect samples in a clean glass bottle. Do not pre-rinse the bottle with the sample. If the sample cannot be analyzed immediately, store the sample at 4 °C. Samples may be kept for as long as 14 days. Warm the samples to room temperature before analysis.

Diluting Water Samples

Other levels of Alachlor can be tested by diluting the sample and comparing the results to the 0.1 ppb Calibrator. Select the appropriate sample volume from Table 4, place it in a graduated mixing cylinder, and dilute it to 50 mL with deionized water.

Table 4 Sample Volume and Concentration

mL Sample	Representative Concentration using 0.1 ppb Calibrator
0.5	10 ppb
1.0	5 ppb
2.5	2 ppb
5.0	1 ppb

Example:
Dilute 2.5 mL of sample to 50 mL with deionized water. Run the diluted sample in the procedure along with the 0.1 ppb calibrator. If the absorbance of the diluted sample is less than the 0.1 ppb calibrator, the concentration of the original sample is greater than 2 ppb.

Summary of Method

Immunoassay tests use antigen/antibody reactions to test for specific organic compounds in water and soil. Alachlor-specific antibodies, attached to the walls of plastic cuvettes, selectively bind and remove Alachlor from complex sample matrices. A prepared sample and a reagent containing enzyme-conjugate molecules (analyte molecules attached to molecules of an enzyme) are added to the Antibody Cuvettes. During incubation, enzyme-conjugate molecules and Alachlor compete for binding sites on the antibodies. Samples with higher levels of analyte will have more antibody sites occupied by Alachlor and fewer antibody sites occupied by the enzyme-conjugate molecules.

After incubation, the sample and unbound enzyme conjugate are washed from the cuvette and a color-development reagent is added. The enzyme in the conjugate catalyzes the development of color. Therefore, there is an inverse relationship between color intensity and the amount of Alachlor in the sample. The resulting color is then compared with a calibrator to determine whether the Alachlor concentration in the sample is greater or less than the threshold levels. Test results are measured at 450 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Alachlor Reagent Set[1]	20 cuvettes	28130-00

[1] Immunoassay components are manufactured by Beacon Analytical Systems, Inc.

Required Apparatus

Description	Unit	Cat. No.
Adapter, 1-cm square cell	each	
Caps, flip spout	2/pkg	25818-02
Marker, laboratory	each	20920-00
Pipet, TenSette®, Pipet, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Rack, for 1-cm Micro Cuvettes	each	48799-00
Wipes, disposable	box	20970-00

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Glasses, Safety	each	27568-00
Gloves, Disposable, Nitrile, Medium[1]	each	25505-02
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96

[1] Other sizes available.

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Updated November 2005 Edition 1

Aluminum

Method 8012

Aluminon Method[1]

Powder Pillows

(0.008 to 0.800 mg/L)

Scope and Application: For water and wastewater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

Before starting the test:

Digestion is required for determining total aluminum.
Clean all glassware with 6.0 N HCl and deionized water before use to remove contaminants from the glass.
Check the sample temperature. It must be between 20–25 °C (68 –77 °F) for accurate results.

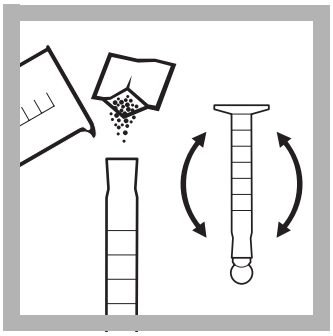
Collect the following items:	**Quantity**
AluVer® 3 Aluminum Reagent Powder Pillow	1
Ascorbic Acid Powder Pillow	1
Bleaching 3 Reagent Powder Pillow	1
50-mL graduated mixing cylinder with glass stopper	1
Sample cells, 1-inch square, 10 mL	2

Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows — Method 8012

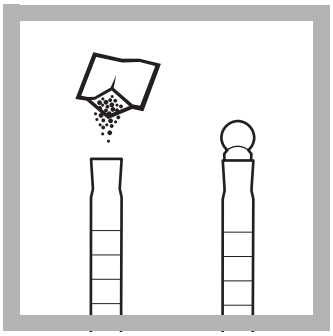
1. Press **STORED PROGRAMS**.

2. Select the test.



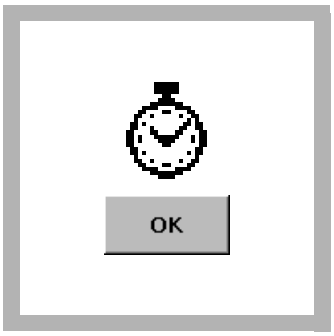
3. Fill the cylinder to the 50-mL mark with sample. Add one Ascorbic Acid Powder Pillow.

Stopper. Invert several times to dissolve the powder.

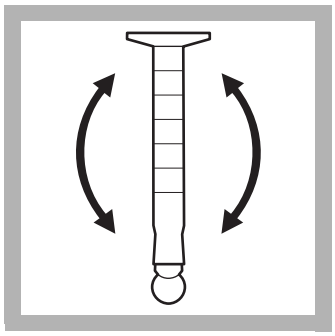


4. Add one AluVer 3 Aluminum Reagent Powder Pillow. Insert the stopper.

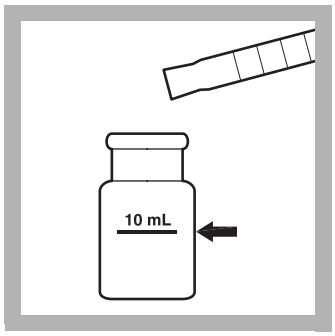
An orange to orange-red color will develop if aluminum is present.



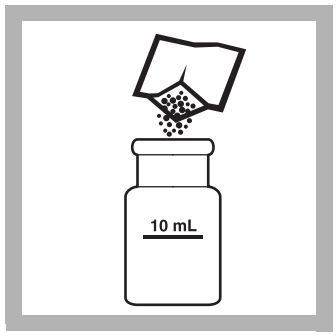
5. Press **TIMER>OK**.



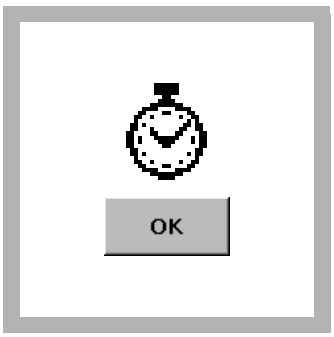
6. Invert repeatedly for one minute to dissolve the powder. Undissolved powder will cause inconsistent results.



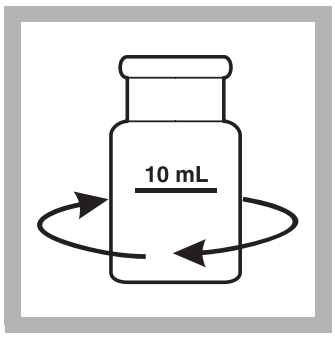
7. Blank Preparation: Pour 10 mL of the mixture into a square sample cell.



8. Add one Bleaching 3 Reagent Powder Pillow to the blank.



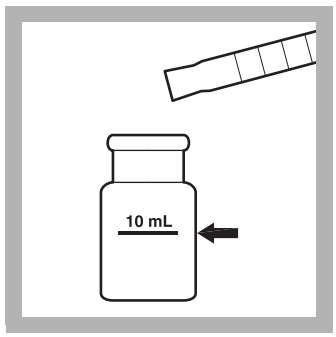
9. Press **TIMER>OK**.



10. Vigorously swirl the cell for 30 seconds. The solution should turn a light to medium orange.

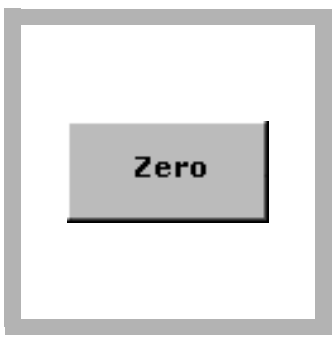
11. Press **TIMER>OK**.

A 15-minute reaction period will begin.



12. Prepared Sample: Pour 10 mL of solution from the cylinder into a second square sample cell.

13. Within five minutes after the timer expires, wipe and dry the blank and place it into the cell holder with the fill line facing right.



14. Press **ZERO**.

The display will show:

0.000 mg/L Al^{3+}

15. Immediately wipe and dry the prepared sample and place it into the cell holder with the fill line facing right.

16. Press **READ**.

Results are in mg/L Al^{3+}.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 300 mg/L as $CaCO_3$. Samples with greater than 300 mg/L acidity as $CaCO_3$ must be treated as follows: **1.** Add one drop of m-Nitrophenol Indicator Solution[1] to the sample taken in step 3. **2.** Add one drop of 5.0 N Sodium Hydroxide Standard Solution[1]. Stopper the cylinder. Invert to mix. Repeat as often as necessary until the color changes from colorless to yellow. **3.** Add one drop of 5.25 N Sulfuric Acid Standard Solution[1] to change the solution from yellow back to colorless. Continue with the test.
Alkalinity	1000 mg/L as $CaCO_3$. Interferences from higher alkalinity concentrations can be eliminated by the following pretreatment: **1.** Add one drop of m-Nitrophenol Indicator Solution[1] to the sample taken in step 3. A yellow color indicates excessive alkalinity. **2.** Add one drop of 5.25 N Sulfuric Acid Standard Solution[1]. Stopper the cylinder. Invert to mix. If the yellow color persists, repeat until the sample becomes colorless. Continue with the test.
Fluoride	Interferes at all levels. See Figure 1 on page 4.
Iron	Greater than 20 mg/L
Phosphate	Greater than 50 mg/L
Polyphosphate	Polyphosphate interferes at all levels by causing negative errors and must not be present. Before running the test, polyphosphate must be converted to orthophosphate by acid hydrolysis as described under the phosphorus procedures.

[1] See Optional Reagents on page 6 for reorder information.

Fluoride interferes at all levels by complexing with aluminum. The actual aluminum concentration can be determined using the Fluoride Interference Graph (Figure 1) when the fluoride concentration is known.

To use the fluoride interference graph:

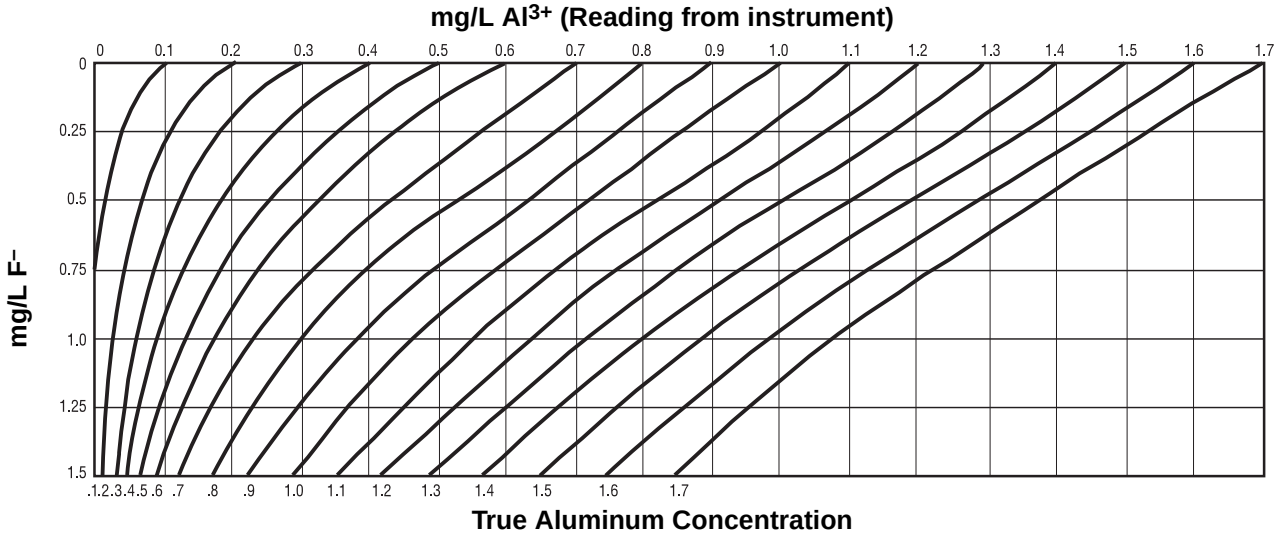
1. Select the vertical grid line along the top of the graph that represents the aluminum reading obtained in step 15.

- Locate the point on the line where it intersects with the horizontal grid line that indicates how much fluoride is present in the sample.

- Extrapolate the true aluminum concentration by following the curved lines on either side of the intersect point down to the true aluminum concentration.

For example, if the aluminum test result was 0.7 mg/L Al and the fluoride present in the sample was 1 mg/L F^-, the point where the 0.7 grid line intersects with the 1 mg/L F^- grid line falls between the 1.2 and 1.3 mg/L Al curves. In this case, the true aluminum content would be 1.27 mg/L.

Figure 1 Fluoride Interference Graph



Sample Collection, Storage, and Preservation

Collect samples in a clean glass or plastic containers. Preserve the sample by adjusting the pH to 2 or less with nitric acid (about 1.5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 3.5–4.5 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row.

4. Open an Aluminum Voluette® Ampule Standard, 50-mg/L Al.

5. Prepare three sample spikes. Fill three mixing cylinders (Cat. No. 1896-41) with 50 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 0.4-mg/L aluminum standard solution as follows: Pipet 1.00 mL of Aluminum Standard Solution, 100-mg/L as Al^{3+}, into a 250-mL volumetric flask.

2. Dilute to the mark with deionized water. Prepare this solution daily. Perform the aluminum procedure as described above.

Or, use the followign alternative procedure:

1. Using the TenSette Pipet, add 0.8 mL of solution from an Aluminum Voluette Ampule Standard Solution (50-mg/L as Al) into a 100-mL volumetric flask.

2. Dilute to volume with deionized water. Perform the aluminum procedure as described.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Aluminon indicator combines with aluminum in the sample to form a red-orange color. The intensity of color is proportional to the aluminum concentration. Ascorbic acid is added to remove iron interference. The AluVer 3 Aluminum Reagent, packaged in powder form, shows exceptional stability and is applicable for fresh water applications. Test results are measured at 522 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Aluminum Reagent Set (100 Tests), includes:	—	—	22420-00
(1) AluVer® 3 Aluminum Reagent Powder Pillow	1	100/pkg	14290-99
(1) Ascorbic Acid Powder Pillow	1	100/pkg	14577-99
(1) Bleaching 3 Reagent Powder Pillow	1	100/pkg	14294-49
Hydrochloric Acid, 6.0 N	varied	500 mL	884-49
Water, deionized	varied	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated mixing, 50-mL, with glass stopper	1	each	1896-41
Sample Cell, 1-inch square, 10-mL matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Aluminum Standard Solution, 100-mg/L as Al^{3+}	100 mL	14174-42
Aluminum Standard Solution, 10-mL Voluette® Ampule, 50-mg/L as Al	16/pkg	14792-10

Optional Reagents

Description	Cat. No.
m-Nitrophenol Indicator Solution	2476-32
Nitric Acid Solution, 1:1	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	2450-26
Sulfuric Acid Standard Solution, 5.25 N	2449-32

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Updated November 2005 Edition 1

Aluminum

Method 8326

Eriochrome Cyanine R Method[1]

Powder Pillows

(0.002 to 0.250 mg/L Al^{3+})

Scope and Application: For water

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

Before starting the test:

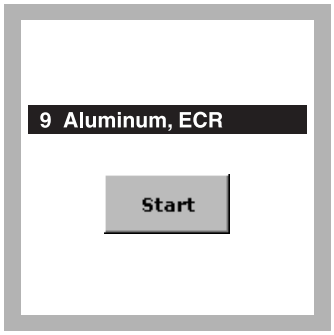
Clean all glassware with 6.0 N HCl and deionized water before use to remove contaminants from the glass.
Check the sample temperature. It must be between 20–25 °C (68 –77 °F) for accurate results.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

Collect the following items:	**Quantity**
ECR Reagent Powder Pillow	1
ECR Masking Reagent Solution	1 drop
Hexamethylene-tetramine Buffer Reagent Powder Pillow	1
25-mL graduated mixing cylinder with glass stopper	1
Sample cells, 1-inch square, 10 mL	2

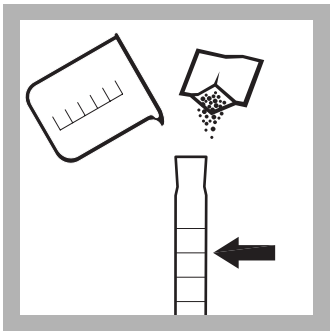
Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows — Method 8326

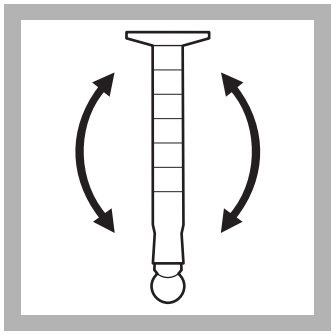
1. Press **STORED PROGRAMS**.



2. Select the test.



3. Fill the 25-mL mixing cylinder to the 20-mL mark with sample. Add one ECR Reagent Powder Pillow for 20-mL sample size.

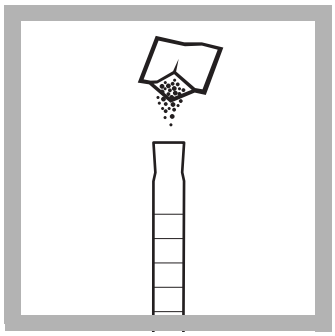


4. Insert the stopper. Invert several times to completely dissolve powder.

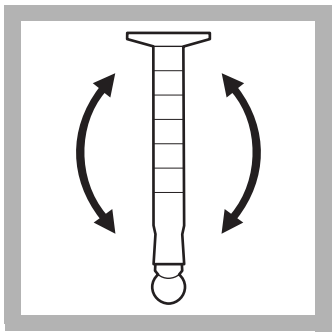
Undissolved reagent will cause inconsistent results.

5. Press **TIMER>OK**.

A 30-second reaction period will begin.

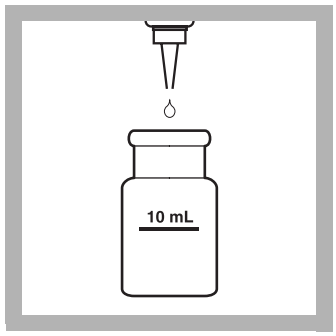


6. After the timer expires, add one Hexamethylene-tetramine Buffer Reagent powder pillow.

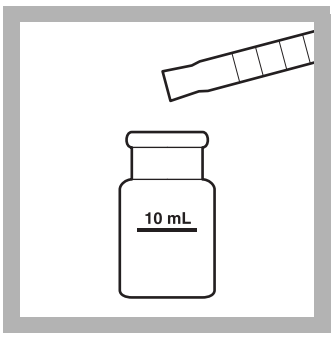


7. Insert the stopper. Invert several times to dissolve powder.

A red-orange color will develop if aluminum is present.

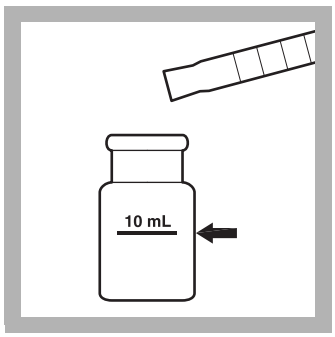


8. Blank Preparation: Put one drop of ECR Masking Reagent Solution into a clean square sample cell.



9. Pour 10 mL from the mixing cylinder into the blank cell. Swirl to mix.

The solution will begin to turn yellow.

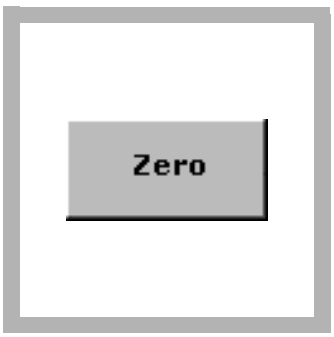


10. Prepared Sample: Fill a second square sample cell to the 10-mL mark with the remaining solution in the cylinder.

11. Press **TIMER>OK**.

A 5-minute reaction period will begin.

12. Within five minutes after the timer expires, wipe the blank then insert it into the cell holder with the fill line facing right.



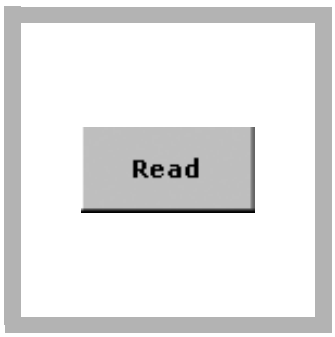
13. Press **ZERO**.

The display will show:

0.004 mg/L Al^{3+}

This test uses a non-zero intercept for the calibration curve.

14. Immediately wipe and dry the prepared sample then insert it into the cell holder with the fill line facing right.



15. Press **READ**.

Results are in mg/L Al^{3+}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 62 mg/L as $CaCO_3$
Alkalinity	Greater than 750 mg/L as $CaCO_3$
Ca^{2+}	Greater than 1000 mg/L as $CaCO_3$
Cl^-	Greater than 1000 mg/L as $CaCO_3$
Cr^{6+}	0.2 mg/L (error is –5% of reading)
Cu^{2+}	2 mg/L (error is –5% of reading)
Fe^{2+}	Greater than 4 mg/L (error is positive and equals mg/L Fe2+ x 0.0075)
Fe^{3+}	Greater than 4 mg/L (error is positive and equals mg/L Fe2+ x 0.0075)
F^-	See Table 2 on page 4.
Hexameta phosphate	0.1 mg/L as PO_4^{3-} (error is –5% of reading)
Mg^{2+}	Greater than 1000 mg/L as $CaCO_3$
Mn^{2+}	Greater than 10 mg/L
NO_2^-	Greater than 5 mg/L
NO_3^-	Greater than 20 mg/L
pH	2.9–4.9 or 7.5–11.5. A sample pH between about 4.9 and 7.5 causes dissolved aluminum to partially convert to colloidal and insoluble forms. This method measures much of that hard-to-detect aluminum without any pH adjusting pretreatment as is necessary in some other methods.
PO_4^{3-} (ortho)	4 mg/L (error is –5% of reading)
Polyphosphate	See procedure below.
SO_4^{2-}	Greater than 1000 mg/L
Zn^{2+}	Greater than 10 mg/L

Polyphosphate interference can be reduced by converting polyphosphate to orthophosphate by the following steps:

a. Rinse a 50-mL graduated mixing cylinder and a 125-mL Erlenmeyer flask containing a magnetic stir bar with 6 N hydrochloric acid*. Rinse again with deionized water. This will remove any aluminum present.

Note: *Rinse two Erlenmeyer flasks if a reagent blank is used; see* step b *below.*

b. Measure 50 mL deionized water into the 125-mL Erlenmeyer flask using the graduated cylinder. This is the reagent blank. Because of the test sensitivity, this step must be done only when any of the reagents used in the following pretreatment are replaced—even if the new reagent has a matching lot number. When the pretreated sample has been analyzed, correct for the aluminum concentration of the reagent blank by pressing **OPTIONS>MORE**, then **REAGENT BLANK**. Press **ON**. Enter the reagent blank value and press **OK**.

c. Measure 50 mL sample into the 125-mL Erlenmeyer flask using the graduated cylinder. Use a small amount of deionized water to rinse the cylinder contents into the flask.

d. Add 4.0 mL of 5.25 N Sulfuric Acid Standard Solution*.

* See Optional Reagents on page 6 for reorder information.

e. Use a combination hot plate/stirrer to boil and stir the sample for at least 30 minutes. Add deionized water as needed to maintain a sample volume of 20-40 mL. Do not boil dry.

f. Cool the solution to near room temperature.

g. Add 2 drops of Bromphenol Blue Indicator Solution*.

h. Add 1.5 mL of 12.0 N Potassium Hydroxide Standard Solution* using the calibrated, plastic dropper provided. Swirl to mix. The solution color should be yellow or green but not purple. If the color is purple, begin with step a again using an additional 1 mL Sulfuric Acid Standard Solution in step d.

i. While swirling the flask, add 1.0 N Potassium Hydroxide Solution*, a drop at a time, until the solution turns a dirty green color.

j. Pour the solution into the graduated cylinder. Rinse the flask contents into the graduated cylinder with deionized water to bring the total volume to 50 mL.

k. Use this solution in step 3 of the ECR method.

Fluoride interference can be corrected by using Table 2.

Example:
If the fluoride concentration is known to be 1.00 mg/L F^- and the ECR method gives a reading of 0.060 mg/L aluminum, what is the true mg/L aluminum concentration?

Intermediate values can be found by interpolation. Do not use correction graphs or charts found in other publications.

Answer: 0.183 mg/L

Table 2 Fluoride Concentration (mg/L)

(mg/L)	0.00	0.20	0.40	0.60	0.80	1.00	1.20	1.40	1.60	1.80	2.00
0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
0.010	0.010	0.019	0.030	0.040	0.052	0.068	0.081	0.094	0.105	0.117	0.131
0.020	0.020	0.032	0.046	0.061	0.077	0.099	0.117	0.137	0.152	0.173	0.193
0.030	0.030	0.045	0.061	0.077	0.098	0.124	0.146	0.166	0.188	0.214	0.243
0.040	0.040	0.058	0.076	0.093	0.120	0.147	0.174	0.192	0.222	—	—
0.050	0.050	0.068	0.087	0.109	0.135	0.165	0.188	0.217	—	—	—
0.060	0.060	0.079	0.100	0.123	0.153	0.183	0.210	0.241	—	—	—
0.070	0.070	0.090	0.113	0.137	0.168	0.201	0.230	—	—	—	—
0.080	0.080	0.102	0.125	0.152	0.184	0.219	—	—	—	—	—
0.090	0.090	0.113	0.138	0.166	0.200	0.237	—	—	—	—	—
0.100	0.100	0.124	0.150	0.180	0.215	—	—	—	—	—	—
0.120	0.120	0.146	0.176	0.209	0.246	—	—	—	—	—	—
0.140	0.140	0.169	0.201	0.238	—	—	—	—	—	—	—
0.160	0.160	0.191	0.226	—	—	—	—	—	—	—	—
0.180	0.180	0.213	—	—	—	—	—	—	—	—	—
0.200	0.200	0.235	—	—	—	—	—	—	—	—	—
0.220	0.220	—	—	—	—	—	—	—	—	—	—
0.240	0.240	True Aluminum Concentration (mg/L) Al									

* See Optional Reagents on page 6 for reorder information.

Sample Collection, Storage, and Preservation

Collect samples in clean glass or plastic containers. Preserve samples by adjusting the pH to 2 or less with concentrated nitric acid* (about 1.5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 2.9–4.9 with 12.0 N Potassium Hydroxide Standard Solution* and/or 1 N Potassium Hydroxide Solution*. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

Standard Solution Method
Prepare a 0.100 mg/L aluminum standard solution as follows:

1. Using Class A glassware, pipet 1.00 mL of Aluminum Standard Solution.

   100-mg/L as Al^{3+}, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the aluminum procedure as described above.

   OR

   Add 2.0 mL of solution from an Aluminum Voluette® Ampule Standard Solution (50-mg/L as Al) into a 1000-mL volumetric flask. Dilute to volume with deionized water. Prepare this solution daily. Perform the aluminum procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Eriochrome Cyanine R combines with aluminum in a sample to produce an orange-red color. The intensity of color is proportional to the aluminum concentration. Test results are measured at 535 nm.

*See Optional Reagents on page 6.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Aluminum Reagent Set (100 Tests), includes:	—	—	26037-00
ECR Reagent Powder Pillows	1	100/pkg	26038-49
Hexamethylenetetramine Buffer Reagent Powder Pillows	1	100/pkg	26039-99
ECR Masking Reagent Solution	1 drop	25 mL SCDB	23801-23

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated mixing, 25-mL, with glass stopper	1	each	1896-40
Sample Cell, 1-inch Square, 10 mL	2	2/pkg	24954-02
Thermometer, –10 to 110 °C	—	each	1877-01

Recommended Standards

Description	Unit	Cat. No.
Aluminum Standard Solution, 100-mg/L as Al^{3+}	100 mL	14174-42
Aluminum Standard Solution, 10-mL Voluette® Ampule, 50-mg/L as Al	16/pkg	14792-10
Water, deionized	4 L	272-56

Optional Reagents

Description	Unit	Cat. No.
Bromphenol Blue Indicator Solution	100 mL	14552-32
Hydrochloric Acid, 6.0 N	500 mL	884-49
Nitric Acid 1:1	500 mL	2540-49
Potassium Hydroxide Standard Solution, 12.0 N	100 mL	230-32
Potassium Hydroxide Solution, 1.0 N	50 mL	23144-26
Sodium Hydroxide Standard Solution, 5.0 N	50 mL	2450-26
Sulfuric Acid Standard Solution, 5.25 N	100 mL	2449-32

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Updated November 2005 Edition 1

Aluminum

Method 10215 Chromazurol S Method
TNTplus™ 848 (0.02 to 0.50 mg/L Al)

Scope and Application: For drinking water, surface water, swimming pool water, wastewater, and process analysis.

Test Preparation

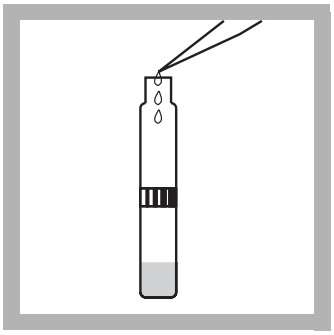
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read the Safety Advice and Expiration Date on the package.
Recommended sample pH is 2.5–3.5.
Recommended sample and reagent temperature is 20–23 °C (68–73.4 °F).
Recommended reagent storage is 15–25 °C (59–77 °F).
If test is not performed at the recommended temperature an incorrect result may be obtained.
A higher pH causes precipitation or complexing of the aluminum so that low-bias results are obtained. If necessary the pH of the sample must be adjusted by adding a small amount of nitric acid.
TNTplus methods are activated directly from the Main Menu screen when the prepared sample vial is inserted into the sample cell holder.

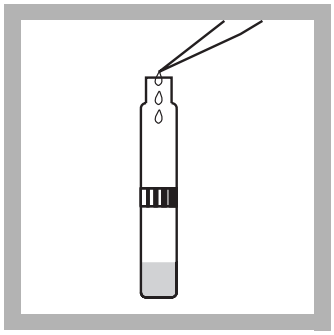
Collect the following items:	**Quantity**
Aluminum TNT848 Reagent Set	1
Light Shield	1
Pipettor, variable 1–5 mL	1
Pipettor tips for 1–5 mL pipettor	1

Note: *Reorder information for consumables and replacement items is on page 4.*

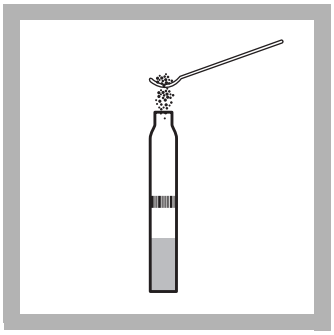
TNTplus Method 10215



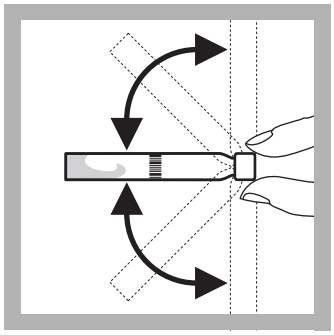
1. Pipet 2.0 mL of Solution A into the vial.



2. Pipet 3.0 mL of sample into the vial.



3. Add one level spoonful of Reagent B to the vial.



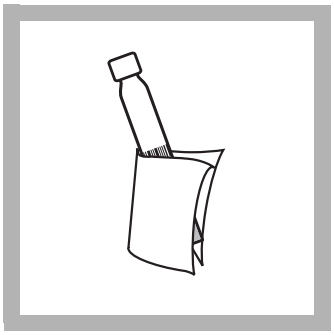
4. Cap and invert the vial 2–3 times until no more streaks can be seen in the solution.

5. Wait 25 minutes.

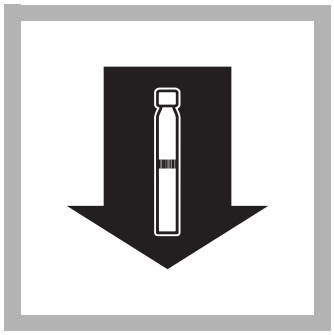
Install the Light Shield in Cell Compartment #2.

6. Insert the Zero vial from the sample vial lot into the sample cell holder.

The instrument reads the barcode, then selects the method and sets the instrument to zero. The instrument displays L1 when zeroing is complete.



7. Thoroughly clean the outside of the prepared sample vial.



8. Insert the prepared vial into the cell holder. The instrument reads the barcode and reads the sample.

Results are in mg/L Al.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 6. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

If the Sample Blank value falls within the allowable range, this value will be used to correct the result automatically. The instrument will subtract the Sample Blank from the uncorrected result. Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Higher concentrations of heavy metals than those given, as well as fluoride, phosphate and relatively rare elements such as e.g. beryllium, thorium, titanium, zirconium and vanadium interfere with the determination. Aluminum oxide hydrates and hydroxide are only partially determined.

Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
Mg^{2+}, K^{+}, Na^{+}, NH_4^{+}, Cl^{-}, NO_3^{-}, SO_4^{2-}, Ca^{2+}	500 mg/L
Ag^{+}, Mn^{2+}	100 mg/L
Cd^{2+}, Co^{2+}, Ni^{2+}, Sn^{2+}, Pb^{2+}, PO_4^{3-}	50 mg/L
Cu^{2+}, Hg^{2+}	10 mg/L
Fe^{2+}, Fe^{3+}, Zn^{2+}, Si^{4+}	5 mg/L
Cr^{3+}, Cr^{6+}	0.5 mg/L
F^{-}	0.1 mg/L

Sample Collection, Preservation, and Storage

Collect samples in an acid-washed glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Adjust the pH to between 2.5 and 3.5 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Prepare a 0.4 mg/L aluminum standard solution by pipetting 1.0 mL of a 100 mg/L aluminum standard solution into a 250 mL volumetric flask.

2. Dilute to volume with deionized water. Prepare this solution daily. Use 3.0 mL of this standard in place of the sample in the procedure.

Summary of Method

Chromazurol S forms a green colored lake with aluminum in weakly acidic acetate-buffered solutions. The amount of color formed is directly proportional to the amount of aluminum present in the sample. Test results are measured at 620 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Aluminum TNT 848 Reagent Set	1	24/pkg	TNT848

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 1–5 mL	1	each	27951-00
Pipettor Tips, for 27951-00 pipettor	1	100/pkg	27952-00

Recommended Reagents and Standards

Description	Unit	Cat. No.
Aluminum Standard Solution, 100 mg/L	100 mL	14174-42
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	2450-26
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, Sampling, low density poly, w/cap, 500 mL	12/pk	20870-79
Flask, volumetric 250 mL	each	14574-46
Pipet, volumetric 1.0 mL	each	14515-35
Test Tube Rack, 13-mm vials	each	24979-00
Vials, sample blank	—	TNT919

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Updated November 2005 Edition 1

Arsenic

★Method 8013 — Silver Diethyldithiocarbamate Method[1] (0 to 0.200 mg/L As)

Scope and Application: For water, wastewater, and seawater; distillation is required; USEPA accepted[2] for reporting for drinking and wastewater analysis (digestion required)

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

[2] Procedure is equivalent to USEPA Method 206.4 for wastewater and Standard Method 3500-As for drinking water analysis.

Test Preparation

Before starting the test:

Create a user-entered program for arsenic. See step 1 and User Programming on page 5.
Prepare the arsenic absorber solution as directed in Reagent Preparation on page 4.
Perform a user-entered calibration for each new lot of arsenic absorber solution. See Calibration on page 5. Some variations of the calibration procedure are possible.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

Collect the following items:	**Quantity**
Apparatus as listed on page 6.	—
Arsenic Standard Solution, 1000-mg/L As	varies
Hydrochloric Acid, ACS	25 mL
Lead Acetate Solution, 10%	1 mL
Potassium Iodide Solution, 20%	3 mL
Pyridine, ACS	50 mL
Sample Cells, 1-inch square, 25 mL, matched pair with stopper	2
Silver Diethyldithiocarbamate	1 g
Stannous Chloride Solution	1 mL
Water, deionized	varies
Zinc, 20-mesh, ACS	6 g

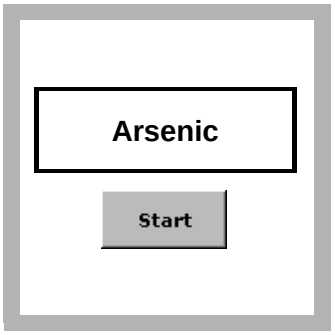
Note: *Reorder information for consumables and replacement items is on page 6.*

Important Note: *The arsenic absorber in this test is a silver solution in pyridine. Both silver (D011) and pyridine (D038) are regulated by the Federal RCRA as hazardous waste. In addition, the cotton ball soaked in lead acetate (D008) solution is a hazardous waste. These materials should not be poured down the drain. Refer to a current MSDS sheet for proper disposal.*

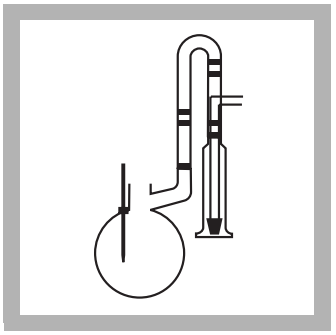
Silver Diethyldithiocarbamate Method 8013

1. Perform the User Programming procedure on page 5. Make note of the program number.

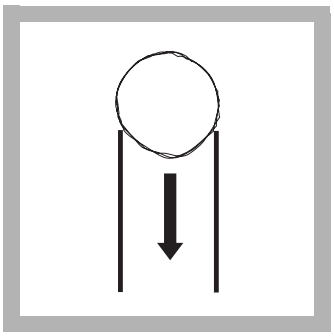
2. To run the test, press **USER PROGRAMS**.



3. Select the test.



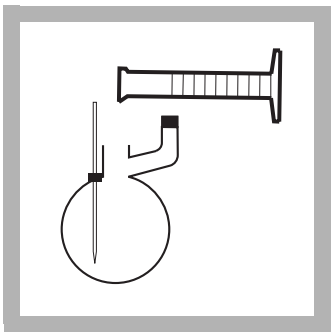
4. Prepare the Distillation Apparatus for arsenic recovery. See the *Distillation Manual* for assembly instructions. Place it under a fume hood to vent toxic fumes.



5. Dampen a cotton ball with 10% Lead Acetate Solution. Insert it in the gas scrubber. Be certain that the cotton seals against the glass.

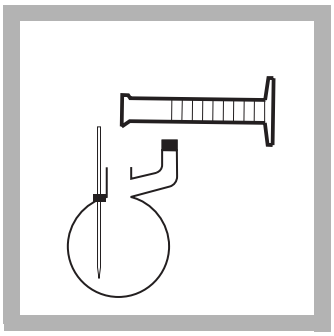
6. Using a graduated cylinder, pour 25-mL of prepared arsenic absorber solution (Reagent Preparation on page 4) into the cylinder/gas bubbler assembly.

Attach it to the distillation apparatus.

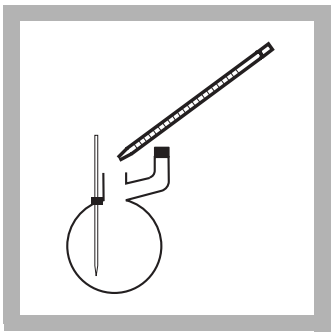


7. Using a graduated cylinder, pour 250 mL of sample into the distillation flask.

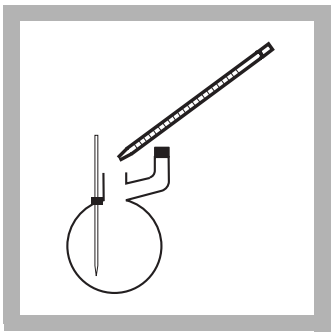
8. Turn on the power switch. Set the stir control to 5. Set the heat control to 0.



9. Using a graduated cylinder, add 25 mL of Hydrochloric Acid, ACS, to the distillation flask.



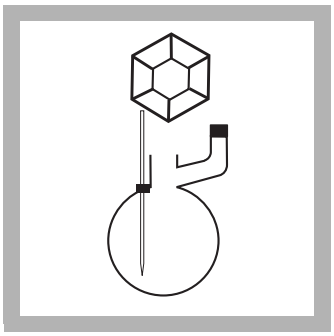
10. Use a serological pipet to add 1 mL of Stannous Chloride Solution to the flask.



11. Use a serological pipet to add 3 mL of Potassium Iodide Solution to the flask. Cap.

12. Press **TIMER>OK**.

A 15-minute reaction period will begin.



13. When the timer expires, add 6.0 g of 20-mesh zinc to the flask. **Cap immediately.**

14. Set the heat control to 3.

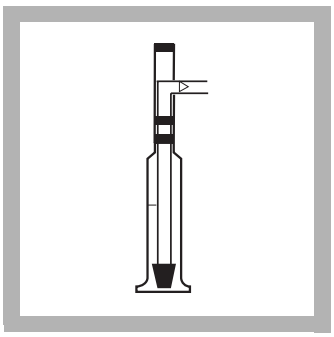
15. Press **TIMER>OK**.

A second 15-minute reaction period will begin.

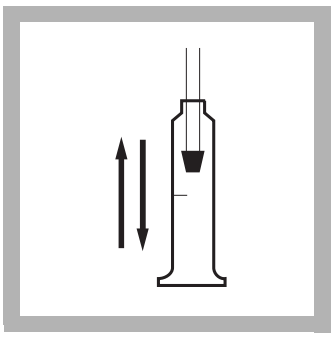
16. When the timer expires, set the heat control to 1.

17. Press **TIMER>OK**.

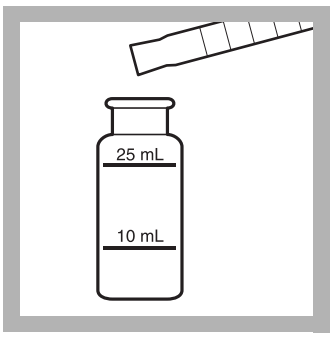
A third 15-minute reaction period will begin.



18. When the timer expires, turn off the heater. Remove the cylinder/gas bubbler assembly as a unit.

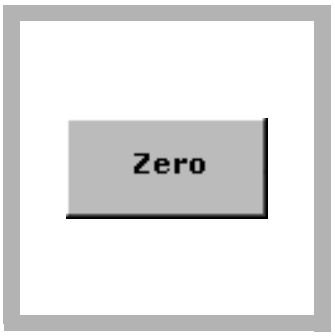


19. Rinse the gas bubbler by moving it up and down in the arsenic absorber solution.

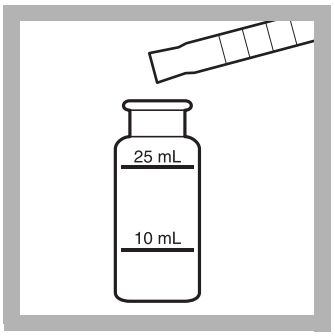


20. Blank Preparation: Fill a dry, 25-mL square sample cell with untreated arsenic absorber solution. Stopper.

21. Wipe the blank and insert it into the cell holder with the fill line facing right.



22. Press **ZERO**. The display will show the intercept as calculated from the user-entered calibration curve. This will probably be a non-zero intercept.



23. Prepared Sample: Pour the reacted arsenic absorber sample into a sample cell. If the solution volume is less than 25 mL, add pyridine to bring the volume to exactly 25 mL. Stopper.

24. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ** to display the results.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Antimony Salts	May interfere with color development.

Sample Collection, Storage, and Preservation

Collect samples in acid washed glass or plastic bottles. Adjust the pH to 2 or less with sulfuric acid (about 2 mL per liter)*. Preserved samples may be stored up to six months at room temperature. Correct the test result for volume additions.

Reagent Preparation

Prepare the arsenic absorber solution as follows:

1. Weigh 1.00 g of silver diethyldithiocarbamate on an analytical balance.
2. Transfer the powder to a 200-mL volumetric flask. Dilute to volume with pyridine. **Use pyridine only in a fume hood and wear chemical resistant gloves.** Read the MSDS before using pyridine.
3. Mix well to dissolve. Store the reagent, tightly sealed, in an amber bottle. The reagent is stable for one month if stored in this manner. Larger volumes of reagent can be prepared if the reagent is used within one month.

* See Optional Reagents and Apparatus on page 7.

Calibration

Standard Preparation
Perform a new calibration for each lot of arsenic absorber solution.

1. Prepare a 10.0-mg/L arsenic working standard by pipetting 10.0 mL of Arsenic Standard Solution, 1000 mg/L As into a 1000-mL volumetric flask.

2. Dilute to volume with deionized water.

3. Into three different 500-mL volumetric flasks, pipet 1.0, 2.0, and 10.0 mL of the 10.0 mg/L As stock solution using Class A glassware.

4. Dilute to the mark with deionized water and mix thoroughly. These standards have concentrations of 0.02, 0.04 and 0.20 mg/L As.

User Programming

1. Press **USER PROGRAMS** on the main menu.

2. If you have not performed an arsenic calibration before, press **PROGRAM OPTIONS** and **NEW**. Key any available program number (950–999) to use for arsenic testing. Press **OK**.

3. Fill in the appropriate fields using the touch screen when the field is highlighted. Use the alphanumeric keys to name the User Program **ARSENIC**. Press **NEXT** to move to the next screen. Set up the rest of the parameters as follows:

   - Program Type: Single Wavelength
   - Units: mg/L
   - Concentration Resolution: 0.001
   - Chemical Form: As
   - Wavelength: 520 nm
   - Calibration: Read Standards

4. After entering Read Standards, press **NEXT>EXIT**. Fill in the appropriate fields for each of the following. Use the touch screen to activate the parameter and press **EDIT** to enter the data entry screen. Set up the rest of the parameters as follows:

   - Timer1: 15 minutes
   - Timer2: 15 minutes
   - Timer3: 15 minutes
   - Upper Limit: 0.220 mg/L
   - Lower Limit: –0.020 mg/L

5. Press **CALIBRATION: C = A + BA**. Press **EDIT**.

6. The Read Standards will be indicated. Enter the standards concentration values to be used to perform the calibration: 0.00, 0.02, 0.04, and 0.20. To enter the concentration values press **+** and enter the value followed by **OK** for each concentration value.

7. After the values are entered, press the **UP** arrow four times to move the cursor to the 0.00 concentration line.

8. Insert the 25-mL sample cell containing only unreacted arsenic absorber solution into the cell holder. Press **ZERO.**

9. Press the **DOWN** arrow once to move to the next concentration. Insert the prepared sample in the cell holder. Press **READ** to accept the absorbance value. Repeat steps for each standard.

10. Press **GRAPH**. Make sure **FORCE ZERO** is off.

11. If the graph is acceptable press **DONE>EXIT**.

12. "Store Program?" will appear on the display. Press **YES**.

The program is ready for use.

Some variations of the calibration procedure are possible. See the user manual for details.

Summary of Method

Arsenic is reduced to arsine gas by a mixture of zinc, stannous chloride, potassium iodide, and hydrochloric acid in a specially equipped distillation apparatus. The arsine is passed through a scrubber containing cotton saturated with lead acetate and then into an absorber tube containing silver diethyldithiocarbamate in pyridine. The arsenic reacts to form a red complex which is read colorimetrically. This procedure requires a manual calibration. Test results are measured at 520 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Arsenic Standard Solution, 1000-mg/L As	varies	100 mL	14571-42
Hydrochloric Acid, ACS	25 mL	500 mL	134-49
Lead Acetate Solution, 10%	1 mL	100 mL	14580-42
Potassium Iodide Solution, 20%	3 mL	100 mL	14568-42
Pyridine, ACS	50 mL	500 mL	14469-49
Silver Diethyldithiocarbamate	1 g	25 g	14476-24
Stannous Chloride Solution	1 mL	100 mL	14569-42
Water, deionized	varies	4 liters	272-56
Zinc, 20-mesh, ACS	6 g	454 g	795-01

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Balance, analytical, SL 3000, 100–240 V	1	each	28018-00
Balls, cotton	1	100/pkg	2572-01
Boat, weighing, 8.9-cm square	2	500/pkg	21790-00
Bottle, amber, 237-mL, glass	1	6/pkg	7144-41
Cap, polypropylene, for amber bottle	1	6/pkg	21667-06
Cylinder, graduated, 25-mL	2	each	508-40
Cylinder, graduated, 250-mL	1	each	508-46
Distillation Apparatus, Arsenic Accessories	1	set	22654-00
Distillation Apparatus, General Purpose Accessories	1	set	22653-00
Flask, volumetric, Class A, 1000-mL, with glass stopper	1	each	14574-53
Flask, volumetric, Class A, 200-mL	1	each	14574-45

Required Apparatus (continued)

Description	Quantity/Test	Unit	Cat. No.
Flask, volumetric, Class A, 500-mL	6	each	14574-49
Pipet Filler, safety bulb	1	each	14651-00
Pipet, serological, 5-mL	2	each	532-37
Pipet, volumetric, Class A, 1.00-mL	2	each	14515-35
Pipet, volumetric, Class A, 2.00-mL	1	each	14515-36
Pipet, volumetric, Class A, 4.00-mL	1	each	14515-04
Pipet, volumetric, Class A, 6.00-mL	1	each	14515-06
Pipet, volumetric, Class A, 8.00-mL	1	each	14515-08
Pipet, volumetric, Class A, 10.00-mL	1	each	14515-38
Sample Cells, 1-inch square, 25 mL, matched pair with stopper	2	2/pkg	26126-02
Select one based on available voltage:			
Distillation Apparatus Heater, 115 VAC, 60 Hz	1	each	22744-00
Distillation Apparatus Heater, 230 VAC, 50 Hz	1	each	22744-02

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing, 25-mL	1896-40
Sulfuric Acid, 1.00 N	1270-32
Gloves, Chemical Resistant, size 9[1]	24101-04

[1] Other sizes available.

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Updated November 2005 Edition 1

Atrazine

Method 10050

Immunoassay Method[1]

Scope and Application: For water

[1] This test is semi-quantitative. Results are expressed as greater or less than the threshold value used.

Test Preparation

This method analyzes for Atrazine in water. Sample calibrators and reagents are added to cuvettes coated with Atrazine-specific antibodies. The color that develops is then measured and compared with the color measurements of the calibrators. The test requires about 30 minutes for complete analysis. As many as 20 cuvettes (18 samples and 2 calibrators) can be run simultaneously.

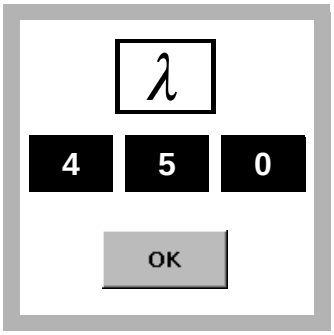
Before starting the test:

Read the entire procedure before starting. Identify and make ready all the necessary reagents, cuvettes, and other apparatus before beginning the analysis.
Timing is critical; follow instructions carefully.
A consistent technique when mixing the cuvettes is critical to this test. The best results come from using the cuvette rack and mixing as described in Using the 1-cm MicroCuvette Rack on page 4. Cuvettes can be mixed individually, but test results may not be as consistent.
Handle the cuvettes carefully. Scratches on the inside or outside may cause erroneous results. Carefully clean the outside of the cuvettes with a clean absorbent cloth or tissue before placing them into the instrument.
Antibody cuvettes and enzyme conjugate are made in matched lots. Do not mix reagent lots.
To avoid damaging the Color Developing Solution, do not expose it to direct sunlight.
The cuvette rack is designed to be inverted with the cuvettes in place. This is especially helpful when running many samples at once; the cuvettes can remain in the rack and be processed together until they are read in the spectrophotometer.
Twenty Antibody Cuvettes are provided with each reagent set. One Antibody Cuvette will be used for each calibrator and each sample. Cuvettes are not reusable.
Protective nitrile gloves are recommended for this procedure.

Collect the following items:	**Quantity**
Atrazine Reagent Set	1
Caps, flip spout	1
Marker, laboratory	1
Rack, for 1-cm Micro Cuvettes	1
Wipes, disposable	1
Pipet, TenSette®, 0.1–1.0 mL	1
Pipet Tips, for TenSette Pipet 19700-01	1

Note: *Reorder information for consumables and replacement items is on page 7.*

Immunoassay for Water

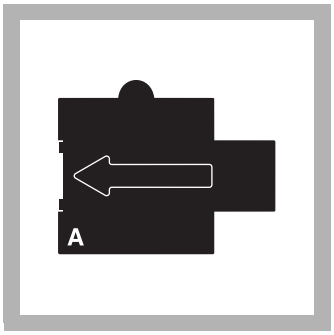


1. Press

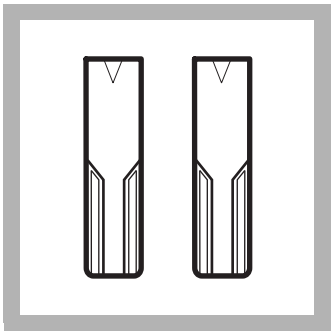
SINGLE WAVELENGTH

Press **OPTIONS** and the λ button. Enter

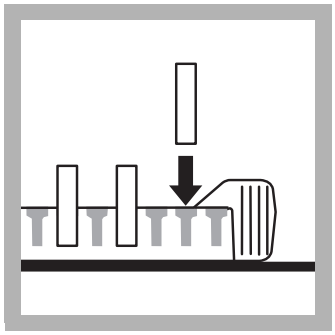
450 nm and press **OK.**



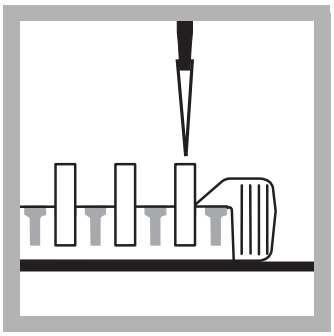
2. Insert Adapter A.



3. Label an Antibody Cuvette for each calibrator and each sample to be tested.

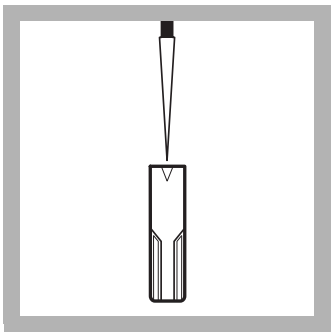


4. Insert the cuvettes into the rack snugly.



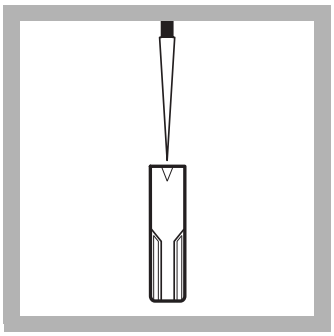
5. Pipet 0.5 mL of each calibrator into the appropriately labeled cuvette.

Use a new pipette tip for each calibrator.

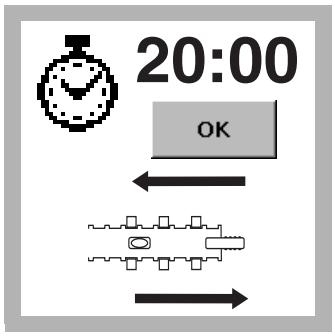


6. Pipet 0.5 mL of each sample to be tested into the appropriately labeled cuvette.

Use a new pipette tip for each sample.



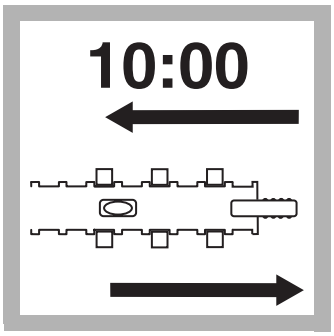
7. Immediately pipet 0.5 mL of Atrazine Enzyme Conjugate into each cuvette.



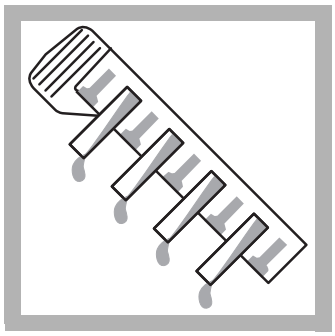
8. Press **OPTIONS**. Press **TIMER**. Enter 20:00 minutes and press **OK.**

A 20-minute reaction time will begin.

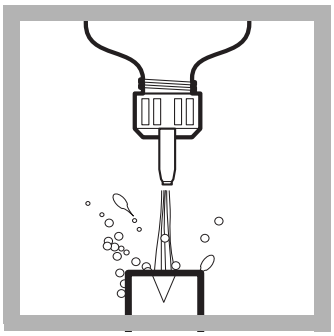
Immediately mix the contents of the cuvettes for 30 seconds (Using the 1-cm MicroCuvette Rack on page 4.)



9. After 10 minutes mix the contents of the rack for 30 seconds (Using the 1-cm MicroCuvette Rack on page 4.)



10. At the end of the 20-minute period, discard the contents of all the cuvettes into an appropriate waste container.

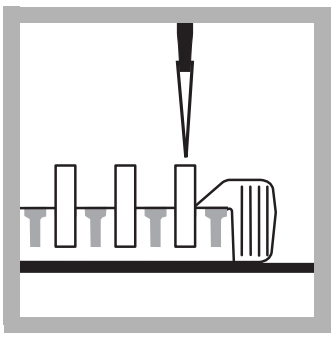


11. Wash each cuvette forcefully and thoroughly four times with deionized water. Empty the rinse water into the waste container.

Ensure that most of the water is drained from the cuvettes by turning the cuvettes upside down and tapping them lightly on a paper towel.

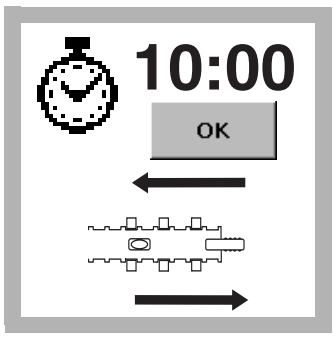
Color Development

Important Note: *Timing is critical. Follow instructions carefully.*



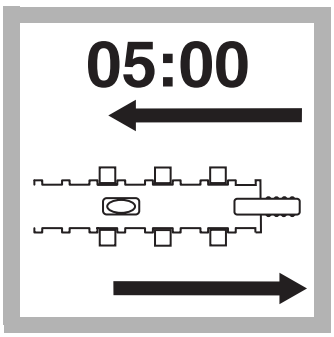
12. With the cuvettes still held snugly in the rack, pipet 0.5 mL of Color Developing Solution into each Antibody Cuvette.

Use a new pipette tip for each cuvette.



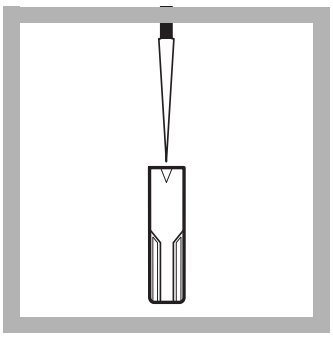
13. Press **OPTIONS**. Press **TIMER**. Enter 10:00 minutes and press **OK.**

A reaction period will begin. Mix, using the instructions on page 4.



14. After 5 minutes, mix the contents of the rack a second time for a period of 30 seconds using the same technique.

Solutions will turn blue in some or all of the cuvettes.

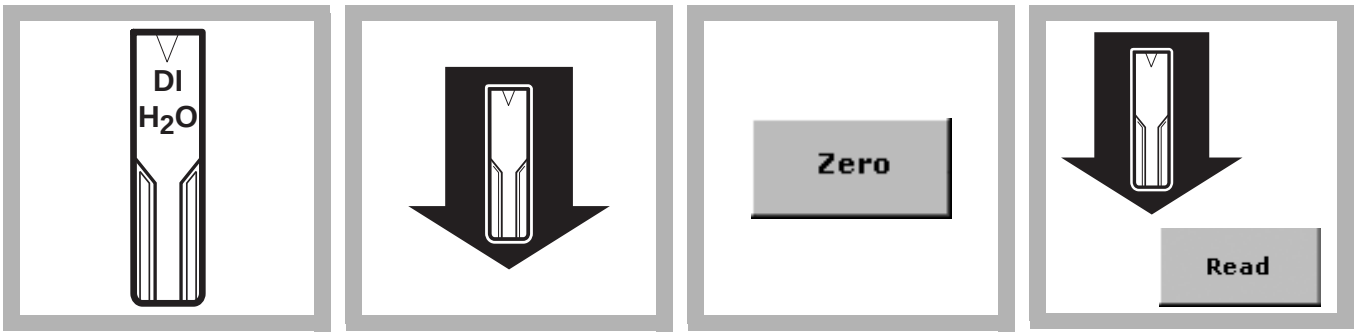


15. At the end of the 10-minute reaction period, pipette 0.5 mL of Stop Solution into each cuvette in the same order as the Color Developing Solution was added in step 12. Use the same pipette tip repeatedly for this step.

Slide the rack for 20 seconds (Using the 1-cm MicroCuvette Rack on page 4.)

Blue solutions will turn yellow with the addition of the Stop Solution.

Measuring the Color



16. Label and fill a Zeroing Cuvette with deionized water. Wipe the outside of all the cuvettes with a tissue to remove water, smudges, and fingerprints.

17. Insert the filled Zeroing Cuvette into the cell holder—arrow pointing to the right.

Orient the arrow in the same direction for all cuvettes.

18. Press **ZERO**.

The display will show:

0.000 Abs

19. Insert the first calibrator into the cell holder.

Press **READ**. The display will give an absorbance reading. Record the results for each calibrator and sample.

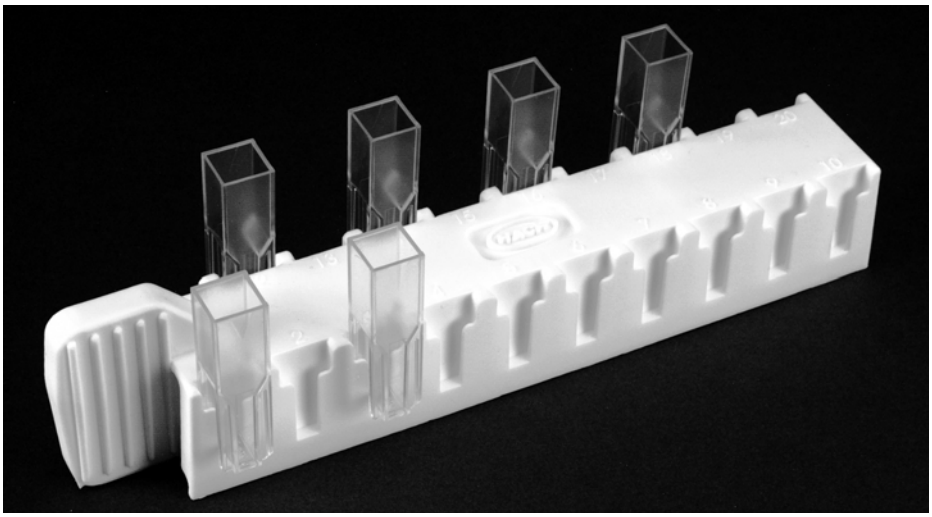
Repeat this step for all remaining calibrators and samples.

See Interpreting and Reporting Results on page 5 for help with interpretation of results.

Using the 1-cm MicroCuvette Rack

The MicroCuvette rack (Figure 1) has been designed specifically to aid in achieving precise and accurate results when using the immunoassay technique to analyze several samples at the same time.

Figure 1 The 1-cm MicroCuvette Rack



Loading the Rack—The cuvette rack is designed so that it may be inverted with the cuvettes in place. Identify each cuvette with a sample or calibrator number and insert all the cuvettes in the rack before beginning the procedure. Fit the cuvettes snugly into the rack, but do not force them or they may be difficult to remove and their contents may spill. The cuvettes should remain in place when the rack is inverted and tapped lightly.

Mixing—Set the rack on a hard, flat surface that is at least twice the length of the rack. Hold the rack by one end and vigorously slide it back and forth along its long axis for 30 seconds. The rack should move through a distance equal to its own length in each direction.

Interpreting and Reporting Results

There is an inverse relationship between the concentration of Atrazine and the reading. In other words, the higher the reading, the lower the concentration of Atrazine.

Table 1 Relative Atrazine Concentration

If the sample reading is...	the sample Atrazine Concentration is...
…less than calibrator reading	…greater than the calibrator concentration
…greater than calibrator reading	…less than the calibrator concentration

Example
Readings:

0.5 ppb Atrazine Calibrator: **0.475 Abs**

3.0 ppb Atrazine Calibrator: **0.245 Abs**

Sample #1: **0.140 Abs**

Sample #2: **0.300 Abs**

Sample #3: **0.550 Abs**

Interpretation
Sample #1—Sample reading is less than the readings for both calibrators. Therefore the sample concentration of Atrazine is greater than both 0.5 ppb and 3.0 ppb Atrazine.

Sample #2—Sample reading is between the readings for the 0.5 ppb and 3.0 ppb Atrazine calibrators. Therefore the sample concentration of Atrazine is between 0.5 ppb and 3.0 ppb.

Sample #3—Sample reading is greater than the readings for both calibrators. Therefore the sample concentration of Atrazine is less than both 3.0 ppb and 0.5 ppb.

Storing and Handling Reagents

- Wear protective gloves and eyewear.
- When storing reagent sets for extended periods of time, keep them out of direct sunlight. Store reagents at a temperature of 4 °C when not in use.
- Keep the foil pouch containing the Antibody Cuvettes sealed when not in use.
- If Stop Solution comes in contact with eyes, wash thoroughly for 15 minutes with cold water and seek immediate medical help.

Sensitivity

The Atrazine immunoassay test cannot differentiate between certain triazines and metabolites, but it detects their presence to differing degrees. Table 2 shows the required concentration for selected chemicals. Table 3 shows compounds not detectalbe at 10,000 ppb.

Table 2 Required Concentrations for Selected Chemicals

Compound	Concentration to give a positive result at 3 ppb (in ppb)
Ametryne	1
Atrazine	3
Atrazine, de-ethylated	115
Atrazine, de-isopropyl	714
Cyanazine	460
Cyromazine	1200
Prometon	8
Prometryne	0.7
Propazine	2.3
Simetryne	5.4
Simazine	37
Terbuthylazine	91
Terbutryne	8.3

Table 3 Compounds not detectable at 10,000 ppb

Alachlor	2, 4-D
Aldicarb	Diaminoatrazine
Carbendazim	Melamine
Carbofuran	Metaolachlor

Sample Collection and Storage

Collect samples in a clean glass bottle. Do not pre-rinse the bottle with the sample. If the sample cannot be analyzed immediately, store the sample at 4 °C. Samples may be kept for as long as 14 days. Warm the samples to room temperature before analysis.

Summary of Method

Immunoassay tests use antigen/antibody reactions to test for specific organic compounds in water and soil. Atrazine-specific antibodies, attached to the walls of plastic cuvettes, selectively bind and remove Atrazine from complex sample matrices. A prepared sample and a reagent containing enzyme-conjugate molecules (analyte molecules attached to molecules of an enzyme) are added to the Antibody Cuvettes. During incubation, enzyme-conjugate molecules and Atrazine compete for binding sites on the antibodies. Samples with higher levels of analyte will have more antibody sites occupied by Atrazine and fewer antibody sites occupied by the enzyme-conjugate molecules.

After incubation, the sample and unbound enzyme conjugate are washed from the cuvette and a color-development reagent is added. The enzyme in the conjugate catalyzes the development of color. Therefore, there is an inverse relationship between color intensity and the amount of Atrazine in the sample. The resulting color is then compared with a calibrator to determine whether the Atrazine concentration in the sample is greater or less than the threshold levels. Test results are measured at 450 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Atrazine Reagent Set[1]	20 cuvettes	27627-00

[1] Immunoassay components are manufactured by Beacon Analytical Systems, Inc.

Required Apparatus

Description	Unit	Cat. No.
Adapter, 1-cm square cell	each	
Caps, flip spout	2/pkg	25818-02
Marker, laboratory	each	20920-00
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Rack, for 1-cm Micro Cuvettes	each	48799-00
Wipes, disposable	box	20970-00

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Atrazine Reagent Set	100 cuvettes	27627-10
Glasses, safety	each	27568-00
Gloves, disposable, nitrile, medium[1]	—	25505-02
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96

[1] Other sizes are available.

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Updated November 2005 Edition 1

Barium

★Method 8014 — Turbidimetric Method[1]

Powder Pillows — (2 to 100 mg/L)

Scope and Application: For water, wastewater, oil-field water, and seawater

[1] Adapted from Snell and Snell, *Colorimetric Methods of Analysis, Vol. II*, 769 (1959).

Test Preparation

Before starting the test:

Perform a standard curve adjustment or a new calibration for each new lot of reagent. See Standard Solutions on page 2 and Calibration Standard Preparation on page 3.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Filter highly colored or turbid water samples using a funnel[1] and filter paper[1]. Large amounts of color or turbidity will interfere and cause high readings.
Immediately after each test, clean the sample cell with soap, water, and a brush to prevent a film from forming inside the sample cell.

Collect the following items:	**Quantity**
BariVer® 4 Barium Reagent Powder Pillows	1
Sample cells, 1-inch square, 10-mL	2

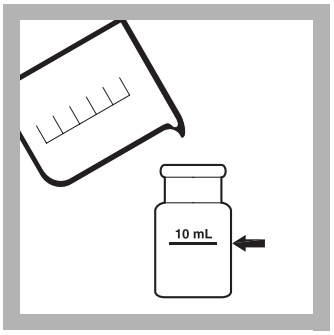
Note: *Reorder information for consumables and replacement items is on page 4.*

[1] See Optional Reagents and Apparatus on page 4.

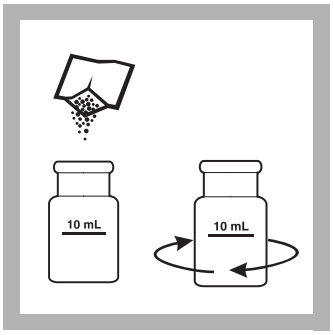
Powder Pillows — Method 8014

1. Press **STORED PROGRAMS**.

2. Select the test.



3. Fill a square sample cell with 10 mL of sample.



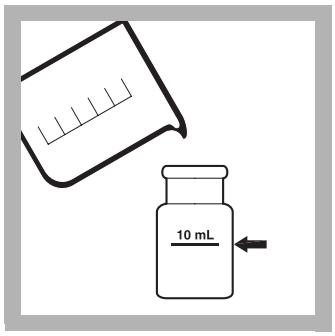
4. Prepared Sample: Add the contents of one BariVer® 4 Barium Reagent Powder Pillow to the cell.

If barium is present, a white turbidity will develop.

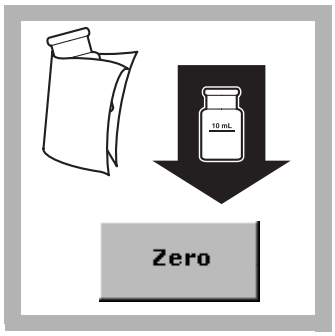
5. Press **TIMER>OK**.

A five-minute reaction period will begin.

Do not disturb the sample during the reaction period.



6. Blank Preparation: Fill another square sample cell with 10 mL of sample.



7. When the timer expires, wipe the blank and insert the blank into the cell holder with the fill line facing right.

Press **ZERO**. The display will show:

0.0 mg/L Ba^{2+}

8. Wipe and the prepared sample and insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Ba^{2+}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium	10,000 mg/L as $CaCO_3$
Magnesium	100,000 mg/L as $CaCO_3$
Silica	500 mg/L
Sodium Chloride	130,000 mg/L as NaCl
Strontium	Interferes at any level. If present, the total concentration between barium and strontium may be expressed as a PS (Precipitated by Sulfate). While this does not distinguish between barium and strontium, it gives an accurate indication of scaling tendency.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Sample Collection, Preservation, and Storage

Collect samples in an acid cleaned glass or plastic container. Adjust the pH to 2 or less with nitric acid* (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 5 with 5.0 N sodium hydroxide*. Correct the test result for volume additions.

Standard Solutions

Prepare a 90.0-mg/L barium standard solution as follows:

1. Pipet 9.00 mL of Barium Standard Solution, 1000-mg/L, into a 100-mL volumetric flask.

2. Dilute to the mark with deionized water.

3. Prepare this solution daily. Perform the barium procedure as described above.

*See Optional Reagents and Apparatus on page 4.

To adjust the calibration curve using the reading obtained with the 90.0-mg/L standard solution:

1. Touch **OPTIONS>MORE** on the current program menu. Touch **STANDARD ADJUST** on the Options menu.

2. Touch **ON**. Touch **ADJUST** to accept the displayed concentration. If an alternate concentration is used, touch the number in the box to enter the actual concentration, then touch **OK**. Touch **ADJUST**.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell, or AccuVac Ampul (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a Barium Standard Solution, 1000-mg/L Ba.

5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to the unspiked sample. Touch the timer icon. After the timer expires, read the result. Press **READ** to accept the reading.

6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Touch the timer icon. After the timer expires, read the result. Press **READ** to accept the reading.

7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Touch the timer icon. After the timer expires, read the result. Press **READ** to accept the reading.

8. Each addition should reflect approximately 100% recovery.

9. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Calibration Standard Preparation

Prepare calibration standard containing 10, 20, 30, 50, 80, 90, and 100 mg/L Ba as follows:

1. Into seven different 100-mL Class A volumetric flasks, pipet 1, 2, 3, 5, 8, 9, and 10 mL of the 1000-mg/L Barium Standard Solution using Class A glassware.

2. Dilute to the mark with deionized water. Mix thoroughly.

3. Using the turbidimetric method and the calibration procedure described in the user manual, generate a calibration curve from the standards prepared above.

Summary of Method

The BariVer® 4 Barium Reagent Powder combines with barium to form a barium sulfate precipitate, which is held in suspension by a protective colloid. The amount of turbidity present caused by the fine white dispersion of particles is directly proportional to the amount of barium present. Test results are measured at 450 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
BariVer® 4 Barium Reagent Powder Pillows	1	100/pkg	12064-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Beaker, 50-mL	1	each	500-41H
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Barium Standard Solution, 1000-mg/L Ba	100 mL	14611-42
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Filter paper for funnel 12, 0.5 cm	100/pkg	1894-57
Funnel, 65 mm	each	1083-67
Nitric Acid 1:1, 500 mL	—	2540-49
Sodium Hydroxide, 5.0 N, 100 mL	—	2450-32

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Updated November 2005 Edition 1

Benzotriazole/Tolyltriazole

Method 8079

Powder Pillows

UV Photolysis Method[1]

(Benzotriazole 1.0 to 16.0 mg/L, Tolyltriazole 1.0 to 20.0 mg/L)

Scope and Application: For cooling or boiler water

[1] Adapted from Harp, D., *Proceedings 45th International Water Conference*, 299 (October 22–24, 1984)

Test Preparation

Before starting the test:

Avoid fingerprints on the quartz surface of the lamp. Rinse the lamp and wipe with a soft, clean tissue between tests.
Check the sample temperature. It must be between 20–25 °C (68 –78 °F) for accurate results.
If sample contains nitrite or borax (sodium borate), adjust the pH to 4–6 with 1 N sulfuric acid.
If the sample contains more than 500 mg/L hardness (as $CaCO_3$), add 10 drops of Rochelle Salt Solution before adding reagent.

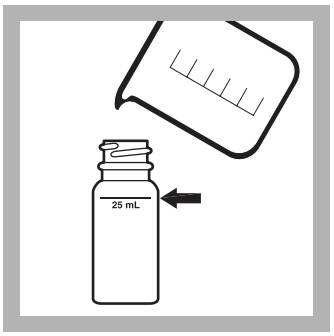
Collect the following items:	**Quantity**
Triazole Reagent powder pillows	1
One-inch square 10-mL sample cells	2
Square bottle, 25-mL	1
Ultra-violet lamp with power supply	1
UV safety goggles	1

Note: *Reorder information for consumables and replacement items is on page 5.*

Powder Pills Method 8079

1. Press **STORED PROGRAMS**.

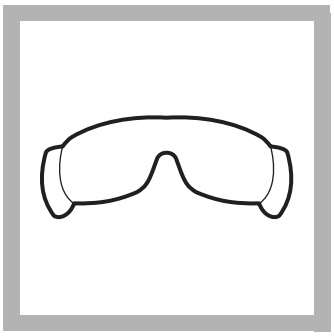
2. Select the test.



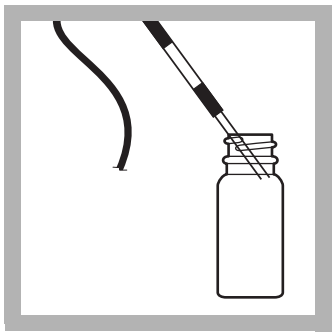
3. Prepared Sample: Fill a marked mixing bottle to the 25 mL line with sample.

4. Add the contents of one Triazole Reagent Powder Pillow.

Swirl to dissolve completely.



5. Put on UV safety goggles.



6. Insert the ultraviolet lamp into the mixing bottle.

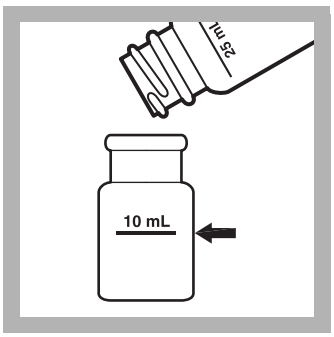
Turn on the UV lamp.

7. Press **TIMER>OK**.

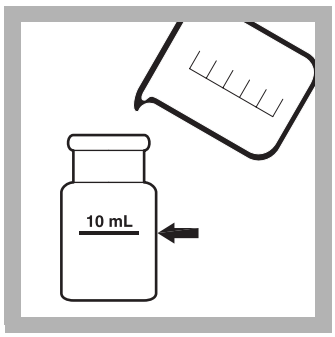
A 5-minute reaction period will begin. Low results will occur if photolysis (lamp on) takes place for more or less than five minutes.

A yellow color will form if triazole is present.

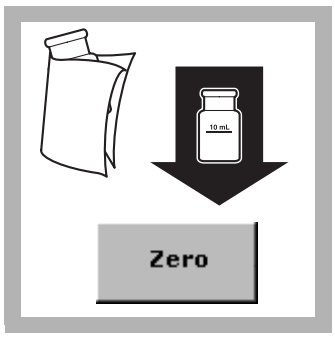
8. When the timer expires, turn the lamp off. Remove the lamp from the bottle. Swirl the bottle to mix thoroughly.



9. Fill a square sample cell with 10 mL of reacted (prepared) sample.



10. Blank Preparation: Fill another square sample cell with 10 mL of sample.



11. Insert the blank into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

0.0 mg/L Benzotriazol

or

0.0 mg/L Tolyltriazol

12. Insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Benzotriazol or mg/L Tolyltriazol.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acrylates (as methyl acrylate)	Greater than 50 mg/L
Alum	Greater than 400 mg/L
Borate (as sodium tetraborate)	Greater than 4000 mg/L. Adjust the pH to 4–6 with 1 N sulfuric acid[1].
Chlorine (as Cl_2)	Greater than 20 mg/L
Chromium (as chromate)	Greater than 12 mg/L
Copper	Greater than 10 mg/L
Hardness	Greater than 500 mg/L as $CaCO_3$. Add 10 drops of Rochelle Salt Solution[1] before adding reagent.
Iron	Greater than 20 mg/L
Lignosulfonates	Greater than 40 mg/L
Magnesium	Greater than 300 mg/L as $CaCO_3$
Molybdenum (as molybdate)	Greater than 200 mg/L
Nitrite	Greater than 4000 mg/L. Adjust the pH to 4–6 with 1 N sulfuric acid[1].
Phosphonates (AMP or HEDP)	Greater than 100 mg/L
Sulfate	Greater than 200 mg/L
Zinc	Greater than 80 mg/L
Strong oxidizing or reducing agents	Interfere at all levels

[1] See Optional Reagents on page 5.

Sample Collection, Storage, and Preservation

The most reliable results are obtained when samples are analyzed as soon as possible after collection.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open a 500-mg/L Benzotriazole Standard Solution*.

* See Optional Reagents on page 5.

5. Prepare three sample spikes. Fill three mixing cylinders with 25 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

UV Lamp Check

To verify the ultraviolet lamp (normal life equals 5000 hours) is working properly, perform the following test:

1. Prepare a 5.0 mg/L benzotriazole standard solution by pipetting 10.0 mL of Benzotriazole Standard Solution, 500-mg/L benzotriazole, into a 1-liter volumetric flask. Dilute to volume.

2. Analyze according to the above procedure. If the result is significantly below 5 mg/L, replace the lamp.

Summary of Method

Benzotriazole or tolyltriazole, used in many applications as corrosion inhibitors for copper and copper alloys, are determined by a proprietary catalytic ultraviolet (UV) photolysis procedure requiring less than 10 minutes to perform. Test results are measured at 425 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Triazole Reagent Powder Pillows	1	100/pkg	21412-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
UV Safety Goggles	1	each	21134-00
Sample Cell, 1-in. Square, 10 mL	2	2/pkg	24954-02
Bottle, Square, with 25 mL mark	1	each	17042-00
Select one based on available voltage:			
Lamp Kit, UV, with power supply, 115 VAC, 60 Hz	1	each	20828-00
Lamp Kit, UV, with power supply, 230 VAC, 50 Hz	1	each	20828-02

Recommended Standards

Description	Quantity/Test	Unit	Cat. No.
Benzotriazole Standard Solution, 500-mg/L	0.6 mL	100 mL	21413-42

Optional Reagents

Description	Cat. No.
Sulfuric Acid, 1 N	1270-32
Rochelle Salt Solution	1725-33

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Updated November 2005 Edition 1

Boron

Method 10061 — Azomethine-H Method[1]

Powder Pillows — LR (0.02 to 1.50 mg/L as B)

Scope and Application: For testing low levels of boron (boric acid or borates) in drinking water, cooling water, industrial process waters, or wastewaters

[1] Adapted from ISO Method 9390 Harp, D.L., Analytica Chimica Acta, 346(1997), pp. 373–379.

Test Preparation

Before starting the test:

For best results, match two cells using the Cell Matching Procedure on page 3.
Sample temperature should be 22–24 °C (72–75 °F) for most accurate results.
If outside this range, measure and record the sample temperature. See .
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

Collect the following items:	**Quantity**
BoroTrace™ Reagent Set Includes:	—
EDTA Solution, 1M	20 drops
BoroTrace™ #2 Reagent Powder Pillow	2
BoroTrace™ #3 Reagent Powder Pillow	2
Water, Ultra-pure, aldehyde-free	25 mL
Sample cell, 1-inch square, plastic, with cap	2

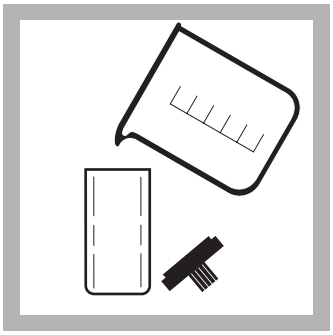
Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows — Method 10061

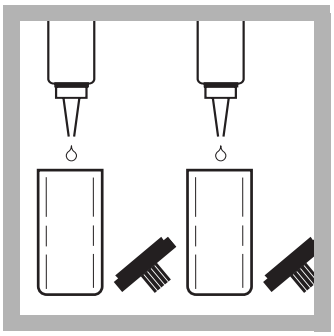
1. Press **STORED PROGRAMS**.

2. Select the test.

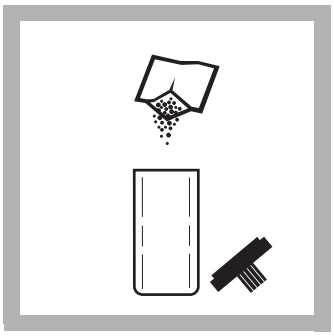
3. Blank Preparation: Fill a clean plastic sample cell to the 25-mL mark with ultra-pure water.



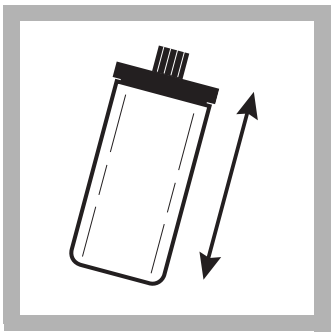
4. Prepared Sample: Fill a second clean plastic sample cell to the 25-mL mark with sample.



5. Add ten drops of EDTA Solution, 1 M, to each cell. Cap and invert each cell twice to mix.



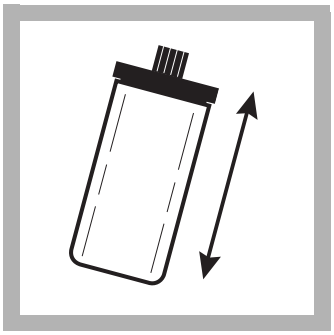
6. Open one pillow BoroTrace™ 2 Reagent and add the contents to the prepared sample.



7. Cap the prepared sample and shake to dissolve the powder. Proceed immediately with steps 8 and 9.

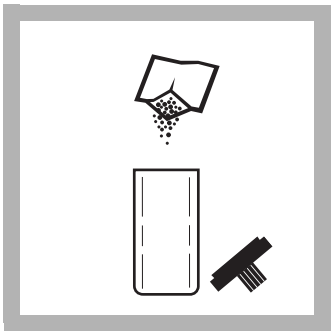
8. Press **TIMER>OK**.

A ten-minute reaction period will begin.

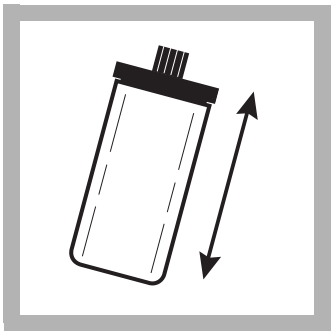


9. Continue shaking vigorously for 30 seconds, or until all powder is dissolved

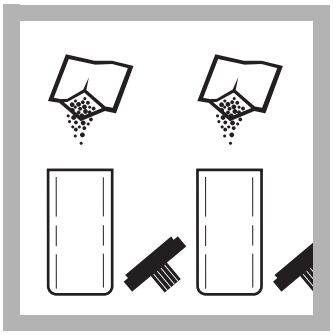
Let the cell sit capped for the remaining reaction period.



10. During the reaction period, add the contents of a BoroTrace™ 2 Reagent Powder Pillow to the blank.



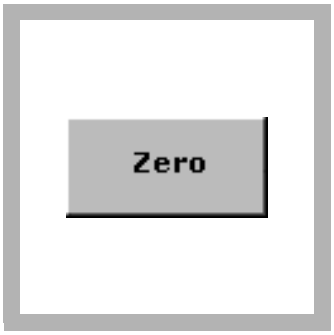
11. Cap the blank and shake vigorously until the powder is dissolved.



12. After the timer expires, add the contents of one BoroTrace™ 3 Reagent Powder Pillow to each cell. Cap and shake to dissolve.

BoroTrace™ 3 Reagent “stops” the reaction.

13. Wipe the blank and insert it into the cell holder.

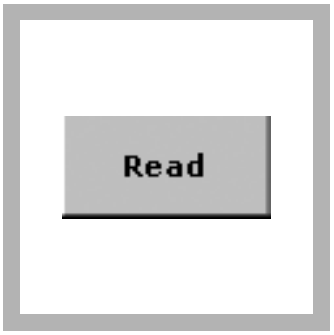


14. Press **ZERO**.

The display will show:

0.00 mg/L B

15. Wipe the prepared sample and insert it into the cell holder.



16. Press **READ**.

Results are in mg/L B.

Cell Matching Procedure

1. Rinse and fill two cells with deionized water.
2. Wipe each cell with a soft cloth or tissue to remove liquid or fingerprints.
3. Using one of the cells, set the instrument absorbance at 410 nm to zero.
4. Read the absorbance of the other cell.
5. Cells that read within 0.002 absorbance are matched.

Interferences

The substances in Table 1 have been tested for interference and found not to interfere up to the indicated levels (in mg/L):

Table 1 Interfering Substances and Levels

Substance	Interference Level Tested
Aluminum ($^{3+}$)	10
Benzotriazole	20
Biocides: Carbamate-type Isothiazolin-type Quat-type Thiocyanate-type	 120 120 90 60
Calcium	1000 (as $CaCO_3$)
Chloride	2500
Copper ($^{2+}$)	20
Magnesium	1000 (as $CaCO_3$)
Manganese ($^{7+}$)	5
Molybdate (Mo^{6+})	60
Phosphonates, AMP	20
Phosphonates, HEDP	20
Polyacrylates	20 (as Acumer 1000, 1100)
Polymaleic Acid	40 (as Belclene 200)
Silica	120
Sulfate	1800
Sulfite	40
Tolyltriazole	20
Zinc ($^{2+}$)	10

Table 2 lists suggested treatments for interferences:

Table 2 Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Alkalinity >500 mg/L (+ or –)	**1.** Adjust sample pH to between 5–7 using 1.0 N Sulfuric Acid Solution[1]. **2.** Continue with step 5 of the analytic procedure.
Color (+)	**1.** Zero the instrument (0.00 mg/L B) using Ultra-Pure Water[2]. **2.** Measure and record the apparent concentration, in mg/L B, due to the sample color. **3.** Subtract the apparent concentration from the result in step 16 of the test procedure.
Halogens (Bromine or Chlorine) all levels (+)	Halogen disinfectants in the sample can produce a red-color after the addition of BoroTrace™ 2 Reagent. To eliminate this interference: **1.** Add 1 pillow Dechlorinating Reagent[1] to 25 mL each of Ultra-Pure Water[2] and sample. **2.** Cap and shake to dissolve. **3.** Continue with step 5 of the test procedure.
Iron (Fe^{3+} or Fe^{2+}), above 8 mg/L (+)	High levels of iron in the sample can produce a red-color after the addition of BoroTrace™ 2 Reagent. To compensate, increase the amount of EDTA[2] from 10 drops to 15 drops to be added to each cell (step 6). Alternatively, dilute the sample with Ultra-Pure Water and continue with step 5 of the test procedure. Correct the results (step 16) using the appropriate dilution factor.
Nitrites, all levels (+)	**1.** Add 0.1 gram scoop Sulfamic Acid[1] to 25 mL each Ultra-Pure Water and sample in plastic cells. **2.** Cap and shake to dissolve. **3.** Uncap and wait 5 minutes. **4.** Add 5 N Sodium Hydroxide Reagent[1] solution to each to adjust pH to 5–8 using pH paper. **5.** Continue with step 4 of the test procedure.
Turbidity (+)	Filter the sample through a 3 µm membrane[1] prior to testing. Do not use a glass fiber filter.

[1] Optional Reagents and Apparatus on page 6
[2] Required Reagents on page 6

Sample Collection, Preservation, and Storage

Collect samples in clean polyethylene bottles. Do not use borate-based detergents or soaps to clean sample containers or labware used for this method. After use, thoroughly rinse all plastic containers with deionized water, allow to air dry, and keep covered.

Sample Temperature Compensation

The reaction chemistry is very dependent on the sample temperature. Calibrations are performed at 23 °C (73 °F). If the sample temperature is outside the range of 22–24 °C (72–75 °F), multiply the results, in mg/L, by the appropriate multiplier (Table 3).

Table 3 Sample Temperature Multipliers

Sample Temp.		Multiplier	Sample Temp.		Multiplier
°C	°F		°C	°F	
5	41	0.70	20	68	0.94
7	45	0.73	25	77	1.04
10	10	0.78	26	79	1.06

Table 3 Sample Temperature Multipliers (continued)

Sample Temp.		Multiplier	Sample Temp.		Multiplier
°C	°F		°C	°F	
12	54	0.81	27	81	1.08
14	57	0.84	28	82	1.10
16	61	0.87	29	84	1.12
18	64	0.91	30	86	1.15

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare a 50.0-mg/L boron standard by pipetting 5.0 mL of a 1000-mg/L Boron Standard Solution into a 100-mL plastic volumetric flask. Dilute with deionized water, stopper and invert to mix.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of the diluted standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

Prepare a 1.0-mg/L B standard as follows:

1. Using plastic pipet, transfer 4.0 mL of Boron Standard Solution, 250-mg/L as B, into a 1000-mL plastic volumetric flask. Dilute to volume with deionized water, stopper and invert to mix.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* Optional Reagents and Apparatus on page 6

Summary of Method

Azomethine-H, a Schiff base, is formed by the condensation of an aminonaphthol with an aldehyde by the catalytic action of boron. Test results are measured at 410 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
BoroTrace™ Reagent Set, includes:	—	—	26669-00
EDTA Solution, 1 M	20 drops	50 mL SCDB	22419-26
BoroTrace™ #2 Reagent Powder Pillows	2	100/pkg	26666-69
BoroTrace™ #3 Reagent Powder Pillows	2	100/pkg	26667-99
Water, Ultra-pure, Aldehyde-free	25-mL	500 mL	25946-49

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample cell, 1-inch square plastic, with cap	2	12/pkg	24102-12

Recommended Standards

Description	Unit	Cat. No.
Boron Standard Solution, 10-mL Voluette® Ampule, 250 mg/L B	16/pkg	14249-10
Boron Standard Solution, 1000-mg/L as B	100 mL	1914-42

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Cylinder, mixing, 25 mL	each	20886-40
Dechlorinating Reagent, Powder Pillows	100/pkg	14363-69
Filter Holder, 25 mm	each	2468-00
Membrane, filter, 3 µm, 25 mm	25/pkg	25940-25
pH Paper, 1.0–11.0	5 rolls/pkg	391-33
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet, tips for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 5.00 mL	each	14515-37
Safety Bulb	each	14651-00
Sodium Hydroxide Reagent, 5.0 N, 50 mL	each	2450-26
Spoon, measuring, 0.1 g	each	511-00
Sulfamic Acid, 113g	each	2344-14
Sulfuric Acid Solution 1.0N, 100 mL	each	1270-32
Syringe, 30 cc Luer-lock	each	22258-00

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Updated November 2005 Edition 1

Boron

Method 8015
Powder Pillows

Carmine Method[1]
(0.2 to 14.0 mg/L)

Scope and Application: For water and wastewater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

Before starting the test:

All labware must be completely dry. Excess water will cause low results.
Use the BoroVer® 3 Reagent with adequate ventilation. See Reagent Preparation on page 3.
Do not cap the sample cells or the Erlenmeyer flasks at any time during sample preparation or reaction time. Samples may be capped immediately prior to placing in the instrument.
Sulfuric acid may contain residual moisture; this will cause low results.
Ensure sulfuric acid suitability by running a known boron standard before running any unknown samples.

Collect the following items:	**Quantity**
BoroVer 3 Reagent Powder Pillow	1
Cylinders, graduated, 50-mL and 100 mL	1 of each
Flask, Erlenmeyer, 125-mL	2
Flask, Erlenmeyer, 250-mL	1
Pipet, volumetric, 2.0 mL	2
Sulfuric Acid, Concentrated	75 mL
Sample Cells, 1-inch square	2
Water, deionized	2.0 mL

Note: *Reorder information for consumables and replacement items is on page 5.*

Powder Pillows — Method 8015

1. Press **STORED PROGRAMS**.

2. Select the test.

3. Using a 100-mL graduated cylinder, measure 75 mL of concentrated sulfuric acid. Pour the acid into a 250-mL Erlenmeyer flask.

4. Add the contents of one BoroVer 3 Reagent Powder Pillow to the flask. Swirl to mix. Allow up to five minutes for the powder to dissolve completely.

5. Blank Preparation: Accurately pipet 2.0 mL of deionized water into a 125-mL Erlenmeyer flask.

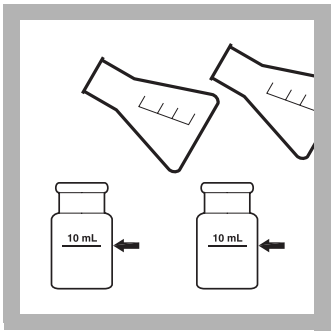
6. Prepared Sample: Accurately pipet 2.0 mL of sample into another 125-mL Erlenmeyer flask.

7. Using a 50-mL graduated cylinder, add 35 mL of the solution prepared in step 4 to each Erlenmeyer flask.

Swirl to mix completely.

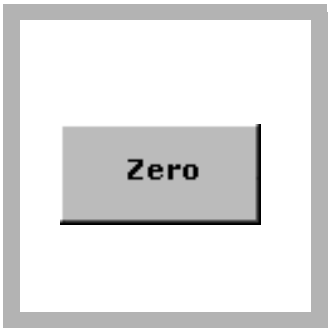
8. Press **TIMER>OK**.

A 25-minute reaction period will begin.



9. When the timer expires, pour at least 10 mL from each flask into separate square sample cells.

10. Wipe the blank and insert the blank into the cell holder with the fill line facing right.



11. Press **ZERO**.

The display will show:

0.0 mg/L B

12. Wipe the prepared sample and insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L B.

Sample Collection, Preservation, and Storage

Collect samples in clean polyethylene or polypropylene bottles, or alkali-resistant, boron-free glass.

Reagent Preparation

To prepare additional BoroVer 3/Sulfuric Acid Solution, mix one BoroVer 3 Reagent Powder Pillow per 75 mL of concentrated sulfuric acid, adding the powder pillows individually while stirring. Preparation of this solution generates gaseous HCl when the indicator pillow is added to the sulfuric acid. Use of a fume hood or other well-ventilated lab area is strongly advised. This solution is stable for up to 48 hours if stored in plastic containers. Do not store in borosilicate glassware (Pyrex® or Kimax®) for longer than one hour; the solution may leach boron from these containers.

The BoroVer 3/Sulfuric Acid Solution is highly acidic. Neutralize to pH 6–9 and flush down the drain for disposal. Refer to the current MSDS for safe handling and disposal instructions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the unspiked sample in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Boron Voluette® Ampule Standard, 250-mg/L B.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze 2.0 mL of each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method
Check the accuracy of the test using Boron Standard Solution, 10-mg/L as B. Prepare this solution as follows:

1. Using Class A glassware, pipet 10.00 mL of the Boron Voluette Ampule Standard, 250-mg/L B, into a 250-mL volumetric flask. Dilute to volume with deionized water. Swirl to mix.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Boron is determined by its reaction with carminic acid in the presence of sulfuric acid to produce a reddish to bluish color. The amount of color is directly proportional to the boron concentration. Test results are measured at 605 nm.

* See Optional Reagents and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
BoroVer® 3 Boron Reagent Powder Pillows	1	100/pkg	14170-99
Sulfuric Acid, ACS, concentrated	75 mL	2.5 L	979-09
Water, deionized	2.0 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 50-mL	1	each	508-41
Cylinder, graduated, 100-mL	1	each	508-42
Flask, Erlenmeyer, 125-mL	2	each	505-43
Flask, Erlenmeyer, 250-mL	1	each	505-46
Pipet, volumetric, 2.0-mL	2	each	14515-36
Sample cells, 10-mL, 1-inch square, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Boron Standard Solution, 10-mL Voluette® Ampule, 250 mg/L B	16/pkg	14249-10

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing, 25 mL	20886-40

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Updated November 2005 Edition 1

Bromine

Method 8016 — DPD Method[1]

Powder Pillows or AccuVac® Ampuls — (0.05 to 4.50 mg/L)

Scope and Application: For testing bromine residuals (including hypobromite, hypobromous acid and bromamines) used as disinfectants in process waters, treated water, estuary water, and seawater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

Test Preparation

Before starting the test:

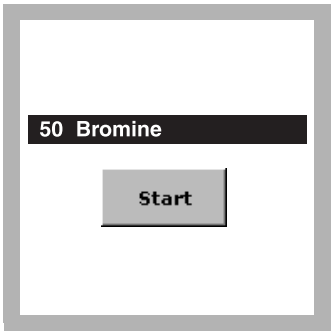
Analyze samples immediately. Do not preserve for later analysis.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
If the sample temporarily turns yellow after reagent addition, dilute a fresh sample and repeat the test. A slight loss of bromine may occur because of the dilution. Multiply the result by the dilution factor.

Collect the following items:	**Quantity**
Powder Pillow Test:	
DPD Total Chlorine Reagent Powder Pillows	1
Sample cells, 1-in. square, 10-mL	2
AccuVac Test:	
DPD Total Chlorine Reagent AccuVac® Ampuls	1
Beaker, 50-mL (AccuVac test)	1
Sample cell, 10-mL round, with cap	1
Stopper, 18 mm tube	1

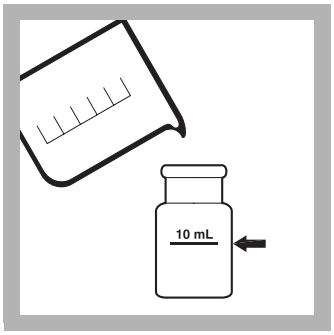
Note: *Reorder information for consumables and replacement items is on page 5.*

Powder Pillows — Method 8016

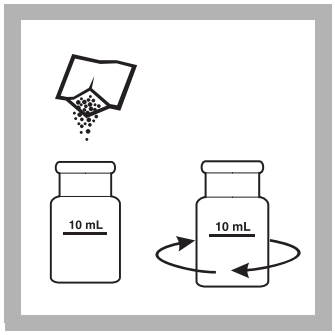
1. Press **STORED PROGRAMS**.



2. Select the test.



3. Fill a square sample cell with 10 mL of sample.



4. Prepared Sample: Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell.

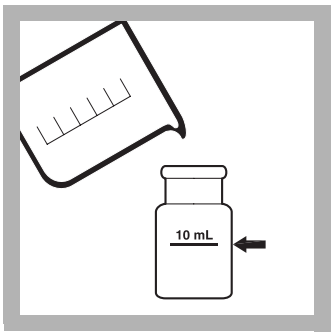
Swirl for 20 seconds to mix.

A pink color will develop if bromine is present.

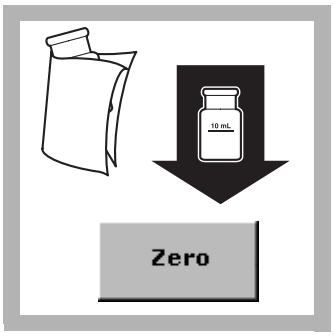
5. Press **TIMER>OK**.

A three-minute reaction period will begin.

Perform steps 6–7 during the reaction period.



6. Blank Preparation: Fill a second square sample cell with 10 mL of sample.



7. Wipe the blank and insert it into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

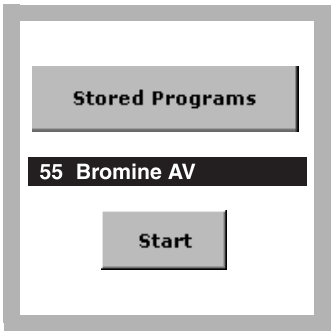
0.00 mg/L Br_2

8. Within three minutes after the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing right.

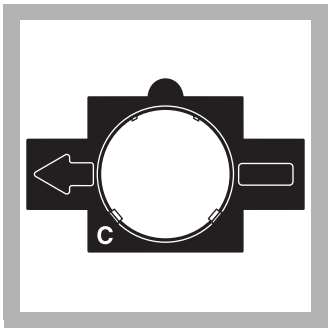
Press **READ**.

Results are in mg/L Br_2.

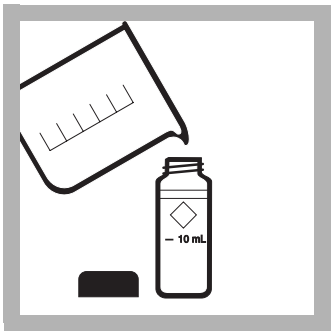
AccuVac® Ampul — Method 8016



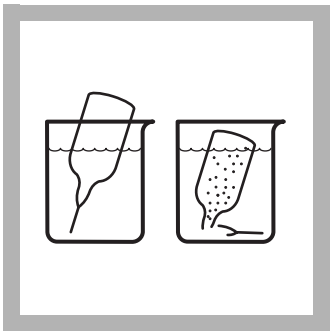
1. Select the test.



2. Insert Adapter C.



3. Blank Preparation: Fill a round 1-inch sample cell with 10 mL of sample.



4. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.

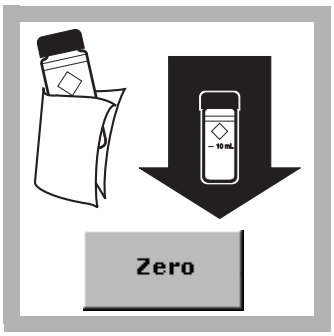
5. Quickly invert the Ampul several times to mix.

A pink color will develop if bromine is present.

6. Press **TIMER>OK**.

A three-minute reaction period will begin.

Perform steps 7 during the reaction period.



7. Wipe the blank and insert it into the cell holder.

Press **ZERO**.

The display will show:

0.00 mg/L Br_2

8. Within three minutes after the timer expires, wipe the Ampul and insert it into the cell holder.

Press **READ**.

Results are in mg/L Br_2.

Interferences

Table 1Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sodium Hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Chlorine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7. **2.** Add 3 drops Potassium Iodide[1] (30-g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops Sodium Arsenite[1, 2] (5-g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct bromine concentration.
Monochloramine	Interferes at all levels
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust to pH 6–7.

[1] See Optional Reagents and Apparatus on page 5.
[2] Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by the Federal RCRA for arsenic (D004).

Sample Collection, Storage, and Preservation

Collect samples in clean, dry glass containers. If sampling from a tap, allow the water to flow at least 5 minutes to ensure a representative sample. Avoid excessive agitation and exposure to sunlight when sampling. Allow several volumes of water to overflow the container and cap the container so there is no headspace above the sample. If sampling with a DR cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Proceed with the analysis immediately.

Summary of Method

Bromine residuals reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a pink color which is proportional to the total bromine concentration. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1	100/pkg	21056-69
OR			
DPD Total Chlorine Reagent AccuVac® Ampuls	1	25/pkg	25030-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, 2-mL Voluette® Ampule, 25–30 mg/L	20/pkg	26300-20

Optional Reagents and Apparatus

Description	Cat. No.
Chlorine Demand-Free Water, 500 mL	26415-49
Cylinder, mixing, 25 mL	20886-40
Cylinder, mixing, 50 mL	1896-41
Potassium Iodide, 30 g/L, 100 mL	343-32
Sodium Arsenite, 5 g/L, 100 mL	1047-32
Sodium Hydroxide, 1 N, 100 mL	1045-32
Sulfuric Acid, 1 N, 100 mL	1270-32
Stopper for 18 mm tube, 6/pkg	1731-06

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Updated November 2005 Edition 1

Cadmium

Method 8017 — Dithizone Method[1]

Powder Pillows — (0 to 80.0 µg/L)

Scope and Application: For water and wastewater; digestion is required to determine total cadmium.

[1] Adapted from Standard Methods for the Examination of Water and Wastewater.

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.
Clean all glassware with 6 N Hydrochloric Acid Solution and rinse with deionized water.
Cloudy and turbid samples may require filtering before running the test. Report results as µg/L soluble cadmium. Use glass membrane type filter to avoid loss of cadmium by adsorption onto the filter paper.
If samples cannot be analyzed immediately, see Sample Collection, Preservation, and Storage on page 4. Adjust the pH of preserved samples before analysis.
The Flow Cell and Sipper Modules cannot be used with this procedure.
The DithiVer powder will not completely dissolve in the chloroform. For further notes see Dithiver Solution Preparation and Storage on page 5.
Read the MSDS before testing. Spilled reagent will affect test accuracy and is hazardous to skin and other materials.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

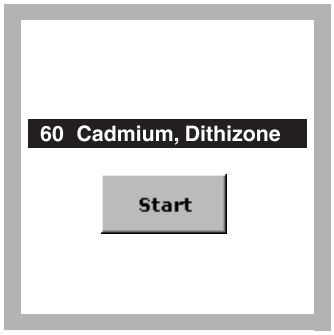
Collect the following items:	**Quantity**
Citrate buffer powder pillows	1
Chloroform	30 mL
DithiVer Metals Reagent powder pillows	1
Potassium Cyanide	0.1 g
Sodium Hydroxide solution, 50%	20 mL
Cotton balls	1
Clippers	1
Cylinder, 25-mL graduated	1
Cylinder, 250-mL graduated	1
Cylinder, 50-mL graduated mixing	1
Funnel, 500-mL separatory	1
Sample Cells	2
Spoon, measuring, 0.1-g	1
Support ring (4-inch) and stand (5 x 8-inch base)	1

Note: *Reorder information for consumables and replacement items is on page 6.*

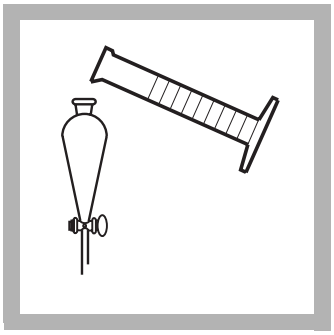
Powder Pillows — Method 8017

DANGER
Cyanide is a deadly poison. Use a fume hood. Maintain cyanide solutions at pH 11 or greater to prevent formation of cyanide gas.

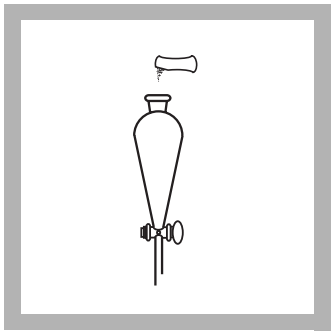
1. Press **STORED PROGRAMS**.



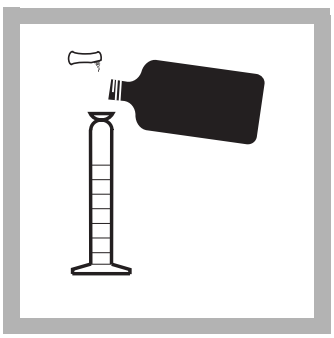
2. Select the test.



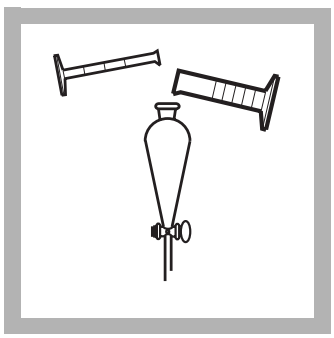
3. Fill a 250-mL graduated cylinder to the 250-mL mark with sample. Pour the sample into a 500-mL separatory funnel.



4. Add the contents of one Buffer Powder Pillow for heavy metals, citrate type. Stopper the funnel and shake to dissolve.



5. DithiVer Solution Preparation:
Add 50 mL of chloroform to a 50-mL mixing graduated cylinder. Add the contents of one DithiVer Metals Reagent Powder Pillow. Stopper the cylinder. Invert several times to mix.

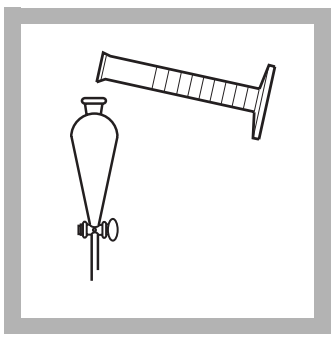


6. Add 20 mL of 50% Sodium Hydroxide Solution. Add a 0.1-g scoop of potassium cyanide to the funnel. Stopper. Shake vigorously for 15 seconds.

7. Remove the stopper.

Press **TIMER>OK**.

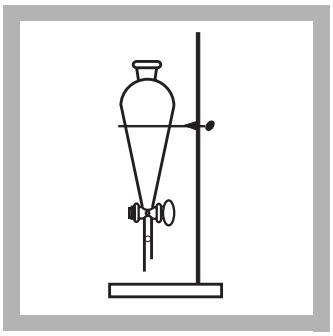
A 1-minute reaction period will begin.



8. Add 30 mL of the DithiVer solution to the 500-mL separatory funnel. Stopper, invert, and open stopcock to vent. Close the stopcock and shake funnel once or twice; vent again.

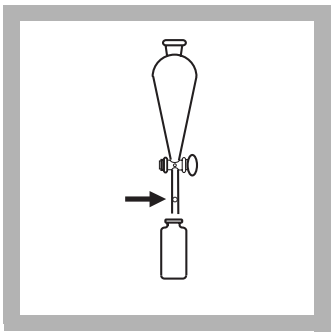
9. Press **TIMER>OK**.

Close the stopcock and shake the funnel vigorously during the 1 minute time period.



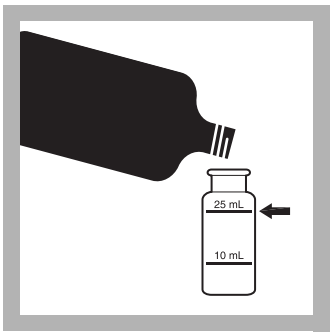
10. Press **TIMER>OK** and allow the funnel to stand undisturbed until the timer expires.

The bottom (chloroform) layer will be orange to pink if cadmium is present.



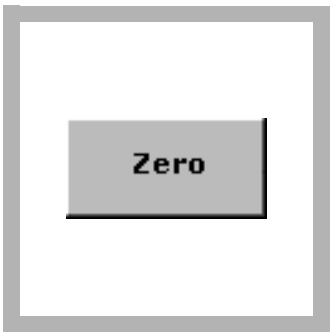
11. Prepared Sample: Insert a cotton plug the size of a pea into the delivery tube of the funnel and slowly drain the bottom (chloroform) layer into a dry 25-mL sample cell (the prepared sample). Stopper.

The cadmium-dithizone complex is stable for more than one hour if the sample cell is kept tightly capped and out of direct sunlight.



12. Blank Preparation: Fill a dry 25-mL sample cell with chloroform. Stopper.

13. Insert the blank into the cell holder with the fill line facing right.



14. Press **ZERO**.

The display will show:

0.0 µg/L Cd

15. Insert the prepared sample into the cell holder with the fill line facing right.

16. Press **READ**.

Results are in µg/L cadmium.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Highly buffered samples or Extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.
Bismuth	Greater than 80 mg/L. See treatment below.
Copper	Greater than 2 mg/L. See treatment below.
Mercury	All levels. See treatment below.
Silver	Greater than 2 mg/L. See treatment below.

Table 2 Substances That Do Not Interfere

Aluminum	Lead
Antimony	Magnesium
Arsenic	Manganese
Calcium	Nickel
Chromium	Tin
Cobalt	Zinc
Iron	

To eliminate interference from the metals in Table 1, insert the following steps into the procedure after step 5.

1. Measure about 5-mL of the DithiVer solution into the separatory funnel. Stopper the funnel, invert and open the stopcock to vent. Close the stopcock and shake the solution vigorously for 15 seconds. Allow the funnel to stand undisturbed until the layers separate (about 30 seconds). A yellow, red, or bronze color in the bottom (chloroform) layer confirms the presence of interfering metals. Draw off and collect the bottom (chloroform) layer for proper disposal.

2. Repeat extraction with fresh 5 mL portions of the DithiVer solution (discarding the bottom layer each time) until the bottom layer shows a pure dark green color for three successive extracts. Extractions can be repeated several times without appreciably affecting the amount of cadmium in the sample.

3. Extract the solution with several 2- or 3-mL portions of pure chloroform to remove any remaining DithiVer, collecting the bottom layer each time for proper disposal.

4. Continue with step 6 of the procedure.

5. In step 8, substitute 28.5 mL of DithiVer solution for the 30 mL.

6. Continue with step 9 of the procedure.

Sample Collection, Preservation, and Storage

Collect samples in an acid-washed glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Adjust the pH to 2.5 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions.

Dithiver Solution Preparation and Storage

Store DithiVer Powder Pillows away from light and heat. A convenient way to prepare this solution is to add the contents of 16 DithiVer Metals Reagent Powder Pillows to a 500-mL bottle of chloroform and invert several times until well mixed (carrier powder may not dissolve completely). Store dithizone solution in an amber glass bottle. This solution is stable for 24 hours.

Accuracy Check

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in top row. See the user manual for more information.

4. Snap the neck off a Cadmium Voluette Ampule Standard, 25-mg/L Cd (25,000-µg/L Cd).

5. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to three 250-mL samples and mix each thoroughly.

6. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 5.0-mg/L cadmium standard solution by pipetting 5.00 mL of Cadmium Standard Solution, 100-mg/L Cd, into a 100-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily.

2. Pipet 2.00 mL of the 5.0-mg/L Cadmium Standard Solution into 248 mL of deionized water in a 500-mL separatory funnel. This is a 40-µg/L cadmium solution. Perform the cadmium test on this solution beginning with step 4 of the procedure.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The dithizone method is designed for the determination of cadmium in water and wastewater. The DithiVer Metals Reagent is a stable powder form of dithizone. Cadmium ions in basic solution react with dithizone to form a pink to red cadmium-dithizonate complex, which is extracted with chloroform. Test results are measured at 515 nm.

Pollution Prevention and Waste Management

Both chloroform (D022) and cyanide (D003) solutions are regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. Chloroform solutions and the cotton plug used in the delivery tube of the separatory funnel should be collected for disposal with laboratory solvent waste. Collect the cyanide solution as a reactive waste. Be sure that cyanide solutions are stored in a caustic solution with a pH >11 to prevent potential release of hydrogen cyanide gas. See the current MSDS for disposal information.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Cadmium Reagent Set (100 Tests)	—	—	22422-00
Includes:(1) 14202-99, (1) 14458-17, (1) 12616-99, (1) 767-14, (4) 2180-49, (1) 2572-01			
Buffer Powder Pillows, citrate	1	100/pkg	14202-99
Chloroform, ACS	30 mL	4 L	14458-17
DithiVer Metals Reagent Powder Pillows	1	100/pkg	12616-99
Potassium Cyanide	0.1 g	125 g	767-14
Sodium Hydroxide Solution, 50%	20 mL	500 mL	2180-49
Cotton Balls, absorbent	1	100/pkg	2572-01

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25-mL	1	each	508-40
Cylinder, graduated, 250-mL	1	each	508-46
Cylinder, graduated, mixing, 50-mL	1	each	1896-41
Sample Cell, 1-in. Square, 25 mL with cap	2	2/pkg	26126-02
Funnel, separatory, 500-mL	1	each	520-49
Spoon, measuring, 0.1-g	1	each	511-00
Support Ring, 4"	1	each	580-01
Support Ring Stand, 5" x 8" base	1	each	563-00

Recommended Reagents and Standards

Description	Unit	Cat. No.
Cadmium Standard Solution, 100-mg/L Cd	100 mL	14024-42
Cadmium Standard Solution, 10-mL Voluette Ampule, 25-mg/L Cd	16/pkg	14261-10
Chloroform, ACS	500 mL	14458-49
Hydrochloric Acid Solution, 6.0 N	500 mL	884-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	2450-26
Water, deionized	4 L	272-56

Recommended Apparatus

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Cylinder, graduated, 5-mL	each	508-37
Filter Discs, glass, 47 mm	100/pkg	2530-00
Filter Holder, glass, for 47-mm filter	each	2340-00
Flask, Erlenmeyer, 500-mL	each	505-49
Flask, filtering, 500-mL	each	546-49
Flask, volumetric, Class A, 100-mL	each	14574-42
Flask, volumetric, Class A, 250-mL	each	14574-46
Flask, volumetric, Class A, 1000-mL, with glass stopper	each	14574-53
Hot Plate, 3½-in. diameter, 120 VAC, 50/60 Hz	each	12067-01
Hot Plate, 3½-in. diameter, 240 VAC, 50/60 Hz, variable control	each	12067-02
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
pH Meter, *sens**ion***™***1***, portable	each	51700-00
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2-mL	each	532-36
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet, volumetric, 2.00-mL, Class A	each	14515-36
Pipet, volumetric, 3.00-mL, Class A	each	14515-03
Pipet, volumetric, 6.00-mL, Class A	each	14515-06
Pipet, volumetric, 8.00-mL, Class A	each	14515-08
Pipet, volumetric, 9.00-mL, Class A	each	14515-09
Pipet, volumetric, 10.00-mL, Class A	each	14515-38
Pipet, volumetric, 20.00-mL, Class A	each	14515-20
Tongs, crucible, 9-inch	each	569-00

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Updated November 2005 Edition 1

Cadmium

Method 10217
TNTplus™ 852

Cadion Method
(0.02 to 0.30 mg/L Cd)

Scope and Application: For wastewater and process control.

Test Preparation

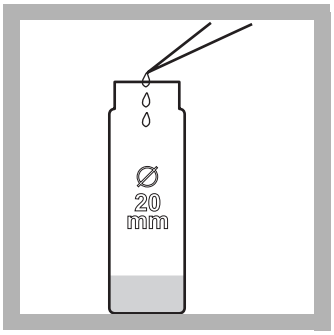
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample pH is 3–10.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F).
Recommended reagent storage is 2–8 °C (35.6–46.4 °F).
Undissolved cadmium or cadmium bound in complexes can only be determined after digestion with Metals Prep Set TNT890 has been performed.
Samples with calcium and magnesium concentrations greater than 50 mg/L require the use of the Calcium Separation Set TNT892.
TNTplus methods are activated directly from the Main Menu when the sample vial is inserted into the sample cell holder.

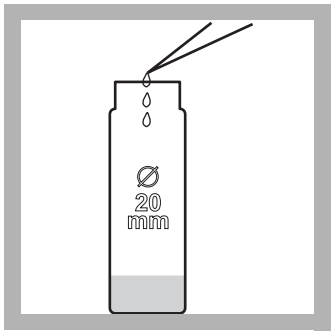
Collect the following items:	**Quantity**
Cadmium TNT852 Reagent Set	1
Light Shield	1
Pipettor, variable 1–5 mL	1
Pipettor tips for 1–5 mL pipettor	2
Pipettor, variable 100–1000 µL	1
Pipettor tips for 100–1000 µL pipettor	1
Pipet, volumetric, 10 mL	1
Safety pipet bulb	1

Note: *Reorder information for consumables and replacement items is on page 4.*

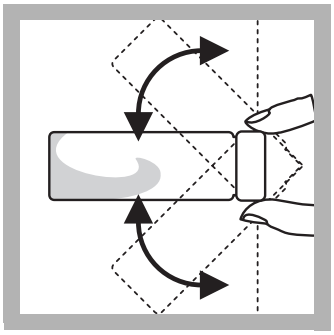
TNTplus Method 10217



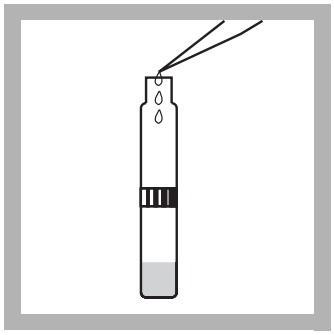
1. Pipet 10 mL of sample into the 20-mm reaction tube.



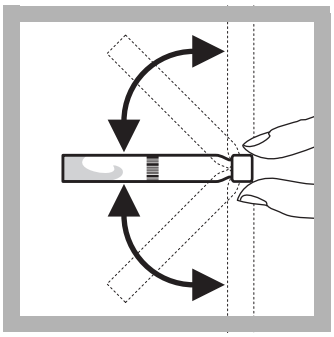
2. Pipet 1 mL of Solution A into the 20-mm reaction tube.



3. Cap the reaction tube and invert 2–3 times.



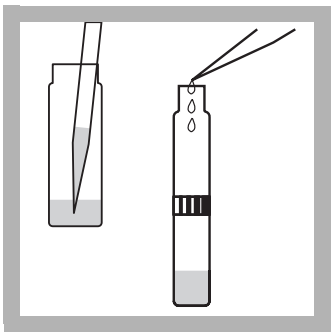
4. Pipet 0.4 mL of Solution B into a sample vial.



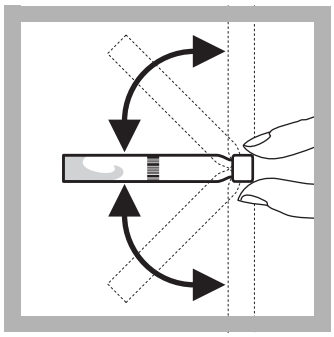
5. Cap and invert the vial 2–3 times.

Install the Light Shield in Cell Compartment #2.

6. Thoroughly clean the outside of the vial and insert it into the sample cell holder. The instrument reads the barcode, then selects the method and sets the blank.

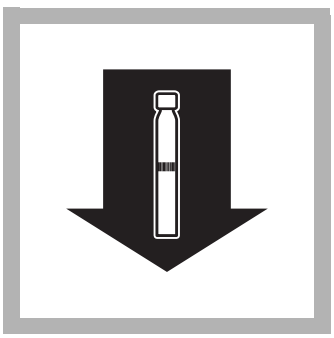


7. Remove the vial from the cell holder and pipet 4.0 mL of the pretreated sample from the 20-mm reaction tube in step 3 into the vial.



8. Cap the vial and invert the vial 2–3 times.

Wait **30 seconds** before proceeding to step 9.



9. Insert the prepared vial into the cell holder. The instrument reads the barcode, then reads the sample.

Results are in mg/L cadmium.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 9. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
SO_4^{2-}	1000 mg/L
Ca^{2+}, Mg^{2+}	50 mg/L
Fe^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, Pb^{2+}, Co^{2+}, Ag^{+}, Au^{+}, Cr^{6+}	25 mg/L
Mn^{2+}	2 mg/L

Sample Collection, Preservation, and Storage

Collect samples in an acid-washed glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Adjust the pH to between 3–6 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method
To check the accuracy of the method:

1. Prepare a 0.20 mg/L cadmium standard solution by pipetting 1.0 mL of a 100 mg/L cadmium standard solution into a 500 mL volumetric flask.

2. Dilute to volume with deionized water. Use 10 mL of this standard in place of the sample in the procedure.

Summary of Method

Cadion forms a complex with cadmium. The reduction in the color intensity of the cadion is used for the determination of cadmium. Test results are measured at 552 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Cadmium TNT852 Reagent Set	1	25/pkg	TNT852

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 1–5 mL	1	each	27951-00
Pipettor Tips, for 27951-00 pipettor	2	100/pkg	27952-00
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27952-00
Pipet, volumetric 10 mL	1	each	14515-38
Pipet filler, safety bulb	1	each	14651-00

Recommended Reagents and Standards

Description	Unit	Cat. No.
Cadmium Standard Solution, 100-mg/L Cd	100 mL	14024-42
Cadmium Standard Solution, 10-mL Voluette Ampule, 25-mg/L Cd	16/pkg	14261-10
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	2450-26
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
Calcium Separation Set TNT892	each	TNT892
Flask, volumetric 500 mL	each	14574-49
Metals Prep Set TNT890	each	TNT890
Pipet, volumetric 1.0 mL	each	14515-35
Test Tube Rack for 13-mm vials	each	24979-00

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Updated November 2005 Edition 1

Chloramine (Mono)

Indophenol Method[1]

Method 10172

HR (0.1 to 10.0 mg/L Cl_2)

Scope and Application: Chlorinated wastewater.

[1] Patent Pending

Test Procedure

Before starting the test:

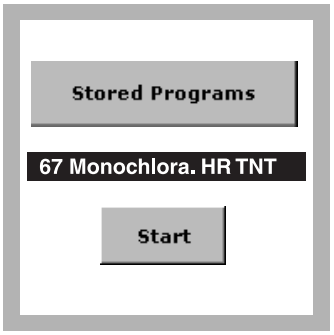
Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

Collect the following items:	Quantity
High-range Monochloramine Diluent Vials	1
Monochlor F Reagent Pillow	1
Funnel, micro	1
Light Shield	1
Pipet, Mohr glass, 2.00 mL	1
Test Tube Rack	1

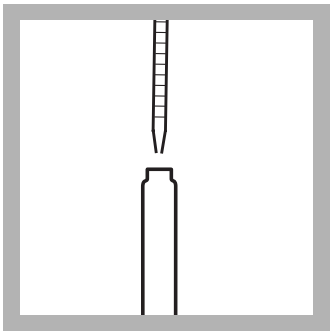
Note: *Reorder information for consumables and replacement items is on page 5.*

Test 'N Tube — Method 10172

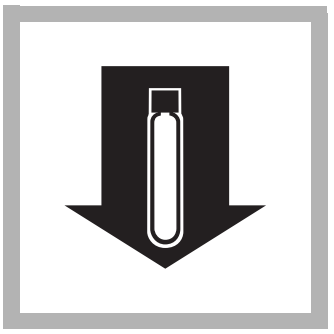


1. Select the test.

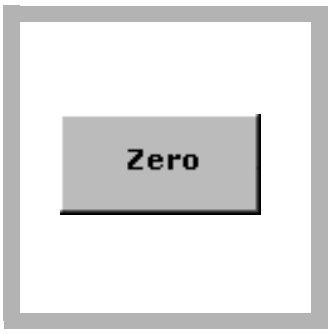
Install the Light Shield in Cell Compartment #2.



2. Remove the cap from one HR Monochloramine Diluent vial. Use a glass pipet to add 2.0 mL sample to the vial. Re-cap and invert several times to mix.



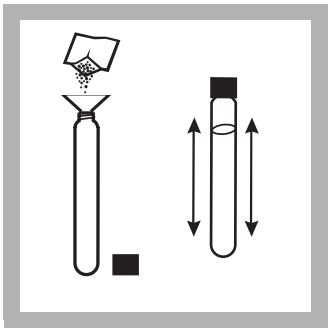
3. Wipe the vial and insert it into the 16 mm cell holder.



4. Press **ZERO**.

The display will show:

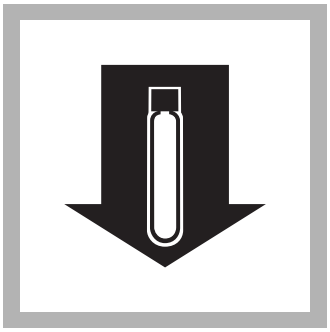
0.0 mg/L Cl_2



5. Remove the vial from the holder. Using a micro-funnel, add the contents of one Monochlor F pillow to the sample. Cap and shake the cell about 20 seconds to dissolve.

6. Press **TIMER>OK**.

A 5-minute reaction period will begin.



7. After the timer expires, re-insert the vial into the cell holder.

8. Press **READ**.

Results are in mg/L Cl_2.

Interferences

The substances in Table 1 have been tested for interference and do not interfere at or below the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br^-
Bromine	15 mg/L Br_2
Calcium	1000 mg/L as $CaCO_3$
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO_2
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Dichloramine	10 mg/L as Cl_2
Fluoride	5 mg/L
Free Chlorine	10 mg/L Cl_2
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	1000 mg/L as $CaCO_3$
Manganese (VII)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO_4

Table 1 Non-interfering Substances (continued)

Substance	Maximum Level Tested
Silica	100 mg/L SiO_2
Sulfate	2600 mg/L
Sulfite	50 mg/L SO_3^{2-}
Tyrosine	1 mg/L N
Urea	10 mg/L N
Zinc	5 mg/L

Table 2 describes suggested treatments for interfering substances.

Table 2 Interfering Substances and Suggested Treatments

Interfering Substance and Effects		Interference Level	Recommended Treatment
Ozone	–	Above 1 mg/L	Usually does not coexist with monochloramine.
Sulfide	+	Turns a “rust” color if present.	Usually does not coexist with monochloramine.
Thiocyanate	–	Above 0.5 mg/L	This method will tolerate up to 2 mg/L.

Sampling and Storage

Analyze samples for monochloramine immediately after collection. Rinse the sample container several times with sample, letting the container overflow each time. If sampling from a tap, let the water flow for at least 5 minutes, then cap the container so that there is no head space (air) above the sample.

Accuracy Check

To check test accuracy, prepare the following 4.5-mg/L (as Cl_2) monochloramine standard immediately before use.

1. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.

2. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N into the flask.

3. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00-mg/L buffered ammonia standard.

4. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

5. Obtain a recent lot of Chlorine Solution Ampules, 50–75 mg/L, and note the actual free chlorine concentration for this lot.

6. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

7. Open an ampule and use a glass Mohr pipet to add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir-plate.

8. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.

9. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5-mg/L (as Cl_2) monochloramine standard.

10. Use this standard within 1 hour of preparation. Analyze according to the Low Range Monochloramine procedure described above.

11. To adjust the calibration curve using the reading obtained with the 4.5-mg/L standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

12. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The sample is first diluted in a Test ‘N Tube. In the presence of a cyanoferrate catalyst, monochloramine (NH_2Cl) in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
HR Monochloramine Test 'N Tubes, 50 tests, includes:	—	—	28051-45
(50) HR Monochloramine Diluent Vials[1]	1	50/pkg	—
Funnel, micro	1	each	25843-35
Monochlor F Reagent Pillows	1	50/pkg	28022-46

[1] Not sold separately

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipet, Mohr, glass 2.00 mL	1	each	20936-36
Safety Bulb	—	each	14651-00
Test Tube Rack	1–2	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Buffer Powder Pillows, pH 8.3	25/pkg	898-68
Chlorine Solution Voluette® Ampule, 50–75 mg/L	16/pkg	14268-10
Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N	500 mL	24065-49
Water, organic-free	500-mL	26415-49

Optional Reagents and Apparatus

Description	Cat. No.
Beaker, 100 mL	500-42H
Clippers	968-00
Flask, volumetric, Class A, 100-mL	14574-42
Pipet, Mohr, glass, 5-mL	20934-37
Pipet, volumetric, Class A, 2.00-mL	14515-36
Pipet, volumetric, Class A, 50.00-mL	14515-41
Safety Bulb	14651-00
Stir bar, octagonal	20953-52
Stirrer, magnetic	28812-00
Shears	23694-00

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Updated November 2005 Edition 1

Chloramine (Mono)

Method 10171

Powder Pillows

Indophenol Method[1]

LR (0.04 to 4.50 mg/L Cl_2)

Scope and Application: Chloraminated drinking water and chlorinated wastewater

[1] U.S. Patent 6,315,950

Test Preparation

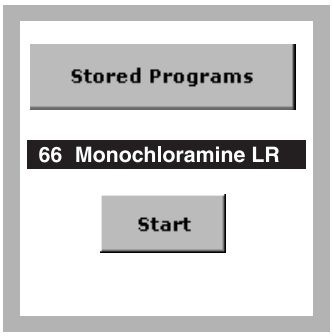
Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
To determine chloramine (mono) and free ammonia on the same sample, use Method 10200 Nitrogen, Free Ammonia and Chloramine (Mono)
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

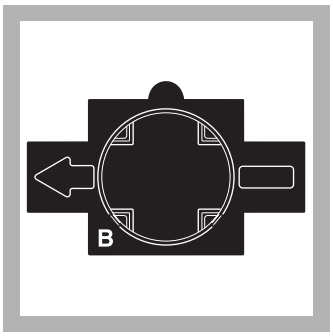
Collect the following items:	**Quantity**
Monochlor F Reagent Pillow	1
Sample Cell	1

Note: *Reorder information for consumables and replacement items is on page 5.*

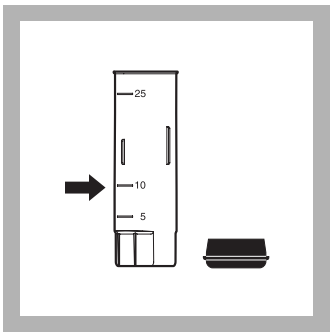
Multi-path Cell — Method 10171



1. Select the test.

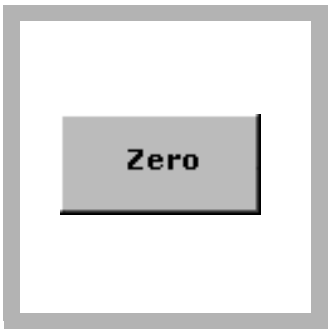


2. Insert Adapter B.



3. Fill the cell to the 10-mL line with sample.

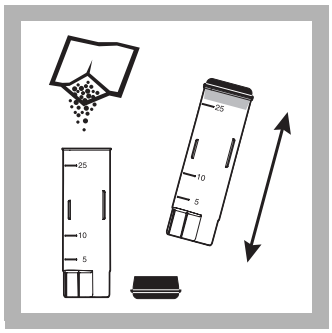
4. Wipe the cell and insert it into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.



5. Press **ZERO**.

The display will show:

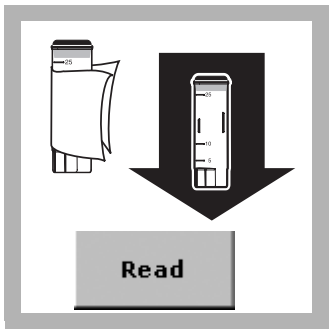
0.00 mg/L Cl_2



6. Remove the cell and add the contents of one Monochlor-F pillow to the sample. Cap and shake the cell about 20 seconds to dissolve.

7. Press **TIMER>OK**.

A 5-minute reaction period will begin. Samples colder than 18 °C will require additional time. See Table 3.



8. After the timer expires, insert the vial into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.

Press **READ**. Results are in mg/L Cl_2.

Interferences

The substances in Table 1 have been tested for interference and do not interfere at or below the indicated levels. Table 2 suggests treatmens for interferences.

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br^-
Bromine	15 mg/L Br_2
Calcium	1000 mg/L as $CaCO_3$
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO_2
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Dichloramine	10 mg/L as Cl_2
Fluoride	5 mg/L
Free Chlorine	10 mg/L Cl_2
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO_4
Silica	100 mg/L SiO_2
Sulfate	2600 mg/L

Table 1 Non-interfering Substances (continued)

Substance	Maximum Level Tested
Sulfite	50 mg/L SO_3^{2-}
Tyrosine	1 mg/L N
Urea	10 mg/L N
Zinc	5 mg/L

Table 2 Interfering Substances and Suggested Treatments

Interfering Substance and Effects		Interference Level	Recommended Treatment
Magnesium	+	Above 400 mg/L $CaCO_3$	Add 5 drops Rochelle Salt Solution prior to testing. OR: use the high range (HR) test.
Magnesium (+7)	–	Above 3 mg/L	Use the HR test; it will tolerate up to 10 mg/L.
Ozone	–	Above 1 mg/L	Usually does not coexist with monochloramine.
Sulfide	+	Turns a “rust” color if present.	Usually does not coexist with monochloramine.
Thiocyanate	–	Above 0.5 mg/L	This method will tolerate up to 2 mg/L.

Table 3 Color Development Based on Sample Temperature

Sample Temperature		Development Time (minutes)
°C	°F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	4
20	73	3
23	75	2.5
25	77	2
greater than 25	greater than 77	2

Sampling and Storage

Analyze samples for monochloramine immediately after collection. Rinse the sample container several times with sample, letting the container overflow each time. If sampling from a tap, let the water flow for at least 5 minutes, then cap the container so that there is no head space (air) above the sample.

Accuracy Check

To check test accuracy, prepare the following 4.5-mg/L (as Cl_2) monochloramine standard immediately before use.

1. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.

2. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N into the flask.

3. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00-mg/L buffered ammonia standard.

4. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

5. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.

6. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

7. Open an ampule and use a glass Mohr pipet to add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir-plate.

8. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.

9. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5-mg/L (as Cl_2) monochloramine standard.

10. Use this standard within 1 hour of preparation. Analyze according to the Low Range Monochloramine procedure described above.

11. To adjust the calibration curve using the reading obtained with the 4.5-mg/L standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

12. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

In the presence of a cyanoferrate catalyst, monochloramine (NH_2Cl) in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Monochlor F Reagent Pillows	1	50/pkg	28022-46

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Sample Cell, multi-path	1	6/pkg	59405-06

Optional Reagents

Description	Unit	Cat. No.
Buffer Powder Pillows pH 8.3	25/pkg	898-68
Chlorine Solution Voluette® Ampule 50–75 mg/L	16/pkg	14268-10
Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N	500 mL	24065-49
Organic-free Water	500-mL	26415-49
Rochelle Salt Solution	29 mL DB	1725-33

Optional Apparatus

Description	Cat. No.
Beaker, 100 mL	500-42H
Clippers	968-00
Flask, volumetric, Class A, 100-mL	14574-42
Pipet, Mohr, glass, 5-mL	20934-37
Pipet, volumetric, Class A, 2.00-mL	14515-36
Pipet, volumetric, Class A, 50.00-mL	14515-41
Stir Bar, octagonal	20953-52
Stirrer, magnetic	28812-00
Shears	23694-00

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Updated November 2005 Edition 1

Chloramine (Mono); Nitrogen, Free Ammonia

Method 10200 **Indophenol Method[1]**

Powder Pillows **(0.01–0.50 mg/L NH_3–N; 0.04–4.50 mg/L Cl_2)**

Scope and Application: For determining Free Ammonia and Monochloramine simultaneously in finished chloraminated water

[1] U.S. Patent 6,315,950

Test Preparation

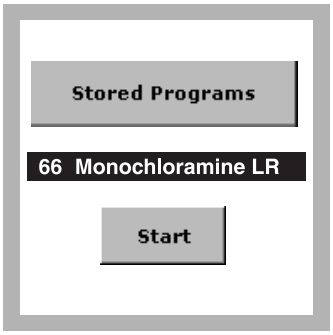
Before starting the test:

For more accurate chloramine results, determine a reagent blank value for each new lot of reagent, using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

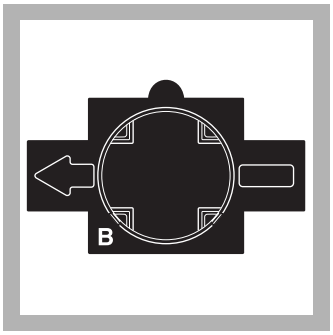
Collect the following items:	**Quantity**
Free Ammonia Reagent Solution	1 drop
Monochlor F Reagent Pillows	2
Sample cell, 10-mL, 1-cm	2

Note: *Reorder information for consumables and replacement items is on page 8.*

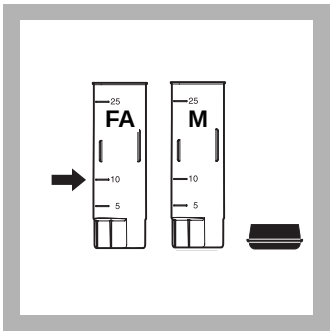
Multi-path Cell Method 10200



1. Select the Monochloramine test.

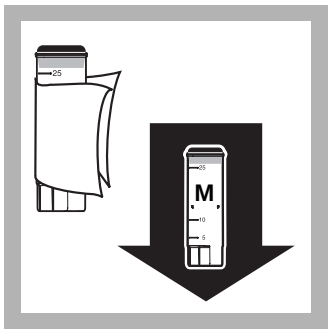


2. Insert Adapter B.

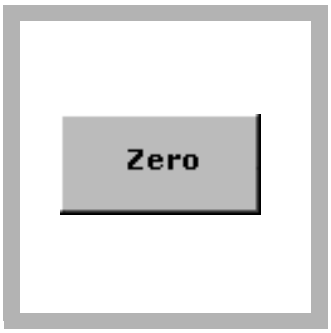


3. Fill two 1-cm cell to the 10-mL line with sample.

Label one cell “Free Ammonia” and one cell “Monochloramine”.



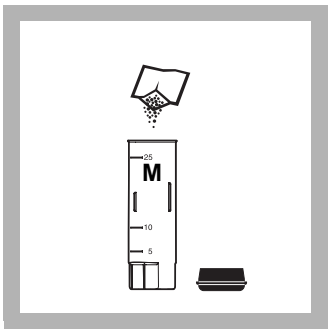
4. Wipe the monochloramine cell and insert it into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.



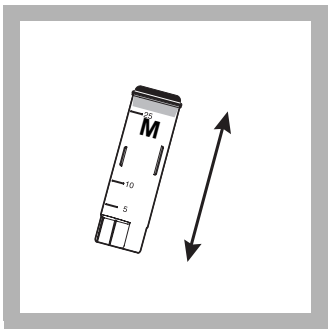
5. Press **ZERO**.

The display will show:

0.00 mg/L Cl_2

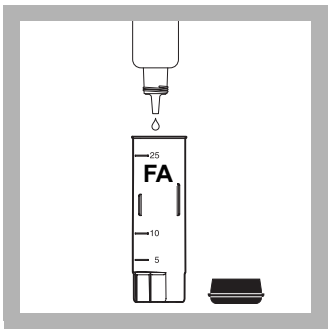


6. Remove the cell and add the contents of one pillow Monochlor-F to the sample for monochloramine measurement.



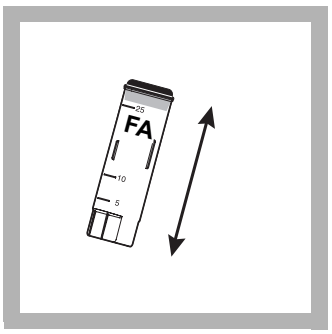
7. Cap and shake the cell about 20 seconds to dissolve.

A green color will develop if monochloramine is present.



8. Add one drop of Free Ammonia Reagent Solution to the cell for Free Ammonia measurement.

9. Cap the reagent bottle to maintain reagent performance and stability.



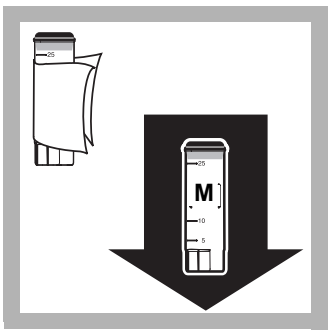
10. Cap and invert the Free Ammonia cell to mix.

If the sample becomes cloudy by the end of the reaction period, pretreat the sample and retest. See Interferences on page 3.

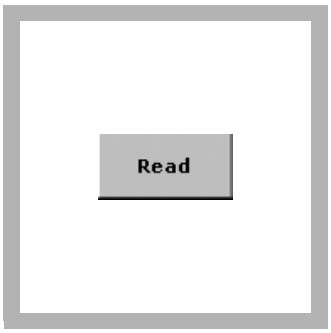
11. Press **TIMER>OK**.

A 5-minute reaction period will begin.

Color development time depends on sample temperature. For accurate results allow the full reaction period to occur. See Table 1 on page 4.



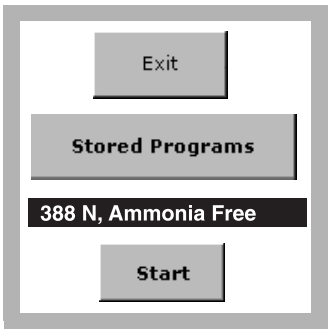
12. When the timer expires, wipe the **Monochloramine** cell and insert it into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.



13. Press **READ**.

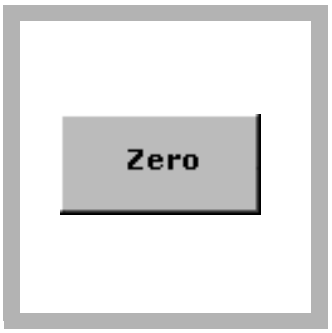
Results are in mg/L Monochloramine (as Cl_2).

Leave the cell in the instrument.



14. Select the Free Ammonia test.

If Display Lock is on, the display will show “Store Data?”. Press **YES** or **NO** and continue.

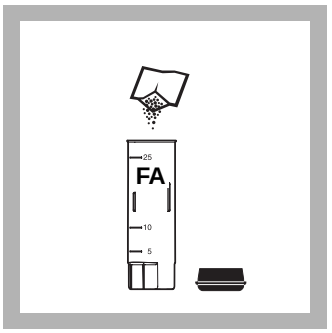


15. With the monochloramine sample still in the cell holder, press **ZERO**.

The display will show:

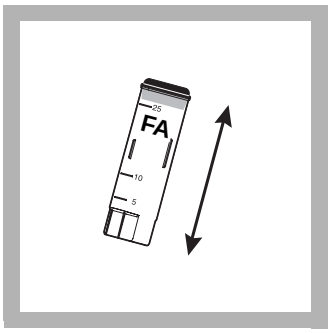
0.00 mg/L NH_3–N f

Remove the monochloramine cell.



16. Add the contents of one pillow Monochlor F to the cell for the Free Ammonia measurement.

The reaction period in step 11 must be complete before adding the Monochlor F to the cell for Free Ammonia measurement.



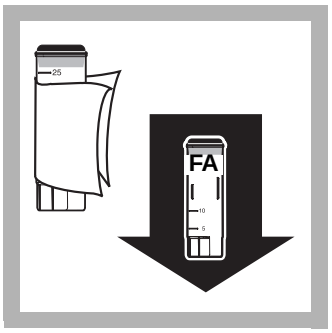
17. Cap and shake the cell about 20 seconds to dissolve the reagent.

A green color will form if monochloramine or ammonia is present.

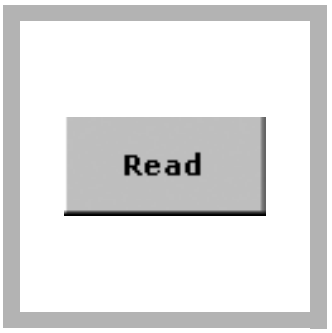
18. Press **TIMER>OK**.

A 5-minute reaction period will begin.

Samples colder than 18 °C will require additional time. See Table 2 on page 4.



19. When the timer expires, insert the vial into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.



20. Press **READ**.

Results are in mg/L NH_3–N f.

Interferences

This method is intended for finished, chloraminated drinking water samples that have a measurable combined (total) chlorine disinfectant residual. Samples where the disinfectant residual has disappeared and samples which exhibit a chlorine demand may produce low ammonia test results. Blanks and ammonia standards analyzed without a disinfectant residual must be prepared using high quality, reagent grade water.

The following do not interfere in free ammonia determination at or below the stated concentration:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	0.2 mg/L
Chloride	1200 mg/L Cl
Copper	1 mg/L Cu
Iron	0.3 mg/L Fe
Manganese	0.05 mg/L Mn
Nitrate	10 mg/L NO_3–N
Nitrite	1 mg/L NO_2–N
Phosphate	2 mg/L o–PO_4
Silica	100 mg/L SiO_2
Sulfate	1600 ppm as $CaCO_3$
Zinc	5 ppm Zn

Samples containing high levels of both Total Hardness and Alkalinity may become cloudy after the addition of the Free Ammonia Reagent Solution. If this occurs by the end of the first reaction period, the sample for Free Ammonia measurement must be pretreated as follows:

Note: *The sample for Monochloramine measurement does not need pretreatment.*

1. Measure 10 mL of sample into the cell for Free Ammonia measurement.
2. Add the contents of one Hardness Treatment Reagent Powder Pillow to the sample.
3. Cap the cell and invert until the reagent is dissolved.
4. Remove the cap.
5. Continue with the analysis at step 3 using the pretreated sample as the Free Ammonia cell.

Color Development Time

Test results are strongly influenced by sample temperature. **Both reaction periods in the procedure are the same and depend on the temperature of the sample.** The reaction periods indicated in the procedure are for a sample temperature of 18–20 °C (64–68 °F). Adjust both reaction periods according to Table 2.

Table 2 Color Development Based on Sample Temperature

Sample Temperature		Development Time (minutes)
°C	°F	
5	41	10
7	45	9
9	47	8
10	50	8
12	54	7

Table 2 Color Development Based on Sample Temperature (continued)

Sample Temperature		Development Time (minutes)
°C	°F	
14	57	7
16	61	6
18	64	5
20	68	5
23	73	2.5
25	77	2
greater than 25	greater than 77	2

Sampling and Storage

Collect samples in clean glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

Accuracy Check (Monochloramine, Program 66)

Standard Solution Method
Important Note: *Because of the strong buffer used in the preparation of this standard, it cannot be used for accuracy verification of the Free Ammonia test.*

To check test accuracy, prepare the following 4.5-mg/L (as Cl_2) monochloramine standard immediately before use.

1. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.

2. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N into the flask.

3. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00-mg/L buffered ammonia standard.

4. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

5. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.

6. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

7. Open an ampule and use a glass Mohr pipet to add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir-plate.

8. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.

9. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5-mg/L (as Cl_2) monochloramine standard.

10. Use this standard within 1 hour of preparation. Analyze according to the Low Range Monochloramine procedure described above.

11. To adjust the calibration curve using the reading obtained with the 4.5-mg/L standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

12. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Accuracy Check (Free Ammonia, Program 388)

Dilution water is required when testing a diluted sample and preparing standard solutions. Dilution water must be free of ammonia, chlorine and chlorine demand. A convenient source is a recirculating, deionizer system with carbon filtration which produces 18 megaohm-cm water.

Standard Additions Method

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentrations, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare three spiked samples. Measure 50 mL of sample into three 50-mL mixing cylinders.

5. Use the TenSette Pipet to add 0.3, 0.6, and 1.0 mL of Ammonium Nitrogen Standard, 10 mg/L as NH_3-N to the three samples. Mix well.

6. Analyze each spiked sample starting with the 0.3 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery. Follow all steps in Method 10200.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 0.20 mg/L ammonia nitrogen standard by diluting 2.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with dilution water. Or, using the TenSette Pipet, prepare a 0.20 mg/L ammonia nitrogen standard by diluting 0.4 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3–N, to 100 mL with dilution water. Analyze the Standard Solution, following all steps in Method 10200.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Monochloramine (NH_2Cl) and free ammonia (NH_3 and NH_4^+) can exist in the same water sample. Added hypochlorite combines with free ammonia to form more monochloramine. In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Free ammonia is determined by comparing the color intensities, with and without added hypochlorite. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Free Ammonia Reagent Set (50 tests), includes:	—	—	28797-00
(1) 28022-99, (1) 28773-36			
Free Ammonia Reagent Solution	1 drop	4 mL SCDB	28773-36
Monochlor F Reagent Pillows	2	100/pkg	28022-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cell, multi-path	1	6/pkg	59405-06

Recommended Standards and Reagents

Description	Unit	Cat. No.
Buffer, pH 8.3 Powder Pillows	25/pkg	898-68
Chlorine Solution Voluette® Ampule	16/pkg	14268-10
Hardness Treatment Reagent Pillows (1 per test)	50/pkg	28823-46
Nitrogen Ammonia Standard Solution, 10 mg/L as NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Ampule, 50 mg/L as NH_3–N, 10 mL	16/pkg	14791-10
Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N	500 mL	24065-49
Water, Organic-free water	500-mL	26415-49

Recommended Apparatus

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Beaker, 100 mL, Polypropylene	each	1080-42
Beaker, 100 mL, Glass	each	500-42H
Cylinder, 50 mL, mixing	each	20886-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet Filler, safety bulb	each	14651-00
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet, Mohr, Glass, 10 mL	each	20934-38
Pipet, volumetric, Class A, 2.0 mL	each	14515-36
Pipet, volumetric, Class A, 50.00 mL	each	14515-41
Scissors	each	28831-00
Stir Bar, octagonal	each	20953-52
Stirrer, magnetic	each	28812-00
Thermometer, –10 to 110 °C	each	1877-01
Wipers, Disposable Kimwipes®, 30 x 30 cm, 280/box	box	20970-00

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Updated November 2005 Edition 1

Chloride

Method 8113

Mercuric Thiocyanate Method
(0.1 to 25.0 mg/L Cl^-)

Scope and Application: For water and wastewater

Test Preparation

Before starting the test:

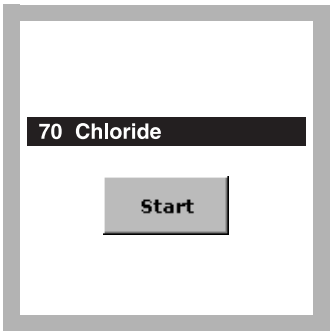
Filter turbid samples with moderately rapid filter paper and a funnel before analysis.
Both the sample and the blank will contain mercury (D009) at a concentration regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain.
Refer to the MSDS sheet for safe handlng and disposal of hazardous waste.

Collect the following items:	**Quantity**
Ferric Ion Solution	1 mL
Mercuric Thiocyanate Solution	2 mL
Deionized Water	10 mL
Sample Cells, 1-inch square, 10-mL	2
Pipet, TenSette®, 0.1 to 1.0 mL	1
Pipet tips for 0.1 to 1.0 mL TenSette pipet	2

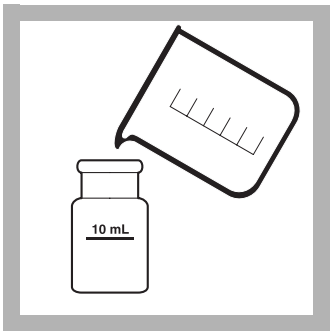
Note: *Reorder information for consumables and replacement items is on page 4.*

Mercuric Thiocyanate — Method 8113

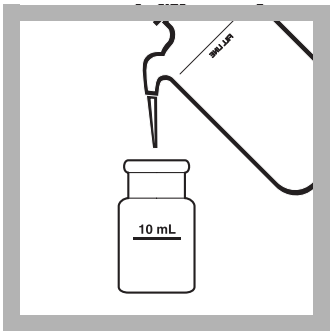
1. Press **STORED PROGRAMS**.



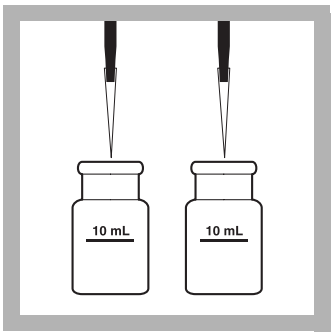
2. Select the test.



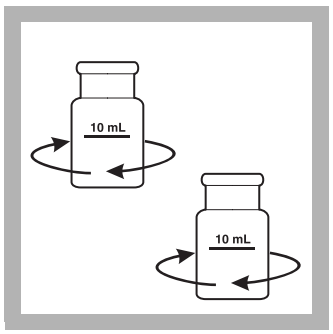
3. Prepared Sample: Fill a square sample cell with 10 mL of sample.



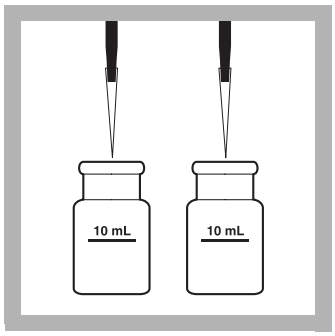
4. Blank Preparation: Fill another square sample cell with 10 mL of deionized water.



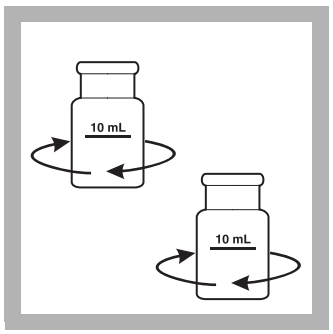
5. Pipet 0.8 mL of Mercuric Thiocyanate Solution into each sample cell.



6. Swirl to mix.



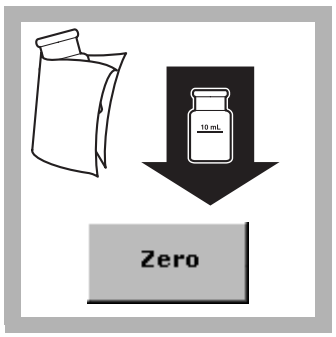
7. Pipet 0.4 mL of Ferric Ion Solution into each sample cell.



8. Swirl to mix. An orange color will develop if chloride is present.

9. Press **TIMER>OK**.

A two-minute reaction time will begin.



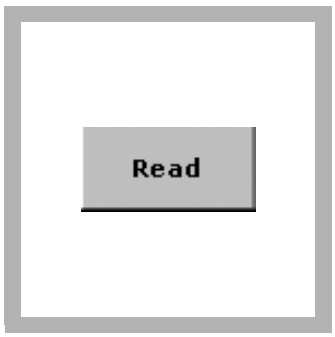
10. Within five minutes after the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

0.0 mg/L Cl⁻

11. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.



12. Press **READ**.

Results are in mg/L Cl⁻.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Extreme pH	Should be about pH 2 after adding reagents. If the sample is strongly acidic or alkaline, adjust a portion of sample before testing to a pH of about 7. Use either 5.0 N Sodium Hydroxide Standard Solution[1] or a 1:5 dilution of perchloric acid. **Use pH paper**; most pH electrodes will contaminate the sample with chloride.

[1] See Optional Reagents and Apparatus on page 4.

Sample Collection, Storage, and Preservation

Collect samples in glass or plastic containers. Samples can be stored for at least 28 days at room temperature.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare three sample spikes. Fill three 50 mL mixing cylinders with 50 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of 1000-mg/L Chloride Standard Solution, respectively, to the cylinders and mix each thoroughly.

5. Analyze a 10 mL portion of each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

6. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

Prepare a 20.0-mg/L chloride standard solution as follows:

1. Using Class A glassware, pipet 10.00 mL of Chloride Standard Solution*, 1000-mg/L, into a 500-mL volumetric flask.

2. Dilute to the mark with deionized water. Perform the chloride procedure as described above.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

*See Optional Reagents and Apparatus on page 4.

Summary of Method

Chloride in the sample reacts with mercuric thiocyanate to form mercuric chloride and liberate thiocyanate ion. Thiocyanate ions react with the ferric ions to form an orange ferric thiocyanate complex. The amount of this complex is proportional to the chloride concentration. Test results are measured at 455 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Chloride Reagent Set (50 Tests)[1], includes:	—	each	23198-00
(1) Ferric Ion Solution	1 mL	100 mL	22122-42
(1) Mercuric Thiocyanate Solution	2 mL	200 mL	22121-29
Water, deionized	10 mL	4 L	272-56

[1] 50 tests equals 25 samples and 25 blanks.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10-mL	2	2/pkg	24954-02
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	varies	50/pkg	21856-96

Recommended Standards

Description	Unit	Cat. No.
Chloride Standard Solution, 1000-mg/L Cl^-	500 mL	183-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Chloride Standard Solution, 2-mL Voluette® Ampule, 12,500-mg/L Cl^-	20/pkg	14250-20
Cylinders, mixing	50 mL	1896-41
Filter Paper, funnel	100/pkg	692-57
Funnel, poly	75 mm	1083-68
Perchloric Acid, ACS	—	757-65
pH Paper	—	391-33
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A	1 mL	14515-35
Pipet, volumetric, Class A	0.5 mL	14515-37
Pipet Filler, safety bulb	—	14651-00
Sodium Hydroxide Standard Solution, 50 mL SCDB	50 mL	2450-26

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Updated November 2005 Edition 1

Chlorine Dioxide

Method 8138

Direct Reading Method

HR (5 to 1000 mg/L)

Scope and Application: For water and wastewater

Test Preparation

Before starting the test:

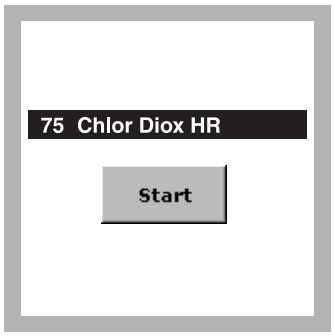
Chlorine dioxide is unstable and volatile. Analyze samples immediately.

Collect the following items:	**Quantity**
Water, deionized	10 mL
Sample cell, 1-inch square glass, 10-mL	2

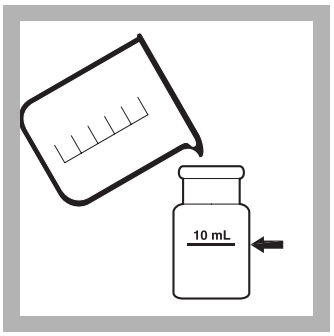
Note: *Reorder information for consumables and replacement items is on page 2.*

Direct Reading Method 8138

1. Press **STORED PROGRAM**.

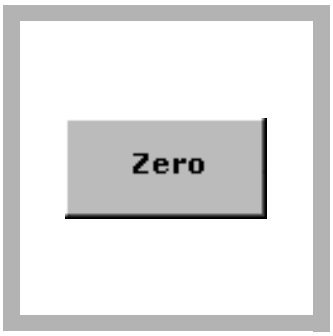


2. Select the test.



3. Blank Preparation: Fill a square sample cell to the 10-mL mark with deionized water.

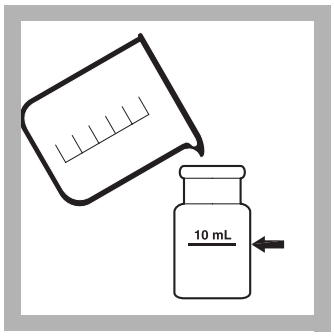
4. Wipe the blank and insert it into the cell holder with the fill line facing right.



5. Press **ZERO**.

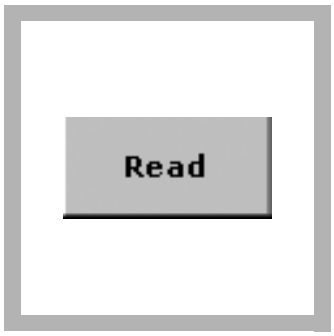
The display will show:

0 mg/L ClO_2



6. Prepared Sample: Fill a second square sample cell to the 10-mL mark with sample.

7. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.



8. Press **READ**.

Results are in mg/L ClO_2.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine dioxide immediately after collection. Chlorine dioxide is a strong oxidizing agent and is unstable in natural waters. It reacts rapidly with various inorganic compounds, but oxidizes organic compounds more slowly. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of chlorine dioxide in water.

Avoid plastic containers since these may have a large chlorine dioxide demand. Pretreat glass sample containers to remove any chlorine or chlorine dioxide demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine dioxide is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the chlorine dioxide analysis immediately.

Accuracy Check

Standard Solution Method

Preparing chlorine dioxide standards is difficult and dangerous. In addition, these standards are both explosive and volatile! Only a trained chemist should prepare the standards using appropriate safety equipment and precautions. The manufacturer does not recommend preparation of chlorine dioxide standards. If independent standard preparation is required, please see the instructions in *Standard Methods for the Examination of Water and Wastewater*, 20th ed., under the headings “Stock chlorine dioxide solution” and “Standard chlorine dioxide solution” (pp 4–74 and 4–75). Prepare a 500-mg/L chlorine dioxide standard.

Summary of Method

Chlorine dioxide, a yellow gas, can be measured directly in a water solution. Test results are measured at 445 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Water, deionized	10 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, glass,10 mL, matched pair	2	2/pkg	24954-02

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Updated November 2005 Edition 1

Chlorine Dioxide

Method 8065

Chlorophenol Red Method[1]

LR (0.01 to 1.00 mg/L)

Scope and Application: For water and wastewater

[1] Adapted from Harp, Klein, and Schoonover, *Jour. Amer. Water Works Assn.*, 73 387–388 (1981).

Test Preparation

Before starting the test:

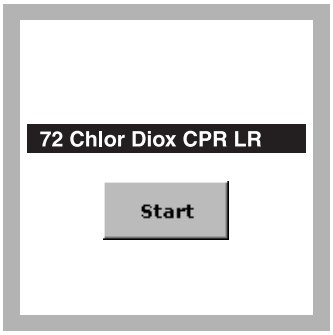
Chlorine dioxide is unstable and volatile. Analyze samples immediately.
For most accurate results, analyze each portion of sample at the same temperature.
A TenSette® Pipet may be used to dispense Chlorine Dioxide Reagent 1 and Chlorine Dioxide Reagent 3

Collect the following items:	**Quantity**
Chlorine Dioxide Reagent 1	2 mL
Chlorine Dioxide Reagent 2	2 mL
Chlorine Dioxide Reagent 3	2 mL
Dechlorinating Reagent Pillows	1
Cylinder, graduated mixing, 50 mL	2
Pipet, volumetric, Class A, 1 mL	3
Pipet Filler, with safety bulb	1
Sample Cells, 1-inch square, 10-mL	2

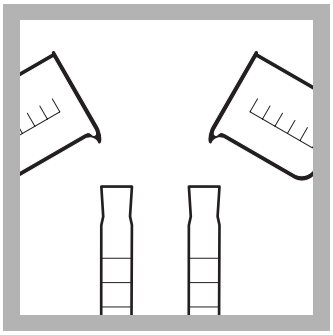
Note: *Reorder information for consumables and replacement items is on page 4.*

Powder Pillows — Method 8065

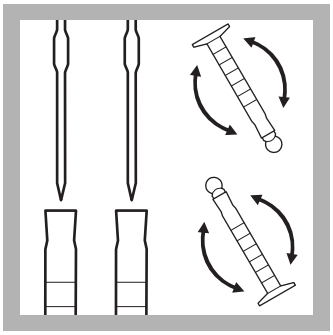
1. Press **Stored Programs**.



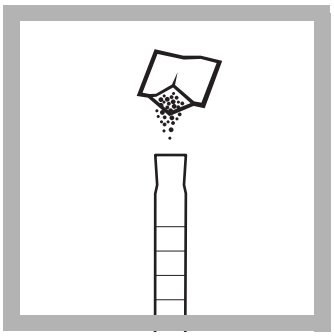
2. Select the test.



3. Fill two 50-mL mixing cylinders to the 50-mL mark with sample.



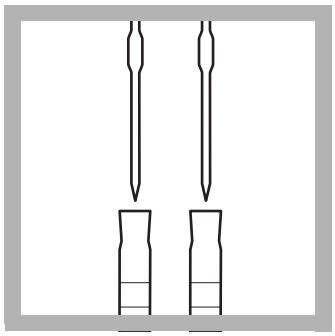
4. Use a volumetric pipet and pipet filler to add 1.0 mL of Chlorine Dioxide Reagent 1 to each cylinder. Stopper. Invert several times to mix.



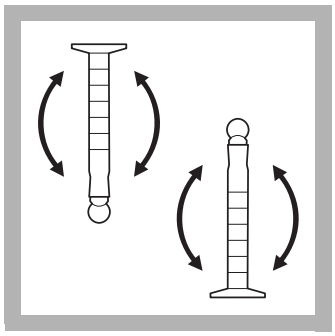
5. Blank Preparation: Add the contents of one Dechlorinating Reagent Powder Pillow to one cylinder. (This is the blank).

Stopper and invert several times until dissolved.

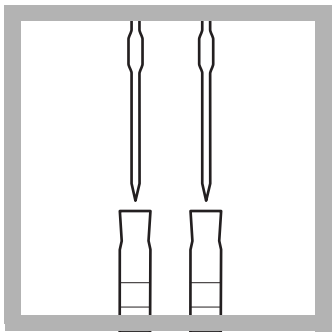
The second cylinder, which does **not** receive dechlorinating reagent, is the prepared sample.



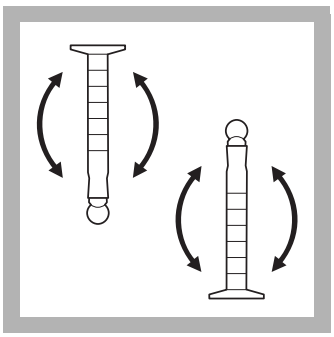
6. Use a volumetric pipet to add exactly 1.00 mL of Chlorine Dioxide Reagent 2 to each cylinder. Stopper.



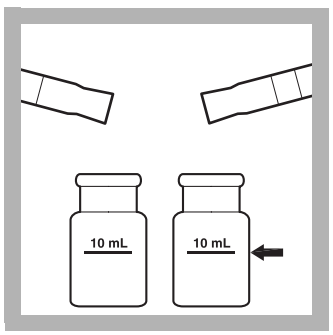
7. Invert several times to mix.



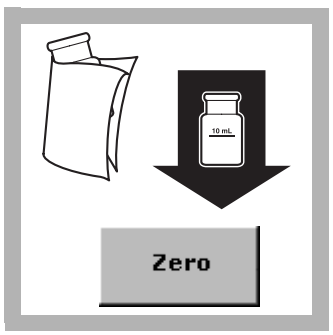
8. Use a volumetric pipet and pipet filler to add 1.0 mL of Chlorine Dioxide Reagent 3 to each cylinder. Stopper.



9. Invert several times to mix.



10. Pour 10 mL from each cylinder into square sample cells.



11. Wipe the blank and insert it into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show: 0.00 mg/L ClO_2

12. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**.

Results are in mg/L ClO_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Highly acidic or alkaline water	May require 2.0 mL each of Chlorine Dioxide Reagent 1 and Chlorine Dioxide Reagent 3 instead of 1.0 mL
ClO^-	Greater than 5.5 mg/L
ClO_2^-	Greater than 6 mg/L
ClO_3^-	Greater than 6 mg/L
CrO_4^{2-}	Greater than 3.6 mg/L
Fe^{3+}	Greater than 5 mg/L
Hardness	Greater than 1000 mg/L
Ozone	Greater than 0.5 mg/L
Turbidity	Greater than 1000 NTU

Sample Collection, Storage, and Preservation

Analyze samples for chlorine dioxide immediately after collection. Chlorine dioxide is a strong oxidizing agent and is unstable in natural waters. It reacts rapidly with various inorganic compounds, but oxidizes organic compounds more slowly. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of chlorine dioxide in water.

Avoid plastic containers since these may have a large chlorine dioxide demand. Pretreat glass sample containers to remove any chlorine or chlorine dioxide demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine dioxide is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample.

Accuracy Check

Standard Solution Method

Preparing chlorine dioxide standards is difficult and dangerous. In addition, these standards are both explosive and volatile! Only a trained chemist should prepare the standards using appropriate safety equipment and precautions. The Manufacturer does not recommend preparation of chlorine dioxide standards. If independent standard preparation is required, please see the instructions in *Standard Methods for the Examination of Water and Wastewater*, 20th ed., under the headings “Stock chlorine dioxide solution” and “Standard chlorine dioxide solution” (pp 4–74 and 4–75). Prepare a 0.50-mg/L chlorine dioxide standard.

Summary of Method

Chlorine Dioxide (ClO_2) is determined by its combination with chlorophenol red at pH 5.2 to form a colorless complex. The net effect is bleaching of the color in an amount proportional to the chlorine dioxide concentration. The method is specific for ClO_2 and is unreactive to other active chlorine or moderate oxidizing compounds. Test results are measured at 575 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Chlorine Dioxide Reagent Set (100 Tests), includes:	—	each	22423-00
(2) Chlorine Dioxide Reagent 1	2 mL	100 mL	20700-42
(2) Chlorine Dioxide Reagent 2	2 mL	100 mL	20701-42
(2) Chlorine Dioxide Reagent 3	2 mL	100 mL	20702-42
(1) Dechlorinating Reagent Powder Pillows	1	100/pkg	14363-69

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated mixing, 50-mL	2	each	1896-41
Pipet, volumetric, Class A, 1.00-mL	3	each	14515-35
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96

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Updated November 2005 Edition 1

Chlorine Dioxide

Amaranth Method[1]

(20 to 500 µg/L)

Scope and Application: For water, drinking water

[1] This method is under license of Elf Atofina. Reagent sets for this method are only available in Europe.

Test Preparation

Before starting the test:

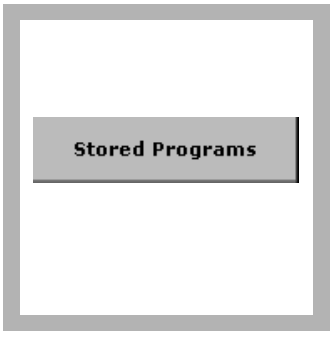
Chlorine dioxide is unstable and volatile. Analyze samples immediately. See Sample Collection, Storage, and Preservation on page 3.
For most accurate results, analyze each portion of sample at the same temperature.
A TenSette® pipet may be used to dispense Chlorine Dioxide Reagent A.

Collect the following items:	**Quantity**
Chlorine Dioxide Reagent Set	1
Volumetric Flask, 25-mL plastic	2
Syringe, 1-mL with needle	1
Sample cell, 1-inch square glass, 10-mL	2

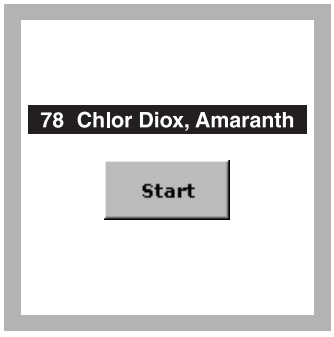
Note: *Reorder information for consumables and replacement items is on page 4.*

Note: *For best precision, measurement of the reagent with a volumetric pipet or high precision pipettor is recommended.*

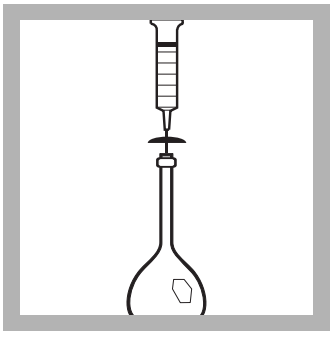
Amaranth Method



1. Press **STORED PROGRAMS**.

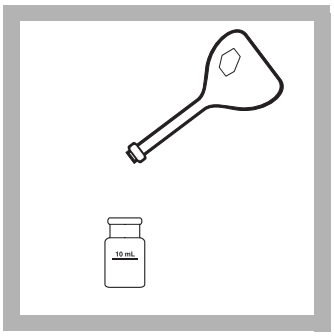


2. Select the test.

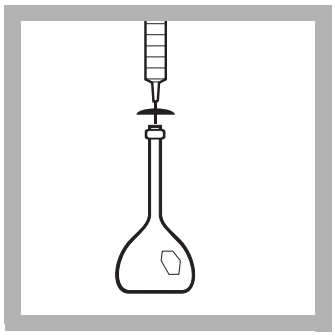


3. Blank Preparation: Using the syringe and needle provided, add 1.0 mL of Chlorine Dioxide Reagent A into a 25-mL volumetric flask.

4. Fill the volumetric flask to the mark with deionized water. Stopper. Invert at least 7 times to mix.

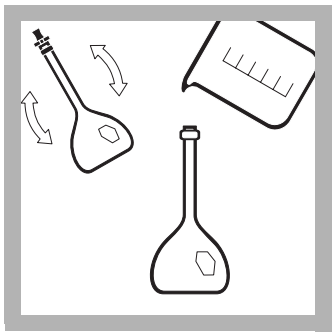


5. Pour 10 mL from the volumetric flask into a 10 mL sample cell.



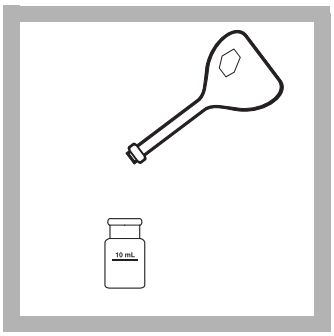
6. Prepared Sample: Add 1.0 mL of Chlorine Dioxide Reagent A into a second 25-mL volumetric flask.

Use a volumetric pipet and pipet filler or a TenSette Pipet to add this reagent.

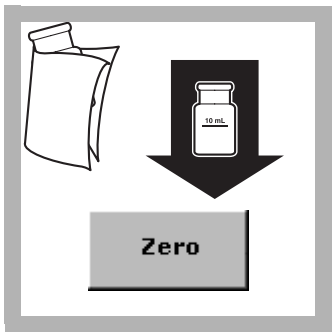


7. Fill the second volumetric flask to the mark with the sample. Stopper. Invert at least 7 times to mix.

8. Press **TIMER>OK**. A 1-minute reaction period will begin.



9. Prepared Sample: Pour 10 mL from the second volumetric flask into a second sample cell.



10. Wipe the blank and insert it into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

0 µg/L ClO_2

11. When the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing right.

12. Press **READ**.

Results are in µg/L ClO_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
ClO^-	Greater than 2.0 mg/L
ClO_2^-	Greater than 2.0 mg/L
ClO_3^-	Greater than 2.0 mg/L
CrO_4^{2-}	Greater than 0.2 mg/L
Fe^{3+}	Greater than 0.5 mg/L
Hardness	Greater than 1000 mg/L
Ozone	Greater than 0.5 mg/L
Turbidity	Greater than 1000 NTU

Sample Collection, Storage, and Preservation

Analyze samples for chlorine dioxide immediately after collection. Chlorine dioxide is a strong oxidizing agent and is unstable in natural waters. It reacts rapidly with various inorganic compounds, but oxidizes organic compounds more slowly. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of chlorine dioxide in water.

Avoid plastic containers since these may have a large chlorine dioxide demand. Pretreat glass sample containers to remove any chlorine or chlorine dioxide demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine dioxide is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample.

Accuracy Check

Standard Solution Method

Preparing chlorine dioxide standards is difficult and dangerous. In addition, these standards are both explosive and volatile! Only a trained chemist should prepare the standards using appropriate safety equipment and precautions. The manufacturer does not recommend preparation of chlorine dioxide standards. If independent standard preparation is required, please see the instructions in *Standard Methods for the Examination of Water and Wastewater*, 20th ed., under the headings “Stock chlorine dioxide solution” and “Standard chlorine dioxide solution” (pp 4–74 and 4–75). Prepare a 0.25-mg/L (250-µg/L) chlorine dioxide standard.

Summary of Method

Chlorine dioxide (ClO_2) is determined by its combination with Amaranth. Color intensity decreases as the level of chlorine dioxide increase. Test results are measured at 521 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Chlorine Dioxide Reagent Set (100 Tests)[1]	1	100/pkg	LYW 240

[1] Available only in Europe.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Chlorine Dioxide Tool Set, includes:	—	each	LZC 140
(2) Flask, volumetric, plastic, 25-mL	2	each	—
(1) Syringe, 1-mL, with needle	1	each	—
Sample Cells, 1-inch square glass, 10-mL	2	2/pkg	24954-02

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	28156-96
Pipet Filler, safety bulb	each	each
Pipet, volumetric, Class A, 1.00-mL	each	each

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Updated November 2005 Edition 1

Chlorine Dioxide

Method 8345

Direct Reading Method

MR (1–50 mg/L)

Scope and Application: For water and wastewater

Test Preparation

Before starting the test:

Chlorine dioxide is unstable and volatile. Analyze samples immediately.

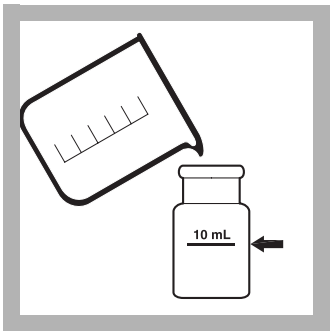
Collect the following items:	**Quantity**
Water, deionized	10 mL
Sample cell, 1-inch square glass, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 2.*

Direct Reading Method 8345

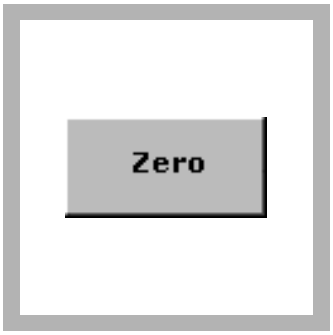
1. Press **STORED PROGRAMS**.

2. Select the test.



3. Blank Preparation: Fill a square sample cell to the 10-mL mark with deionized water.

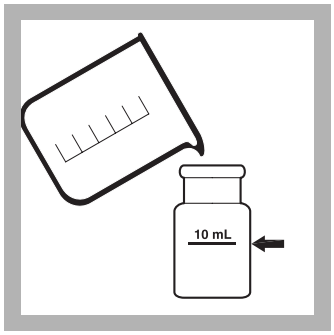
4. Wipe the blank and insert it into the cell holder with the fill line facing right.



5. Press **ZERO**.

The display will show:

0.0 mg/L ClO_2



6. Prepared Sample: Fill a second square sample cell to the 10-mL mark with sample.

7. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

8. Press **READ**.

Results are in mg/L ClO_2.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Samples must be analyzed immediately. Chlorine dioxide is very volatile and unstable.

Accuracy Check

Standard Solution Method

Preparing chlorine dioxide standards is difficult and dangerous. In addition, these standards are both explosive and volatile! Only a trained chemist should prepare the standards using appropriate safety equipment and precautions. The manufacturer does not recommend preparation of chlorine dioxide standards. If independent standard preparation is required, please see the instructions in *Standard Methods for the Examination of Water and Wastewater*, 20th ed., under the headings “Stock chlorine dioxide solution” and “Standard chlorine dioxide solution” (pp 4–74 and 4–75). Prepare a 25.0-mg/L chlorine dioxide standard.

Summary of Method

Chlorine dioxide, a yellow gas, can be measured directly in a water solution. Test results are measured at 360 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Water, deionized	10 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, glass,10 mL, matched pair	2	2/pkg	24954-02

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Updated November 2005 Edition 1

Chlorine Dioxide

★Method 10126 — DPD Method[1]

Powder Pillows and AccuVac® Ampuls — (0.04 to 5.00 mg/L)

Scope and Application: For water and wastewater. USEPA accepted for reporting for drinking water analysis.[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

[2] Procedure is equivalent to *Standard Methods, 18 ed., 4500 ClO_2 D.*

Test Preparation

Before starting the test:

Analyze samples immediately because chlorine dioxide is unstable and volatile. See Sample Collection, Storage, and Preservation on page 5.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using chlorine-free deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
After adding the DPD Free Chlorine Powder Pillow to the sample, a pink color will develop if chlorine dioxide is present.
If the chlorine dioxide concentration in the sample exceeds the upper limit of the test, the color may fade or the sample may turn yellow. Dilute the sample with high quality water that is chlorine demand-free, and repeat the test. Some loss of chlorine dioxide may occur. Multiply the result by the appropriate dilution factor.

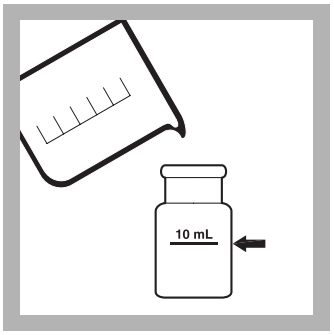
Collect the following items:	**Quantity**
Powder Pillow Test:	
DPD Free Chlorine powder pillow, 10-mL	1
Glycine Reagent	4 drops
Sample cells, 1-inch square, 10-mL	2
Stopper for 18 mm tube	2
AccuVac Test:	
DPD Free Chlorine Reagent AccuVac® Ampuls	1
Glycine Reagent	16 drops
Beaker, 50-mL	1
Sample Cell, 10-mL	1
Stopper for 18 mm tube	1

Note: *Reorder information for consumables and replacement items is on page 7.*

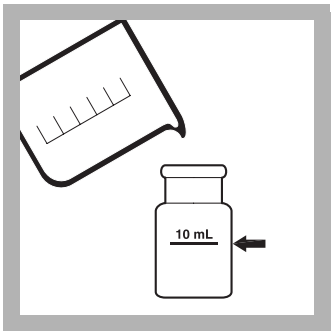
Powder Pillows — Method 10126

1. Press **STORED PROGRAMS**.

2. Select the test.

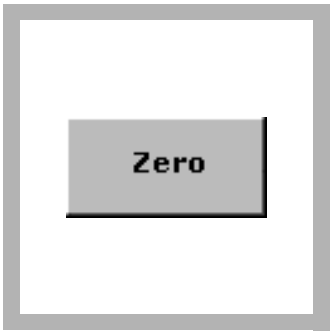


3. Blank Preparation: Fill a square sample cell with 10 mL of sample and stopper.



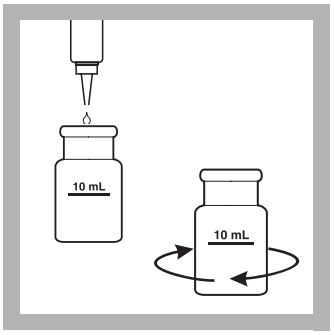
4. Prepared Sample: Fill a second square sample cell with 10 mL of sample and stopper.

5. Wipe the blank and insert it into the cell holder with the fill line facing right.

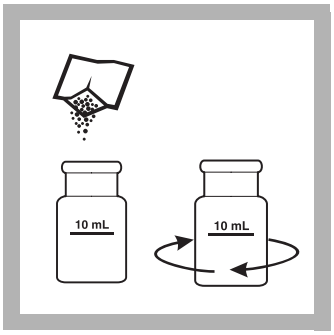


6. Press **ZERO**.

The display will show: 0.00 mg/L ClO_2



7. Add four drops of Glycine Reagent to the sample. Swirl to mix.



8. Add the contents of one DPD Free Chlorine Powder Pillow to the prepared sample cell.

Swirl the sample for 20 seconds to mix.

9. Wait 30 seconds for any undissolved powder to settle.

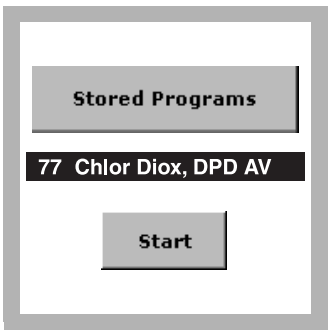
Immediately proceed to step 10.

10. Within one minute of adding the DPD reagent, wipe the sample cell and insert it into the cell holder with the fill line facing right.

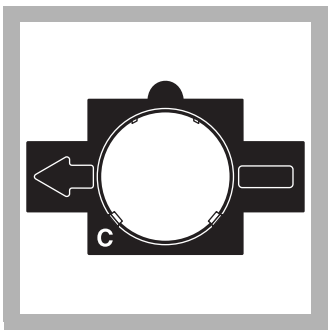
11. Press **READ**.

Results are in mg/L ClO_2.

AccuVac® Ampuls — Method 10126

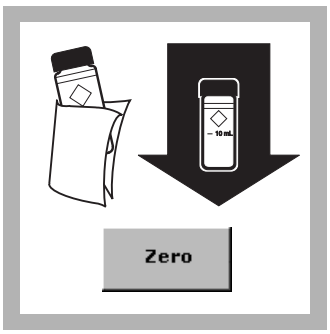


1. Select the test.



2. Insert the 1-inch round cell adapter (Adapter C).

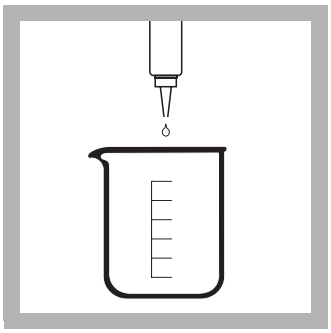
3. Blank Preparation: Fill a round sample cell with 10-mL of sample.



4. Wipe the blank and insert it into the cell holder.

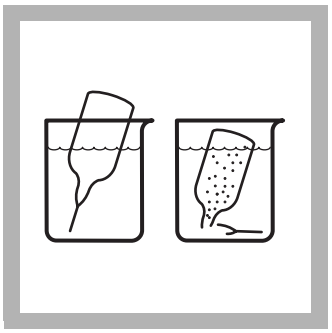
Press **ZERO**.

The display will show: 0.00 mg/L ClO_2

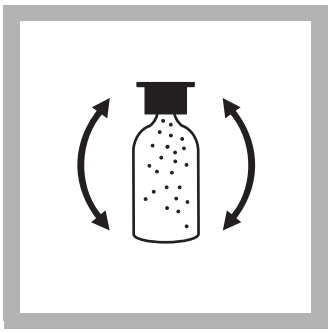


5. Prepared Sample: Fill a 50-mL beaker with 40 mL of sample.

Add 16 drops of Glycine Reagent to the sample in the beaker. Swirl gently to mix.



6. Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.



7. Quickly invert the Ampul several times to mix. Wait 30 seconds for any undissolved powder to settle.

8. Within one minute of adding the sample, wipe the Ampul and insert it into the cell holder.

Press **READ**. Results are in mg/L ClO_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sodium Hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. Color may not develop fully or may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for the volume addition.
Bromine, Br_2	Interferes at all levels.
Chlorine, Cl_2	May interfere at levels greater than 6 mg/L. Additional glycine may be able to compensate for this interference.
Chloramines, organic	May interfere.
Flocculating agents	High levels of most flocculating agents can be tolerated. This tolerance is decreased if chlorine is present. See the information about metals in this table. In the presence of 0.6 mg/L Cl_2, $Al(SO_4)_3$ (< 500 mg/L) and $FeCl_2$ (<200 mg/L) may be tolerated.
Hardness	No effect at less than 1000 mg/L as $CaCO_3$.
Iodine, I_2	Interferes at all levels.
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	Oxidized manganese interferes at all levels. Oxidized chromium interferes at levels greater than 2 mg/L. To remove the interferences: **1.** Adjust sample pH to 6–7. **2.** Add 3 drops Potassium Iodide[1] (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops Sodium Arsenite[1, 2] (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result of this test from the original analysis to obtain the correct chlorine dioxide concentration.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Metals	Various metals may interfere by combining with the glycine needed to remove the chlorine interference. Metal interference is limited except when chlorine is present. In the presence of 0.6 mg/L Cl_2, both copper (>10 mg/L) and nickel (>50 mg/L) interfere. Other metals may also interfere, depending on their ability to prevent glycine from reacting with any Cl_2 in the sample. It may be necessary to add more glycine to overcome this interference.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L ClO_2 increase in the reading.
Ozone	Interferes at levels greater than 1.5 mg/L.
Peroxides	May interfere.
Extreme sample pH	Adjust to pH 6–7.
Highly buffered samples	Adjust to pH 6–7.

[1] See Optional Reagents and Apparatus on page 7.
[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by Federal RCRA for arsenic (D004). Refer to a current MSDS for proper disposal instructions.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine dioxide immediately after collection. Chlorine dioxide is a strong oxidizing agent and is unstable in natural waters. It reacts rapidly with various inorganic compounds, but oxidizes organic compounds more slowly. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of chlorine dioxide in water.

Avoid plastic containers since these may have a large chlorine dioxide demand. Pretreat glass sample containers to remove any chlorine or chlorine dioxide demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine dioxide is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the chlorine dioxide analysis immediately.

Accuracy Check

Because chlorine dioxide is difficult and hazardous to produce, check the DPD and glycine reagents by using chlorine standards. Proceed as follows:

1. Prepare a 1-mg/L free chlorine standard using Method 1 or 2, below:

Method 1

a. Use Free Chlorine Standard*.

b. Determine the concentration of the standard from the certificate of analysis shipped with the standard (50–75 mg/L). Calculate the volume of standard needed as follows:

mL standard needed = 100 ÷ standard concentration

c. Pipet the volume of standard needed into a 100-mL volumetric flask. Dilute to the line with chlorine-demand-free deionized water. Invert to mix.

Method 2

a. Dilute 1 drop of 5% chlorine bleach in 1 liter of chlorine-demand-free deionized water. Use this as the standard.

b. Verify the standard's concentration using the Free Chlorine Method 8021.

c. Perform the chlorine dioxide test on the standard **without** adding glycine.

d. For program 76, the chlorine dioxide reading should be 2.35 times greater than the chlorine result. For program 77, the chlorine dioxide reading should be 2.34 times greater than the chlorine result. If so, this verifies the DPD and the instrument are functioning properly.

e. Repeat the chlorine dioxide test on the chlorine standard, including the glycine addition. The reading should be less than 0.10 mg/L. This verifies that the glycine is eliminating free chlorine interference.

Summary of Method

Chlorine dioxide reacts with DPD (N, N-diethyl-p-phenylenediamine) to the extent of one-fifth of its total available chlorine content corresponding to reduction of chlorine dioxide to chlorite to form a pink color. The color intensity is proportional to the ClO_2 in the sample. Chlorine interference is eliminated by adding glycine, which converts free chlorine to chloroaminoacetic acid, but has no effect on chlorine dioxide at the test pH. Test results are measured at 530 nm.

* See Optional Reagents and Apparatus on page 7.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Chlorine Dioxide DPD/Glycine Reagent Set (100 tests), includes:			27709-00
(1) DPD Free Chlorine Reagent Powder Pillows, 10-mL	1	100/pkg	21055-69
(1) Glycine Reagent	4 drops	29 mL	27621-33
OR			
Chlorine Dioxide DPD/Glycine AccuVac® Ampul Reagent Set (25 tests), includes:			27710-00
(1) DPD Free Chlorine Reagent AccuVac® Ampuls	1	25/pkg	25020-25
(1) Glycine Reagent	16 drops	29 mL	27621-33

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper for 18 mm tube	2	6/pkg	1731-06

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00
Stopper	1	6/pkg	1731-06

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L	16/pkg	14268-10
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description		Cat. No.
Potassium Iodide, 30 g/L, 100 mL	each	343-32
Sodium Arsenite, 5 g/L, 100 mL	each	1047-32
Sodium Hydroxide, 1 N, 100 mL	each	1045-32
Stopper	25/pkg	1731-25
Sulfuric Acid, 1 N, 100 mL	each	1270-32

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Updated November 2005 Edition 1

Chlorine, Free

★Method 8021 — DPD Method[1]

Powder Pillows or AccuVac® Ampuls — (0.02 to 2.00 mg/L)

Scope and Application: For testing free chlorine (hypochlorous acid and hypochlorite ion) in water, treated waters, estuary, and seawater. USEPA accepted for reporting for drinking water analyses.[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

[2] Procedure is equivalent to USEPA method 330.5 and Standard Method 4500-Cl G for drinking water.

Test Preparation

Before starting the test:

If the test overranges, dilute the sample with a known volume of high quality, chlorine demand-free water and repeat the test. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Alternatively, samples with high chlorine concentrations may be analyzed directly without dilution by using Method 10069, Chlorine, Free HR.

Collect the following items:	**Quantity**
Powder Pillow Test:	
DPD Free Chlorine powder pillow	1
Sample cells, 1-inch square, 10-mL	2
AccuVac Test:	
DPD Free Chlorine AccuVac® Ampul	1
Beaker, 50-mL	1
Sample cell, 10-mL round	1

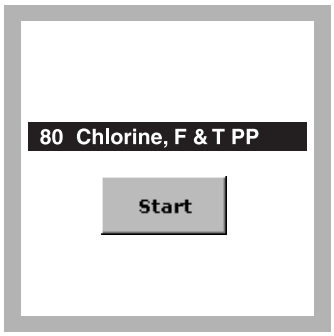
Note: *Reorder information for consumables and replacement items is on page 5.*

Note: *The SwifTest Dispenser for Free Chlorine can be used in place of the powder pillow in step 6.*

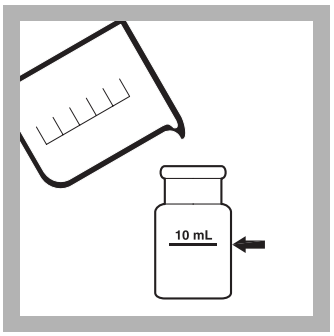
Important Note: *Analyze samples immediately. Do not preserve for later analysis.*

Powder Pillows — Method 8021

1. Press **STORED PROGRAMS**.

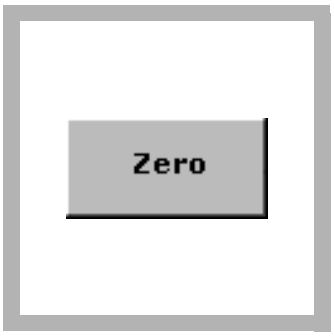


2. Select the test.



3. Blank Preparation: Fill a square sample cell with 10 mL of sample.

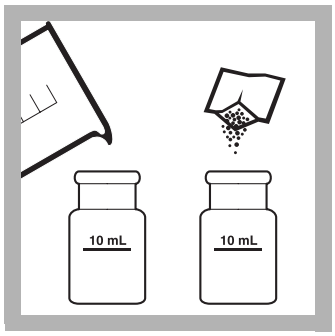
4. Wipe the blank and insert it into the cell holder with the fill line facing right.



5. Press **ZERO**.

The display will show:

0.00 mg/L Cl_2

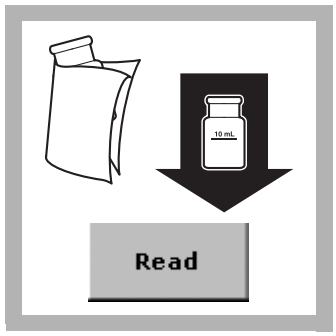


6. Prepared Sample: Fill a second square cell with 10 mL of sample.

Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell.

7. Swirl the sample cell for 20 seconds to mix.

A pink color will develop if chlorine is present. Proceed to step 8 immediately.

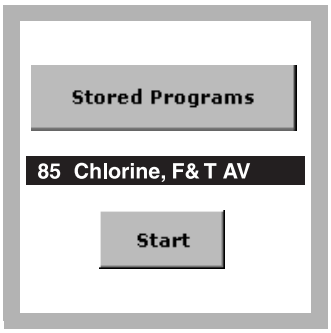


8. Within one minute of adding the reagent, insert the prepared sample into the cell holder with the fill line facing right.

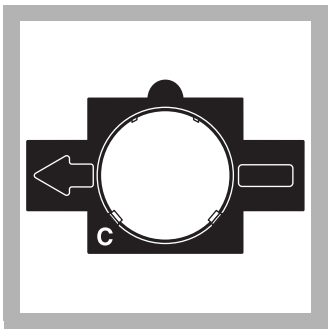
Press **READ**.

Results are in mg/L Cl_2.

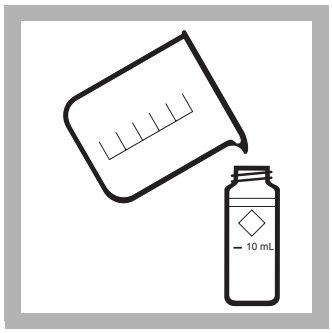
AccuVac® Ampuls — Method 8021



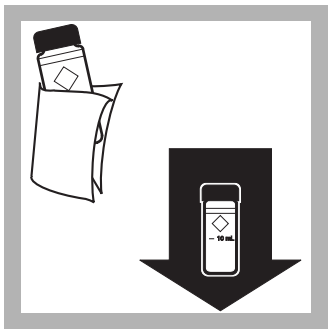
1. Select the test.



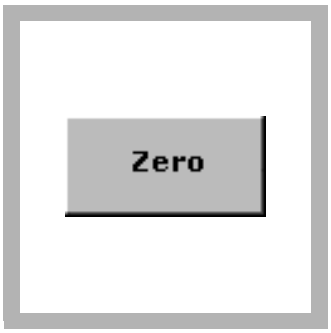
2. Insert Adapter C.



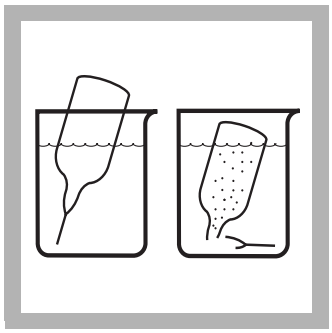
3. Blank Preparation: Fill a round sample cell with 10-mL of sample.



4. Wipe the blank and insert it into the cell holder.



5. Press **ZERO**. The display will show: 0.00 mg/L Cl_2



6. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.

7. Quickly invert the Ampul several times to mix. Wipe off any liquid or fingerprints.

8. Within one minute after sample addition, wipe the AccuVac Ampul and insert it into the cell holder.

Press **READ**.

Results are in mg/L Cl_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sodium Hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as $CaCO_3$
Iodine, I_2	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	Adjust sample pH to 6–7. Add 3 drops Potassium Iodide[1] (30-g/L) to a 10-mL sample. Mix and wait one minute. Add 3 drops Sodium Arsenite[1], [2] (5-g/L) and mix. Analyze 10 mL of the treated sample as described in the procedure. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or Highly buffered samples	Adjust to pH 6–7 using acid (Sulfuric Acid[1], 1.000 N) or base (Sodium Hydroxide[1], 1.00 N).

[1] See Optional Reagents and Apparatus on page 5.

[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by Federal RCRA for arsenic (D004). See the current MSDS for proper disposal of hazardous material.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine immediately after collection. Free chlorine is a strong oxidizing agent and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the chlorine analysis immediately.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the Chlorine Voluette®* Ampules. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **EDIT** to change these values. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a LR Chlorine Voluette Ampule Standard, 25–30 mg/L Cl_2.

5. Prepare three sample spikes. Fill three mixing cylinders with 10 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

Note: *For AccuVac® Ampuls, fill three mixing cylinders with 50 mL of sample and spike with 0.4 mL, 0.8 mL, and 1.2 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

*See Optional Reagents and Apparatus on page 5.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color, the intensity of which is proportional to the chlorine concentration. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
DPD Free Chlorine Reagent Powder Pillows, 10-mL	1	100/pkg	21055-69
OR			
DPD Free Chlorine Reagent AccuVac® Ampuls	1		25020-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, 2-mL Voluette® Ampule, 25–30 mg/L	20/pkg	26300-20

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Chlorine-demand Free Water	—	26415-49
Cylinder, mixing	25 mL	20886-40
Cylinder, mixing	50 mL	1896-41
Sodium Hydroxide, 1 N	100 mL	1045-32
Sulfuric Acid, 1 N	100 mL	1270-32
Potassium Iodide, 30-g/L	100 mL	343-32
Sodium Arsenite, 5-g/L	100 mL	1047-32
SwifTest Dispenser for Free Chlorine	—	28023-00

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Updated November 2005 Edition 1

Chlorine, Free

Method 10102 **DPD Method[1]**

Test ‘N Tube™ Vials **(0.09 to 5.00 mg/L)**

Scope and Application: For testing higher levels of free chlorine (hypochlorous acid and hypochlorite ion) in drinking water, cooling water, and industrial process water.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test Preparation

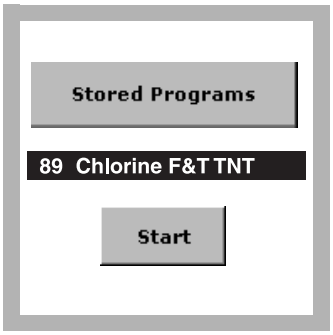
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Analyze samples immediately. Do not preserve samples for later analysis.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
After adding sample to the Test ‘N Tube™, a pink color will develop if free chlorine is present.

Collect the following items:	**Quantity**
Light Shield	LZV646
Test ‘N Tube™ DPD Free Chlorine Reagent	1

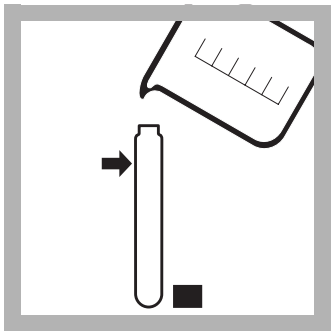
Note: *Reorder information for consumables and replacement items is on page 4.*

Test ‘N Tube Method 10102



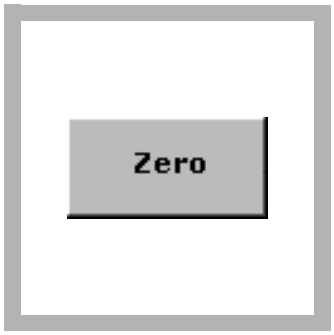
1. Select the test.

Install the Light Shield in Cell Compartment #2.



2. Blank Preparation: Fill an empty Test ‘N Tube vial to the top of the label with sample.

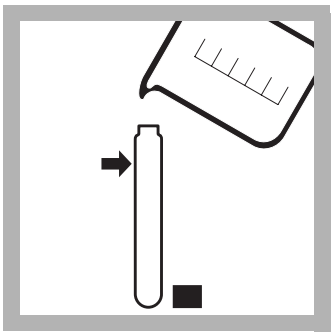
3. Wipe the blank and insert it into the 16 mm cell holder.



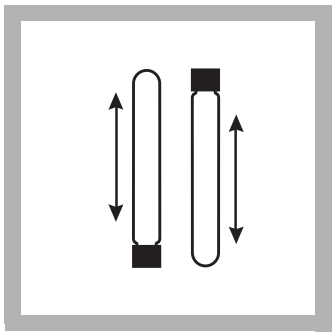
4. Press **ZERO**.

The display will show:

0.00 mg/L Cl_2



5. Remove the cap from a Free Chlorine DPD Test 'N Tube. Add 10 mL of sample to the tube. Fill the vial to the top of the label.



6. Prepared Sample: Cap and invert at least 10 times to dissolve the powder.

7. Immediately wipe the sample and insert it in the 16 mm cell holder.

8. Press **READ**.

Results are in mg/L Cl_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with1 N sodium hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sulfuric acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as $CaCO_3$
Iodine, I_2	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	Adjust sample pH to 6–7. Add 3 drops potassium iodide[1] (30-g/L) to a 25-mL sample. Mix and wait 1 minute. Add 3 drops sodium arsenite[2] (5-g/L) and mix. Analyze 10 mL of the treated sample as described in the procedure. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration in the sample.
Monochloramine	For conventional free chlorine disinfection (beyond the breakpoint), typical monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test depends on the sample temperature, relative amount of monochloramine to free chlorine, and the time required to do the analysis.Typical interference levels of monochloramine in the free chlorine test are listed below (as mg/L Cl_2).

NH_2Cl (as Cl_2)	Sample Temp. °C (°F)			
	5 (40)	10 (50)	20 (68)	30 (83)
1.2 mg/L	+0.15	0.19	0.30	0.29
2.5 mg/L	+0.35	0.38	0.55	0.61
3.5 mg/L	+0.38	0.56	0.69	0.73

Ozone, O_3	Interferes at all levels

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Peroxides	May interfere
Extreme sample pH or Highly buffered samples	Adjust to pH 6–7 using acid (Sulfuric Acid[1], 1.000 N) or base (Sodium Hydroxide[1], 1.00 N).

[1] See Optional Reagents and Apparatus on page 4.
[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by Federal RCRA for arsenic (D004). Refer to reagent MSDS for disposal instructions.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine immediately after collection. Free chlorine is a strong oxidizing agent and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the chlorine analysis immediately.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify that the units displayed are in mg/L.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the standard solution. Press **OK**. A summary of the Standard Additions procedure will appear. Press **OK**.

3. Snap the neck off of a HR Chlorine PourRite® Ampule Standard*, 50–75 mg/L Cl_2.

4. Use the TenSette® Pipet to add 0.1 mL to a 10-mL sample. Mix thoroughly.

5. Analyze the standard addition sample as described in the procedure above. Accept the standard additions reading by pressing **READ**. The addition should reflect approximately 100% recovery.

*See Optional Reagents and Apparatus on page 4.

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color which is proportional to the chlorine concentration. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Test 'N Tube™ DPD Free Chlorine Reagent	1	50/pkg	21055-45

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Chlorine Standard Solution, 2-mL PourRite® Ampule, 50–75 mg/L	20/pkg	14268-20

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Kimwipes®	280/pkg	20970-00
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Sulfuric Acid, 1.000 N, 100 mL	each	1270-32
Sodium Hydroxide, 1.00 N, 100 mL	each	1045-32

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Updated November 2005 Edition 1

Chlorine, Free

Method 10059
Pour-Thru™ Cell

DPD Rapid Liquid Method[1]
(0.02 to 2.00 mg/L)

Scope and Application: For treated water.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

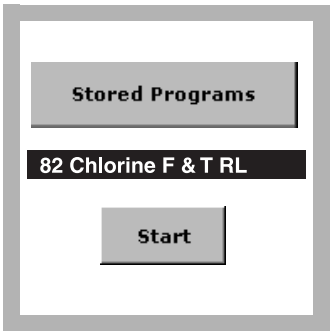
Before starting the test:

Analyze samples immediately. Do not preserve for later analysis.
Refer to the instrument User Manual for Pour-Thru cell and module assembly and installation.
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel.
The indicator reagent must be prepared in advance. See Reagent Preparation on page 2.

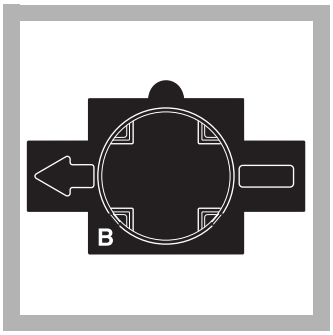
Collect the following items:	**Quantity**
DPD Indicator Powder	varies
Free Chlorine Indicator Solution	1 mL
Free Chlorine Buffer Solution	1 mL
Cylinder, glass, mixing, 100-mL	1
Dispenser, 1.0 mL fixed volume, Repipet Jr.	2
Pour-Thru Cell Module and Cell	1

Note: *Reorder information for consumables and replacement items is on page 5.*

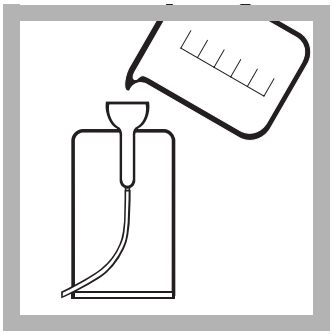
Pour-Thru Cell — Method 10059



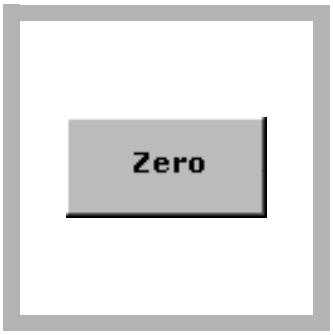
1. Select the test.



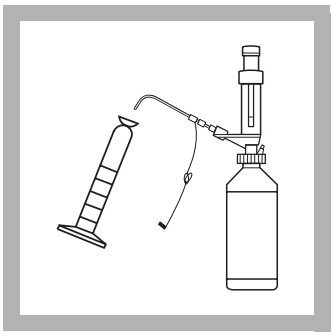
2. Insert Adapter B. Install the Pour-Thru Cell module and cell with the 1-inch (round) path in line with the arrow on the adapter. Flush the Pour-Thru cell with 50 mL of deionized water.



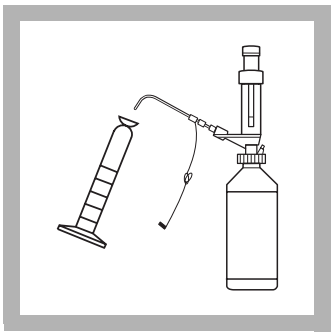
3. Pour approximately 50 mL of sample into the Pour-Thru Cell.



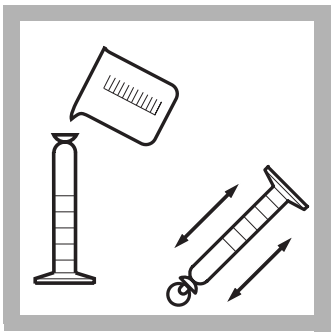
4. When the flow stops, press **ZERO**. The display will show: 0.00 mg/L Cl_2.



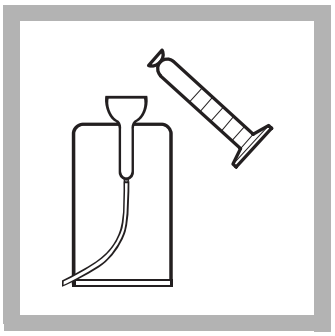
5. Add 1.0 mL of Free Chlorine Buffer Solution to a clean, dry 100-mL glass mixing cylinder using the Repipet Jr. Dispenser.



6. Add 1.0 mL of prepared Free Chlorine Indicator Solution to the same mixing cylinder using the Repipet Jr. Dispenser. Swirl to mix the reagents. Proceed to step 7 immediately.



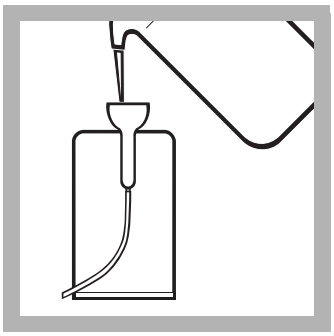
7. Carefully fill the mixing cylinder to the 80-mL mark with sample. Stopper the cylinder and gently invert it twice to mix. Proceed to step 8 immediately.



8. Fill the funnel of the Pour-Thru Cell with the reacted sample from the mixing cylinder.

(It is not necessary to pour the entire sample into the Pour-Thru Cell; approximately half of the sample may be discarded.)

After the flow stops, press **READ**. Results will appear in mg/L Cl_2.



9. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Reagent Preparation

The Free Chlorine Indicator Solution must be prepared before use. Using a powder funnel, add the contents of one 24 g bottle of DPD Powder* to one 473-mL bottle of Free Chlorine Indicator Solution*. Invert several times and swirl until the powder is completely dissolved. A pink color may develop, but should not affect results.

This solution will give accurate results for at least one month after mixing when stored at 20–25 °C (68–77 °C). Write the date of preparation on the Indicator Solution Bottle. Discard any remaining solution after one month. Use of this reagent after one month may result in high reagent blanks and low values at high concentration. Do not combine fresh reagent with previously mixed reagent.

* See Required Reagents on page 5.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Alkalinity	Greater than 400 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels.
Hardness	Levels below 1000 mg/L as $CaCO_3$ will not interfere.
Iodine, I_2	Interferes at all levels.
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7 with 1.000 N Sulfuric Acid[1]. **2.** Add 9 drops Potassium Iodide (30 g/L)[1] to an 80-mL sample. **3.** Mix and wait 1 minute. **4.** Add 9 drops Sodium Arsenite[1, 2] (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure above. **6.** Subtract the result of this test from the original analysis to obtain the correct concentration.
Monochloramine (NH_2Cl)	Samples containing monochloramine will cause a gradual drift to higher chlorine readings. When read within one minute of reagent addition, 3.0 mg/L monochloramine will cause an increase of less than 0.1 mg/L in the free chlorine reading.
Ozone	Interferes at all levels.

[1] See Optional Standards and Apparatus on page 5.

[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for safe handling and disposal instructions.

Sampling and Storage

Samples must be analyzed immediately and cannot be preserved for later analysis. A common testing error is introduced if the analyst does not obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. Perform the chlorine analysis immediately.

Avoid plastic containers since these may have a chlorine demand. Pre-treat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized water.

If sample containers are rinsed thoroughly with deionized water after use, only occasional pretreatment is necessary. A pre-treated glass BOD bottle with a ground-glass stopper makes an ideal sample container for chlorine analysis.

Treating Analysis Labware

Glassware used in this test must be chlorine demand-free. Fill the 100-mL mixing cylinder and sample container with a dilute solution of chlorine bleach prepared by adding 1 mL of commercial bleach to 1 liter of water. Soak in this solution at least one hour. After soaking, rinse thoroughly with deionized water and allow to dry before use. If the mixing cylinder is thoroughly rinsed with deionized water and allowed to dry after each use, only occasional pretreatment is necessary. Do not use the same mixing cylinder for Free and Total Chlorine analysis.

Treat the Pour-Thru Cell similarly with dilute bleach and let stand for several minutes. Rinse several times with deionized water.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored reaction products, especially if the reacted solutions are allowed to remain in the cell for long periods after measurement. Remove the buildup by rinsing the cell with 5.25 N Sulfuric Acid* followed by several rinsings with deionized water.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the chlorine voluette ampules. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **Edit** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the top off a Chlorine Voluette® Ampule Standard Solution, 50 to 75-mg/L Cl_2.

5. Prepare three sample spikes. Use the TenSette® Pipet to add 0.3, 0.6 and 0.9 mL of standard to three 80-mL samples, respectively. Swirl gently to mix.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Method Performance

Precision

Standard: 1.18 mg/L Cl_2

Program	95% Confidence Limits of Distribution
82	1.17–1.19 mg/L Cl_2

Sensitivity

Portion of Curve	Abs	Concentration
Entire range	0.010	0.02 mg/L Cl_2

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a red color which is proportional to the chlorine concentration. Test results are measured at 530 nm.

* See Optional Standards and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Free Chlorine Reagent Set, includes:	—	—	25569-00
DPD Indicator Powder	varies	24 g	22972-55
Free Chlorine Indicator Solution	1 mL	473 mL	23140-11
Free Chlorine Buffer Solution	1 mL	473 mL	23141-11

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 100-mL, poly	1	each	1896-42
Dispenser, fixed-volume, 1.0-mL, Repipet Jr.	2	each	21113-02
Pour-Thru Cell Module Kit	1	each	59404-00
Powder Funnel	1	each	22644-67

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, Voluette® Ampule, 50-75 mg/L, 10-mL	16/pkg	14268-10
OR		
Chlorine Standard Solution, Voluette® Ampule, 50-75 mg/L, 2-mL	20/pkg	14268-20
Water, deionized	4 L	272-56

Optional Standards and Apparatus

Description	Cat. No.
Pipet, TenSette® 0.1–1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01, 50/pkg	21856-96
Potassium Iodide, 30 g/L, 100 mL	343-32
Sodium Arsenite, 5 g/L, 100 mL	1047-32
Sulfuric Acid, 1 N, 100 mL	1270-32
Sulfuric Acid, 5.25 N, 100 mL	2449-53

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Updated November 2005 Edition 1

Chlorine, Free

Method 10069 **DPD Method[1]**

Powder Pillows **HR (0.1 to 10.0 mg/L as Cl_2)**

Scope and Application: For testing higher levels of free chlorine (hypochlorous acid and hypochlorite ion) in drinking water, cooling water, and industrial process waters

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test Preparation

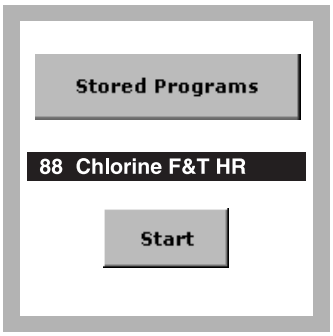
Before starting the test:

Analyze samples immediately. Do not preserve for later analysis.
If the chlorine concentration is less than 2 mg/L, use Method 8021, program number 80.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

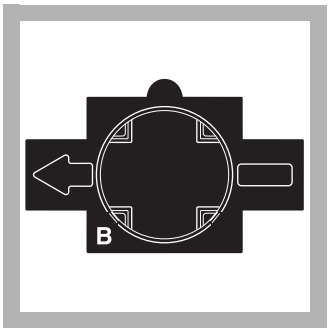
Collect the following items:	**Quantity**
DPD Free Chlorine Reagent Powder Pillows	1
Sample Cell, 1-cm, 10-mL	1

Note: *Reorder information for consumables and replacement items is on page 5.*

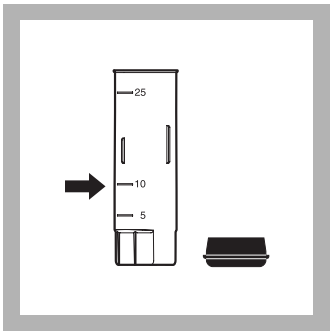
Multi-path Cell Method 10069



1. Select the test.

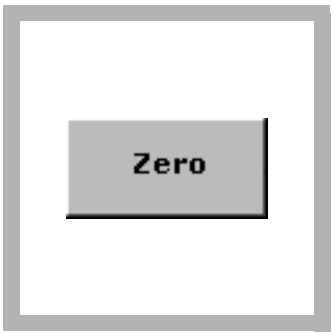


2. Insert cell Adapter B.



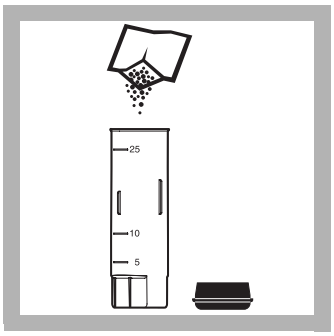
3. Fill the sample cell to the 5-mL line with sample.

4. Wipe the cell and insert it into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.

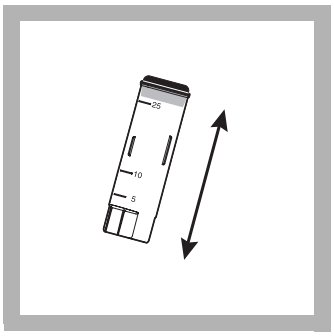


5. Press **ZERO**.

The display will show: 0.0 mg/L Cl_2

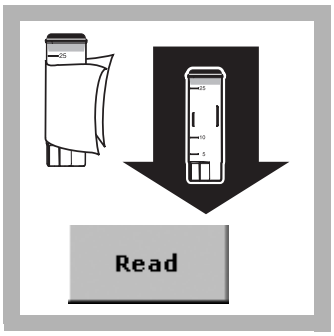


6. Remove the cell and add the contents of one DPD Free Chlorine powder pillow for 25-mL samples to the sample.



7. Cap and shake the cell about 20 seconds to dissolve reagent.

A pink color will develop if chlorine is present.



8. Insert the prepared sample into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.

Press **READ**. Results are in mg/L Cl_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with1 N Sodium Hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I_2	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7 with 1.000 N Sulfuric Acid[1]. **2.** Add 2 drops Potassium Iodide[1] (30 g/L) to a 5-mL sample. **3.** Mix and wait 1 minute. **4.** Add 2 drops Sodium Arsenite[1, 2] (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure above. **6.** Subtract the result of this test from the original analysis to obtain the correct concentration.

Table 1 Interfering Substances and Levels (continued)

<table>
<tr><th>Interfering Substance</th><th>Interference Levels and Treatments</th></tr>
<tr><td>Monochloramine (NH_2Cl)</td><td>For conventional free chlorine disinfection (beyond the “breakpoint”), typical monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test is dependent on sample temperature, relative concentration of monochloramine to free chlorine, and the time required to perform the analysis.
Typical interference levels of NH_2Cl (1 minute test time, interference as mg/L Cl_2):
<table>
<tr><th rowspan="2">NH_2Cl Level</th><th colspan="4">Sample Temperature °C (° F)</th></tr>
<tr><th>5 (41)</th><th>10 (50)</th><th>20 (68)</th><th>30 (86)</th></tr>
<tr><td>1.2 mg/L</td><td>+0.15</td><td>0.19</td><td>0.30</td><td>0.29</td></tr>
<tr><td>2.5 mg/L</td><td>+0.35</td><td>0.38</td><td>0.55</td><td>0.61</td></tr>
<tr><td>3.5 mg/L</td><td>+0.38</td><td>0.56</td><td>0.69</td><td>0.73</td></tr>
<tr><td>5.0 mg/L</td><td>+0.68</td><td>0.75</td><td>0.93</td><td>1.05</td></tr>
</table></td></tr>
<tr><td>Ozone</td><td>Interferes at all levels</td></tr>
<tr><td>Peroxides</td><td>May interfere</td></tr>
<tr><td>Extreme sample pH or highly buffered samples</td><td>Adjust to pH 6–7 using acid (Sulfuric Acid[1]) or base (Sodium Hydroxide[1]).</td></tr>
</table>

[1] See Optional Reagents and Apparatus on page 5.
[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for safe handling and disposal instructions.

Sampling and Storage

Analyze samples for chlorine immediately after collection. Free chlorine is a strong oxidizing agent and reacts rapidly with various compounds. Many factors such as sunlight, pH, temperature and sample composition will influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the chlorine voluette ampules. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a High Range Chlorine PourRite® Ampule Standard, 50–75 mg/L.

5. Prepare three sample spikes. Fill three mixing cylinders* with 5-mL of sample. Using the TenSette® Pipet*, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

Summary of Method

The range of analysis using the DPD method for free chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Free Chlorine Reagent is added to a 5-mL sample portion.

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N, N-diethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration. Test results are measured at 530 nm.

* See Optional Reagents and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
DPD Free Chlorine Reagent Powder Pillows for 25-mL samples	1	100/pkg	14070-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Sample cell, multi-path	1	6/pkg	59405-06

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, 2-mL PourRite® Ampules, 50–75 mg/L	20/pkg	14268-20

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing, 25 mL tall form	20886-40
Pipet, TenSette®, 0.1 to 1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01	21856-96
Potassium Iodide, 30 g/L, 100 mL	343-32
Sodium Arsenite, 5 g/L, 100 mL	1047-32
Sodium Hydroxide.1 N, 100 mL	1045-32
Sulfuric Acid, 1 N, 100 mL	1270-32

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Updated November 2005 Edition 1

Chlorine, Total

★Method 8167 — DPD Method[1]

Powder Pillows or AccuVac® Ampuls — (0.02 to 2.00 mg/L)

Scope and Application: For testing residual chlorine and chloramines in water, wastewater, estuary water, and seawater; USEPA-accepted for reporting[2] for drinking and wastewater analyses.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

[2] Procedure is equivalent to USEPA method 330.5 and Standard Method 4500-Cl G for drinking water and wastewater analyses.

Test Preparation

Before starting the test:

Samples must be analyzed immediately and cannot be preserved for later analysis
If the test overranges, dilute the sample with a known volume of high quality, chlorine demand-free water, and repeat the test. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Or, analyze samples with high chlorine concentrations directly without dilution by using Method 10070, Chlorine, Total HR.
For chloramination disinfection control, use Method 10172, Chloramine (Mono), Low Range (program number 66) or High Range (program number 67).
The SwifTest Dispenser[1] for Total Chlorine can be used in place of the powder pillow in step 4.

[1] Optional Reagents and Apparatus on page 6.

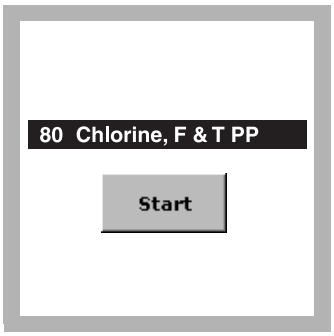
Collect the following items:	**Quantity**
Powder Pillow Test:	
DPD Total Chlorine Reagent powder pillow, 10-mL	1
Sample Cells, 1-inch square, 10-mL	2
AccuVac Test:	
Collect at least 40 mL of sample in a 50-mL beaker	40 mL
DPD Total Chlorine Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cell, 10-mL round	1

Note: *Reorder information for consumables and replacement items is on page 6.*

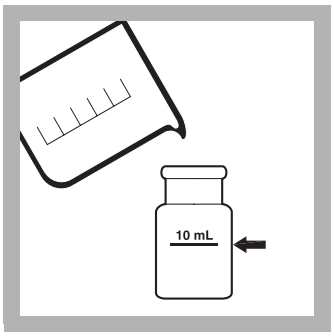
Note: *After adding the reagent, a pink color will develop if chlorine is present.*

Powder Pillows — Method 8167

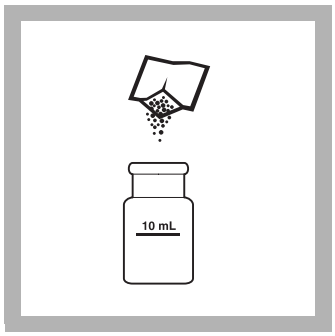
1. Press **STORED PROGRAMS**.



2. Select the test.



3. Fill a square sample cell with 10 mL of sample.

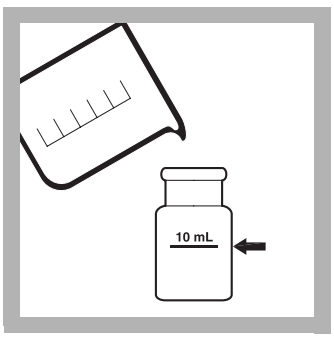


4. Prepared Sample: Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell.

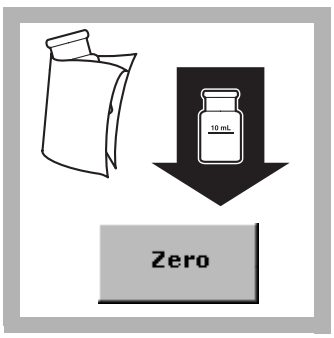
Swirl the sample cell for 20 seconds to mix.

5. Press **TIMER>OK**.

A three-minute reaction period will begin. Perform steps 6 and 7 during this time period.



6. Blank Preparation: Fill a second square sample cell with 10-mL of sample.



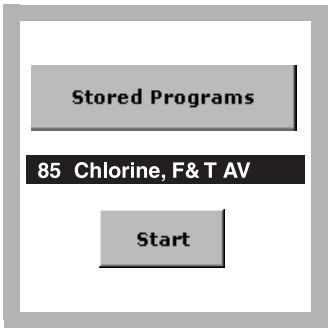
7. Wipe the blank sample cell and insert it into the cell holder with the fill line facing right.

Press **ZERO**. The display will show: 0.00 mg/L Cl_2

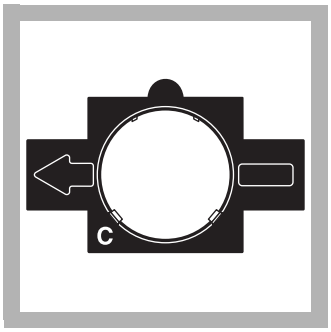
8. Within three minutes after the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Cl_2.

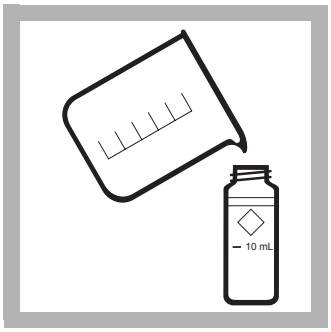
AccuVac® Ampul — Method 8167



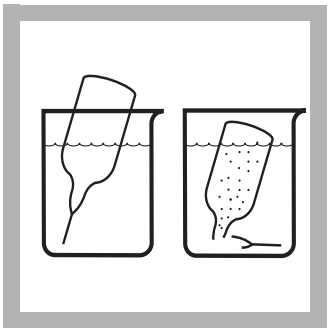
1. Select the test.



2. Insert Adapter C.



3. Blank Preparation: Fill a round sample cell with 10-mL of sample.

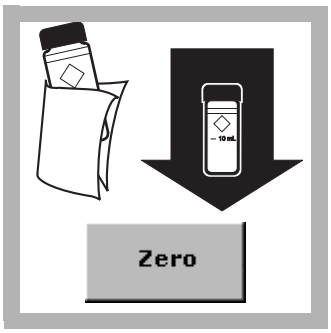


4. Prepared Sample: Fill a DPD Total Chlorine Reagent AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely.

5. Quickly invert the Ampul several times to mix. Wipe off any liquid or fingerprints.

6. Press **TIMER>OK**.

A three-minute reaction period will begin. Perform steps 7 and 8 during this time period.



7. Wipe the blank and insert it into the cell holder.

Press **ZERO**. The display will show: 0.00 mg/L Cl_2

8. Within three minutes after the timer expires, wipe the AccuVac Ampul and insert it into the cell holder.

Press **READ**. Results are in mg/L Cl_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sodium hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 300 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sulfuric acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as $CaCO_3$
Iodine, I_2	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	Adjust sample pH to 6–7. Add 3 drops potassium iodide[1] (30 g/L) to a 25-mL sample. Mix and wait one minute. Add 3 drops sodium arsenite[1, 2] (5 g/L) and mix. Analyze 10 mL of the treated sample as described in the procedure. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or Highly buffered samples	Adjust to pH 6–7 using acid (Sulfuric Acid[1], 1.000 N) or base (Sodium Hydroxide[1], 1.00 N).

[1] See Optional Reagents and Apparatus on page 6.
[2] Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by the Federal RCRA for arsenic (D004). Reference the current MSDS for more information on proper disposal of these materials.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine immediately after collection. Chlorine is a strong oxidizing agent and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, cap the sample containers so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the chlorine analysis immediately.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the Chlorine Voluette® Ampules. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a LR Chlorine Voluette® Ampule Standard, 25–30 mg/L Cl_2.

5. Prepare three sample spikes. Fill three mixing cylinders* with 10 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 10-mL samples and mix each thoroughly.

Note: *For AccuVac® Ampuls, fill three mixing cylinders with 50-mL of sample and spike with 0.4 mL, 0.8 mL, and 1.2 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers*. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Summary of Method

Chlorine can be present in water as free chlorine and as combined chlorine. Both forms can exist in the same water and be determined together as the total chlorine. Free chlorine is present as hypochlorous acid or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine and free chlorine reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run a free chlorine test. Subtract the results of the free chlorine test from the total chlorine test to obtain the combined chlorine concentration. Test results are measured at 530 nm.

* See Optional Reagents and Apparatus on page 6.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows, 10-mL	1	100/pkg	21056-69
OR			
DPD Total Chlorine Reagent AccuVac® Ampuls	1	25/pkg	25030-25
Deionized Water	varies	4 L	272-56

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, round with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, 2-mL Voluette® Ampule, 25–30 mg/L	20/pkg	26300-20

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Beakers, 50-mL	—	500-41H
Chlorine Demand-Free Water	—	26415-49
Cylinder, mixing	25 mL	20886-40
Cylinder, mixing	50 mL	1896-41
Potassium Iodide (30 g/L)	10 mL	343-32
Sodium Arsenite (5 g/L)	10 mL	1047-32
Sodium Hydroxide, 1 N	10 mL	1045-32
Sulfuric Acid, 1 N	10 mL	1270-32
SwifTest Dispenser for Total Chlorine	—	28024-00

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Updated November 2005 Edition 1

Chlorine, Total

Method 10070 **DPD Method[1]**

Powder Pillows **HR (0.1 to 10.0 mg/L as Cl_2)**

Scope and Application: For testing higher levels of total chlorine (free and combined) in drinking water, cooling water, and industrial process waters

[1] USEPA accepted for reporting drinking water analyses. Procedure is equivalent to USEPA Method 330.5 for Wastewater, and Standard Method 4500-Cl-G for Drinking Water

Test Preparation

Before starting the test:

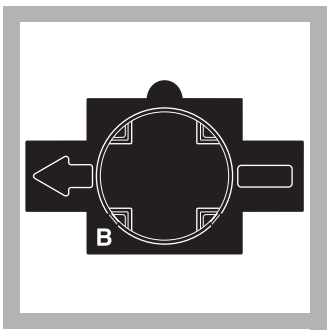
Analyze samples immediately. Do not preserve for later analysis.
If the chlorine concentration is less than 2 mg/L, use Method 8021, program number 80.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

Collect the following items:	**Quantity**
DPD Total Chlorine Reagent Powder Pillows	1
Sample Cell, 1-cm, 10-mL	1

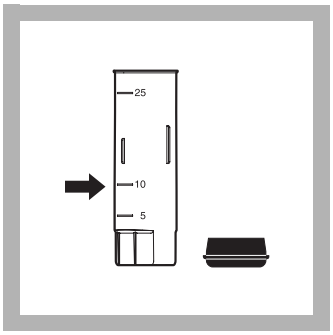
Note: *Reorder information for consumables and replacement items is on page 4.*

Multi-path Cell Method 10070

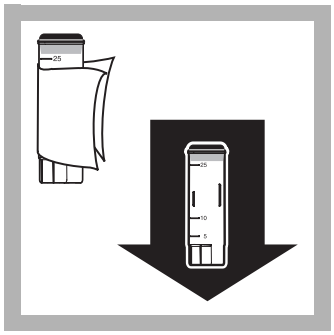
1. Select the test.



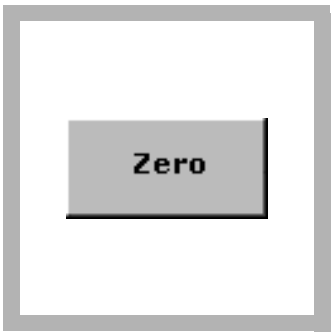
2. Insert Adapter B.



3. Fill the sample cell to the 5-mL line with sample.

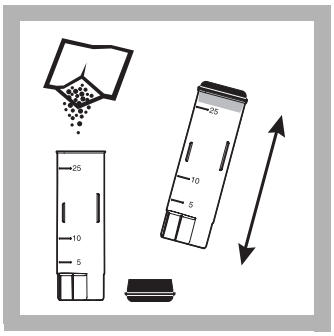


4. Wipe the cell and insert it into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.



5. Press **ZERO**.

The display will show: 0.0 mg/L Cl_2

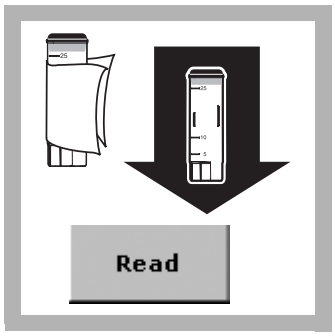


6. Remove the cell and add the contents of one DPD Total Chlorine powder pillow for 25-mL samples to the sample. Cap and shake the cell about 20 seconds to dissolve reagent.

A pink color will develop if chlorine is present.

7. Press **TIMER>OK**.

A 3-minute reaction period will begin.



8. Insert the prepared sample into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.

Press **READ**. Results are in mg/L Cl_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with1 N Sodium Hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I_2	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7 with 1.000 N Sulfuric Acid[1]. **2.** Add 2 drops Potassium Iodide[1] (30 g/L) to a 5-mL sample. **3.** Mix and wait 1 minute. **4.** Add 2 drops Sodium Arsenite[1, 2] (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure above. **6.** Subtract the result of this test from the original analysis to obtain the correct concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust to pH 6–7 using acid (Sulfuric Acid[1]) or base (Sodium Hydroxide[1]).

[1] See Optional Reagents and Apparatus on page 4.

[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for safe handling and disposal instructions.

Sampling and Storage

Analyze samples for chlorine immediately after collection. Free and combined chlorine are strong oxidizing agents and react rapidly with various compounds. Many factors such as sunlight, pH, temperature and sample composition will influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the chlorine voluette ampules. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a High Range Chlorine PourRite® Ampule Standard, 50–75 mg/L.

5. Prepare three sample spikes. Fill three mixing cylinders* with 5-mL of sample. Using the TenSette® Pipet*, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

* See Optional Reagents and Apparatus on page 4.

Summary of Method

The range of analysis using the DPD method for total chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Total Chlorine Reagent is added to a 5-mL sample portion.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run free chlorine and total chlorine tests. Subtract the results of the free chlorine test from the results of the total chlorine test to obtain combined chlorine. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows for 25-mL samples	1	100/pkg	14064-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Sample cell, multi-path	1	6/pkg	59405-06

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, 2-mL PourRite® Ampules, 50–75 mg/L	20/pkg	14268-20

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing, 25 mL	1896-40
Pipet, TenSette®, 0.1 to 1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01	21856-96
Potassium Iodide, 30 g/L, 100 mL	343-32
Sodium Arsenite, 5 g/L, 100 mL	1047-32
Sodium Hydroxide.1 N, 100 mL	1045-32
Sulfuric Acid, 1 N, 100 mL	1270-32

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Updated November 2005 Edition 1

Chlorine, Total

Method 10101 **DPD Method[1]**

Test ‘N Tube™ Vials **(0.09 to 5.00 mg/L)**

Scope and Application: For testing higher levels of total (free plus combined) chlorine in drinking water, treated wastewater, cooling water, or industrial process water

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test Preparation

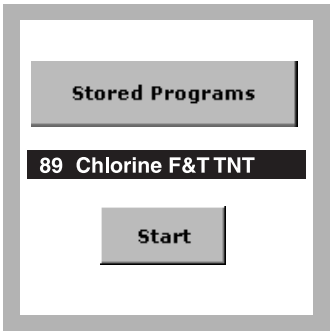
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Analyze samples immediately. Do not preserve samples for later analysis.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
For chloramination disinfection control, use Method 10172, Chloramine (Mono), High Range.
After adding sample to the Test ‘N Tube™, a pink color will develop if free chlorine is present.

Collect the following items:	**Quantity**
Light Shield	1
Test ‘N Tube™ DPD Total Chlorine Reagent	1

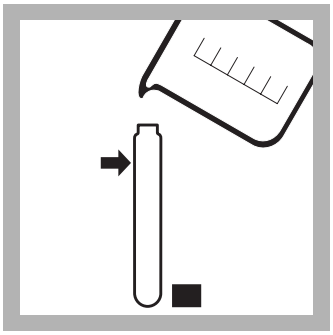
Note: *Reorder information for consumables and replacement items is on page 4.*

Test ‘N Tube Method 10101



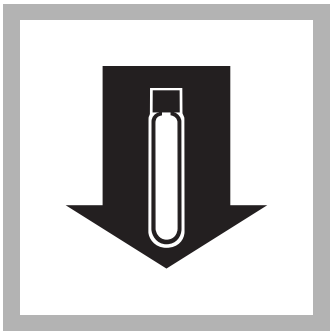
1. Select the test.

Install the Light Shield in Cell Compartment #2.

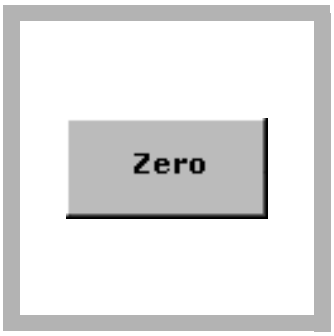


2. Blank Preparation: Fill an empty Test ‘N Tube vial to the top of the label with sample.

3. Wipe the outside of the vial to remove fingerprints and other marks.



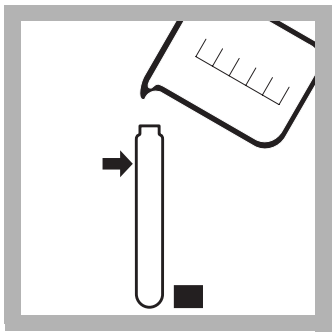
4. Insert the blank into the 16 mm cell holder. Close the cover.



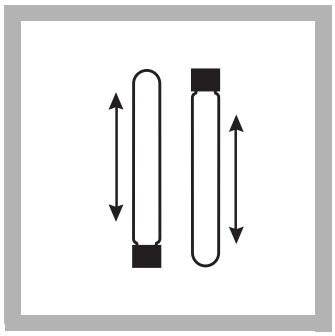
5. Press **ZERO**.

The display will show:

0.00 mg/L Cl_2



6. Prepared Sample: Remove the cap from a Total Chlorine DPD Test 'N Tube™. Add 10 mL of sample to the tube. (Fill the vial to the top of the label.)

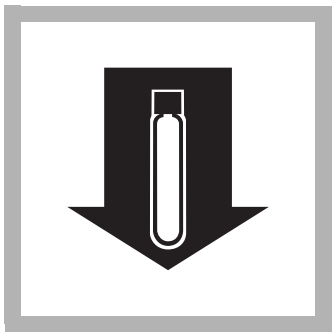


7. Cap and slowly invert at least 10 times to dissolve the powder. (Ten inversions should take at least 30 seconds. One inversion equals turning the vial upside down, then returning it to an upright position.)

8. Press **TIMER>OK**.

A two-minute reaction period will begin.

9. When the timer expires, wipe the outside of the vial that contains the prepared sample.



10. Insert the sample in the 16 mm cell holder.

11. Press **READ**.

Results are in mg/L Cl_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with1 N Sodium Hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 300 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as $CaCO_3$
Iodine, I_2	Interferes at all levels

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatment
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	Adjust sample pH to 6–7. Add 3 drops Potassium Iodide[1] (30-g/L) to a 25-mL sample. Mix and wait 1 minute. Add 3 drops Sodium Arsenite[1], [2] (5-g/L) and mix. Analyze 10 mL of the treated sample as described in the procedure. Subtract the result of this test from the original analysis to obtain the correct chlorine concentration.
Ozone, O_3	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust to pH 6–7 using acid (Sulfuric Acid[1], 1.000 N) or base (Sodium Hydroxide[1], 1.00 N).

[1] See Optional Reagents and Apparatus on page 4.
[2] Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by the Federal RCRA for arsenic (D004). See the current reagent MSDS for safe disposal instructions.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine immediately after collection. Free chlorine and combined chlorine are strong oxidizing agents and are unstable in natural waters. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is obtaining an unrepresentative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the chlorine analysis immediately.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the Chlorine Voluette® Ampules. Press **OK**.
3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off a HR Chlorine PourRite® Ampule Standard, 50-75 mg/L Cl_2.
5. Use the TenSette® Pipet to add 0.1 mL of standard to a 10-mL sample and mix thoroughly.

6. Analyze the standard addition sample as described in the procedure above. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

Summary of Method

Chlorine can be present in water as free chlorine and as combined chlorine. Both forms can exist in the same water and be determined together as the total chlorine. Free chlorine is present as hypochlorous acid or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives.

Free or combined chlorine oxidizes iodide in the reagent to iodine. The iodine and chlorine react with DPD (N,N-diethyl-p-phenylenediamine) to form a magenta color, which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run a free chlorine test. Subtract the results of the free chlorine test from the total chlorine test to obtain the combined chlorine concentration. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Test ‘N Tube™ DPD Total Chlorine Reagent	1	50/pkg	21056-45

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, 2-mL PourRite® Ampule, 50–75 mg/L	20/pkg	14268-20

Optional Reagents and Apparatus

Description	Cat. No.
Pipet, TenSette®, 0.1–1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01, 50/pkg	21856-96
Potassium Iodide 100 mL	343-32
Sodium Arsenite 5 g/L 100 mL	1047-32
Sodium Hydroxide, 1.00 N 100 mL	1045-32
Sulfuric Acid, 1.000 N 100 mL	1270-32

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Updated November 2005 Edition 1

Chlorine, Total

Method 10060
Pour-Thru Cell

DPD Rapid Liquid Method[1]
(0.02 to 2.00 mg/L)

Scope and Application: For treated water.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

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Test Preparation

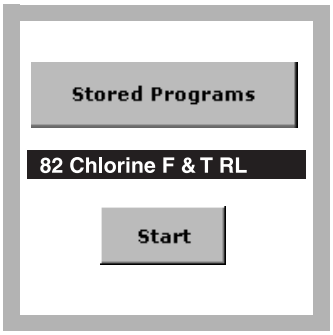
Before starting the test:

Analyze samples immediately. Do not preserve for later analysis.
Refer to the instrument User Manual for Pour-Thru cell and module assembly and installation.
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel. Refer to Reagent Preparation on page 3.

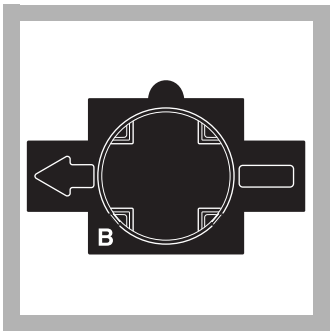
Collect the following items:	**Quantity**
DPD Indicator Powder	varies
Total Chlorine Indicator Solution	1 mL
Total Chlorine Buffer Solution	1 mL
Cylinder, glass, mixing, 100-mL	1
Dispenser, 1.0 mL fixed volume, Repipet Jr.	2
Pour-Thru Module and Cell	1

Note: *Reorder information for consumables and replacement items is on page 5.*

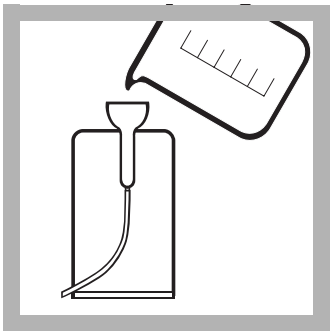
Pour-Thru Cell — Method 10060



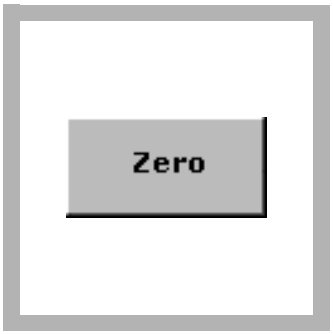
1. Select the test.



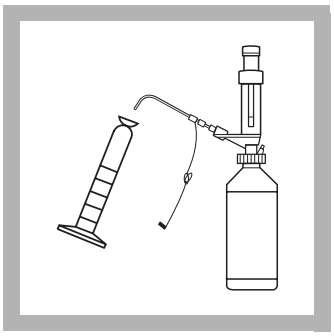
2. Insert Adapter B. Install the Pour-Thru Cell cell with the 1-inch (round) path in line with the adapter arrow. Flush the Pour-Thru cell with 50 mL of deionized water.



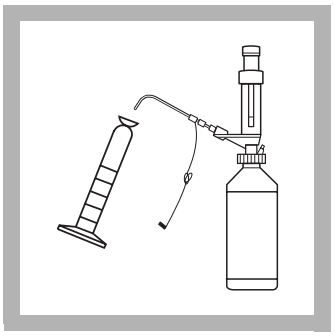
3. Pour approximately 50 mL of sample into the Pour-Thru Cell.



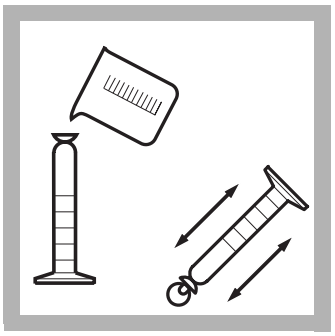
4. When the flow stops, press **ZERO**. The display will show: 0.00 mg/L Cl_2.



5. Add 1.0 mL of Total Chlorine Buffer Solution to a clean, dry 100-mL glass mixing cylinder using the Repipet Jr. Dispenser.



6. Add 1.0 mL of prepared Total Chlorine Indicator Solution to the same mixing cylinder using the Repipet Jr. Dispenser. Swirl to mix the reagents. Proceed to step 7 immediately.

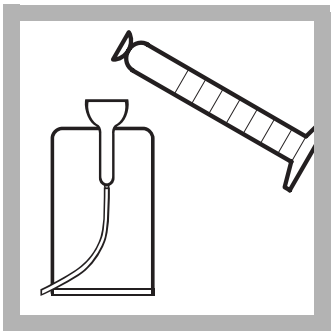


7. Carefully fill the mixing cylinder to the 80-mL mark with sample. Stopper the cylinder and gently invert it twice to mix. Proceed to step 8 immediately.

8. Press **TIMER>OK**.

A two-minute reaction period will begin.

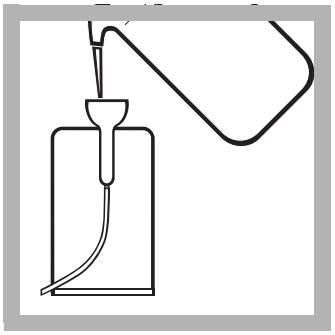
Complete steps 9 and 10 within two minutes after the timer expires.



9. When the timer expires, fill the funnel of the Pour-Thru Cell with the reacted sample from the mixing cylinder.

It is not necessary to pour the entire sample into the Pour-Thru Cell; approximately half of the sample may be discarded.

After the flow stops, press **READ**. Results will appear in mg/L Cl_2.



10. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Reagent Preparation

The Total Chlorine Indicator Solution must be prepared before use. Using a powder funnel, add the contents of one 24 g bottle of DPD Powder* to one 473-mL bottle of Total Chlorine Indicator Solution*. Invert several times and swirl until the powder is completely dissolved. A pink color may develop, but should not affect results.

This solution will give accurate results for at least one month after mixing when stored at 20–25 °C (68–77 °C). Write the date of preparation on the Indicator Solution Bottle. Discard any remaining solution after one month. Use of this reagent after one month may result in high reagent blanks and low values at high concentration. Do not combine fresh reagent with previously mixed reagent.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Alkalinity	Greater than 700 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine, Br_2	Interferes at all levels.
Hardness	Levels below 1000 mg/L as $CaCO_3$ will not interfere.
Hexavalent Chromium	Levels greater than 1 mg/L will cause a positive interference.
Iodine, I_2	Interferes at all levels.
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7 with 1.000 N Sulfuric Acid[1]. **2.** Add 9 drops Potassium Iodide (30 g/L)[1] to an 80-mL sample. **3.** Mix and wait 1 minute. **4.** Add 9 drops Sodium Arsenite[1, 2] (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure above. **6.** Subtract the result of this test from the original analysis to obtain the correct concentration.
Ozone	Interferes at all levels.

[1] See Optional Reagents and Apparatus on page 5.

[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for safe handling and disposal instructions.

Sampling and Storage

Samples must be analyzed immediately and cannot be preserved for later analysis. A common testing error is introduced if the analyst does not obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. Perform the chlorine analysis immediately.

Avoid plastic containers since these may have a chlorine demand. Pre-treat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized water. If sample containers are rinsed thoroughly with deionized water after use, only occasional pretreatment is necessary. A pre-treated BOD bottle with a ground-glass stopper is an ideal sample container for chlorine analysis.

* See Required Reagents on page 5.

Treating Analysis Labware

Glassware used in this test must be chlorine demand-free. Fill the 100-mL mixing cylinder and sample container with a dilute solution of chlorine bleach prepared by adding 1 mL of commercial bleach to 1 liter of water. Soak in this solution at least one hour. After soaking, rinse thoroughly with deionized water and allow to dry before use. If the mixing cylinder is thoroughly rinsed with deionized water and allowed to dry after each use, only occasional pretreatment is necessary. Do not use the same mixing cylinder for Free and Total Chlorine analysis.

Treat the Pour-Thru Cell similarly with dilute bleach and let stand for several minutes. Rinse several times with deionized water.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored reaction products, especially if the reacted solutions are allowed to remain in the cell for long periods after measurement. Remove the buildup by rinsing the cell with 5.25 N Sulfuric Acid* followed by several rinsings with deionized water.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the chlorine voluette ampules. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **Edit** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the top off a Chlorine Voluette® Ampule Standard Solution, 50 to 75-mg/L Cl_2.

5. Prepare three sample spikes. Use the TenSette® Pipet to add 0.3, 0.6 and 0.9 mL of standard to three 80-mL samples, respectively. Swirl gently to mix.

6. Analyze each sample spike as described in the procedure above, starting with the 0.3 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 5.

Summary of Method

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and can be determined together as the total available chlorine. Free chlorine is available as hypochlorous acid or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride, and other chloro derivatives. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run a free chlorine test. Subtract the results from the results of the total chlorine test to obtain combined chlorine. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Rapid Liquid Total Chlorine Reagent Set, includes:	—	—	25570-00
DPD Indicator Powder	varies	24 g	22972-55
Free Chlorine Indicator Solution	1 mL	473 mL	22634-11
Free Chlorine Buffer Solution	1 mL	473 mL	22635-11

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 100-mL, poly	1	each	1896-42
Dispenser, fixed-volume, 1.0-mL, Repipet Jr.	2	each	21113-02
Funnel, powder	1	each	22644-67
Pour-Thru Cell Module Kit	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, Voluette® Ampule, 50-75 mg/L, 10-mL	16/pkg	14268-10
OR		
Chlorine Standard Solution, Voluette® Ampule, 50-75 mg/L, 2-mL	20/pkg	14268-20
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Pipet, TenSette®, 0.1–1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01, 50 pkg	21856-96
Potassium Iodide, 30 g/L 100 mL	343-32
Sodium Arsenite, 5 g/L 100 mL	1047-32
Sulfuric Acid, 1 N 100 mL	1270-32
Sulfuric Acid, 5.25 N 1000 mL	2449-53

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Updated November 2005 Edition 1

Chlorine, Total

★Method 8370 **DPD Method[1]**

Pour-Thru™ Cell **ULR (2 to 500 µg/L as Cl_2)**

Scope and Application: For detecting trace levels of chlorine and chloramines in clean waters relatively free of color and turbidity; USEPA accepted for reporting for drinking water analysis

[1] U.S. Patent 5,362,650

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Test Preparation

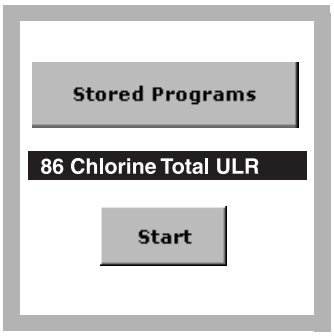
Before starting the test:

Analyze samples immediately. Samples containing chlorine cannot be preserved for later analysis.
A reagent blank value for a combined lot of indicator/buffer reagent solutions should be determined at least once a day. If sample color or turbidity fluctuates frequently during the day, determine a reagent blank for each sample.
Ampules contain more than 1.0 mL of solution for ease of transfer. Discard excess reagent in the ampule.
Refer to the instrument User Manual for Pour-Thru cell and module assembly and installation.
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel. Refer to Treating Analysis Labware on page 6.

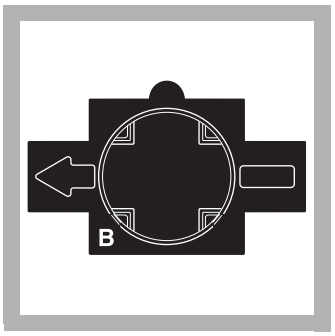
Collect the following items:	**Quantity**
ULR Chlorine Buffer Solution, 1.5-mL ampules	1 mL
DPD Indicator Solution for ULR Chlorine, 1.5-mL ampules	1 mL
Blanking Reagent for ULR Chlorine	1 mL
Beaker, 250 mL	1
Cylinder, graduated mixing, 50-mL.	1
Pipet, TenSette®, 0.1 to 1.0 mL	1
Pipet Tips for TenSette Pipet	2
Pour-Thru Module and cell	1

Note: *Reorder information for consumables and replacement items is on page 8.*

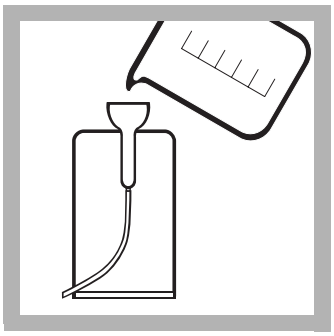
Pour-Thru Cell — Method 8370



1. Select the test.



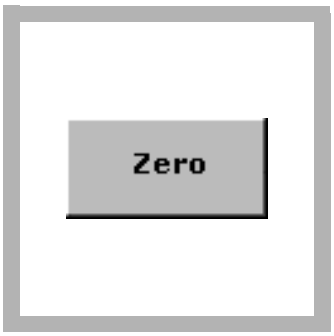
2. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch (round) path in line with the adapter arrow. Flush the Pour-Thru cell with 50 mL of deionized water.



3. Pour at least 50 mL of sample into the Pour-Thru Cell.

4. When the flow stops, press **TIMER>OK**.

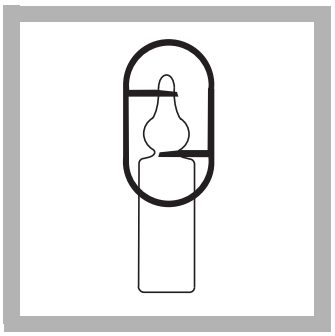
A three-minute reaction period will begin. This time allows turbidity or solids to settle and ensures a stable reading.



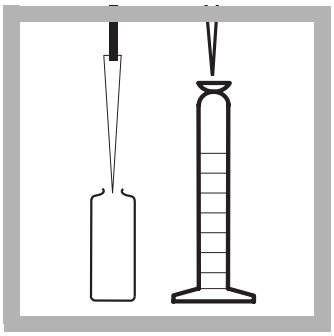
5. When the timer expires, press **ZERO**.

The display will show:

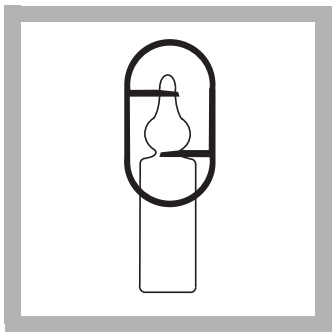
0 µg/L



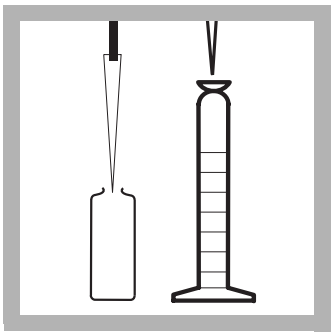
6. Break open one ULR Chlorine Buffer Solution Ampule.



7. Using a TenSette® Pipet and a clean tip, transfer 1.0 mL of buffer from the ampule to a clean, treated 50-mL graduated mixing cylinder.

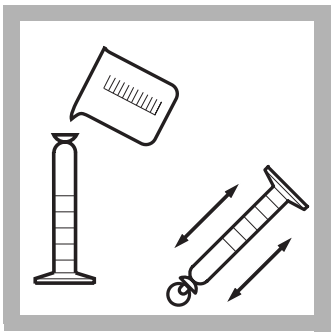


8. Break open one ampule of DPD Indicator Solution for Ultra Low Range Chlorine.



9. Using a TenSette Pipet and a clean tip, transfer 1.0 mL of indicator from the ampule to the graduated mixing cylinder. Swirl to mix.

Proceed to step 10 within one minute.



10. Prepared Sample: Avoiding extra agitation, carefully fill the cylinder to the 50-mL mark with sample. Stopper the cylinder. Gently invert it twice to mix.

11. Press **TIMER>OK**.

A three-minute reaction time will begin.

Measure the reacted sample 3–4 minutes after mixing the sample and reagents. If less than three minutes elapses, the reaction with chloramines may be incomplete. A reading after four minutes may result in higher reagent blank values.

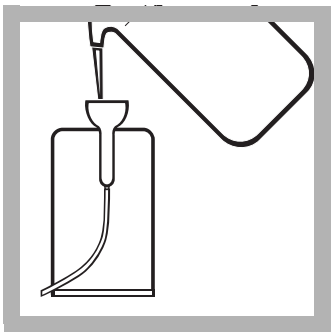
12. Introduce the contents of the graduated mixing cylinder into the Pour-Thru cell.

When the timer expires, press **READ**. Results are in µg/L chlorine.

If a dechlorinating agent (e.g. sulfite or sulfur dioxide) is present, the sample result (corrected for the reagent blank) will read "0" or a slightly negative value.

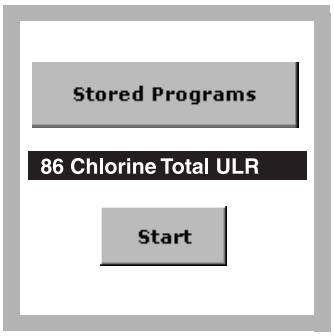
13. When the timer expires, press **READ**. Results are in µg/L chlorine.

If a dechlorinating agent (e.g. sulfite or sulfur dioxide) is present, the sample result (corrected for the reagent blank) will read "0" or a slightly negative value.

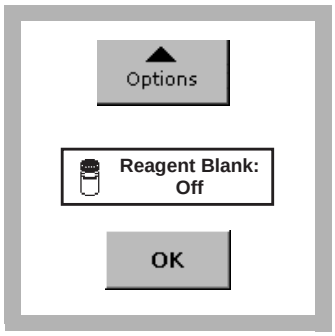


14. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Determining the Reagent Blank Value

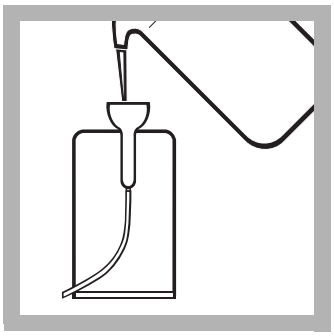


1. Select the test.



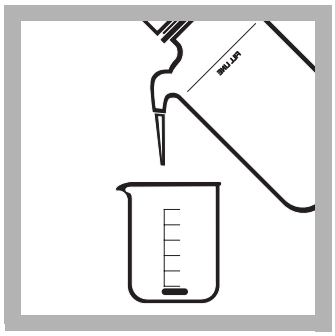
2. Make sure that the reagent blank setting is off.

Press **OPTIONS>MORE> REAGENT BLANK>OFF.** See the user manual for information.

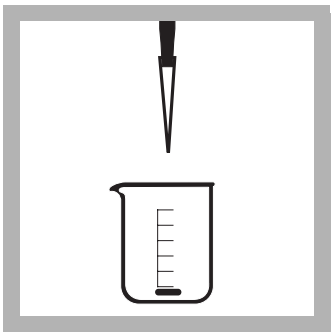


3. Insert Adapter B. Install the Pour-Thru module and cell with the 1-inch (round) path in line with the arrow on the adapter.

Flush the Pour-Thru cell with 50 mL of deionized water.

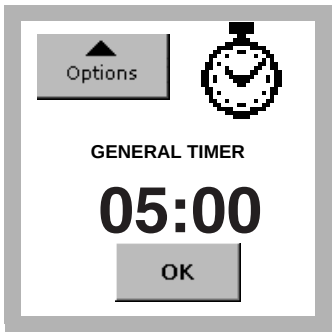


4. Collect about 100 mL of deionized or tap water in a clean, 250-mL beaker.



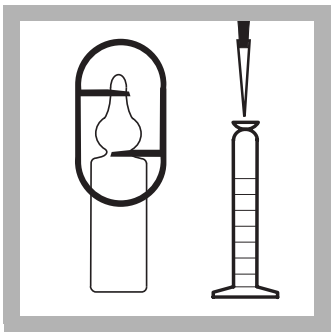
5. Using a TenSette® Pipet, add 1.0 mL of Blanking Reagent to the beaker. Swirl several times to mix.

The Blanking Reagent removes chlorine and chloramines from the water.

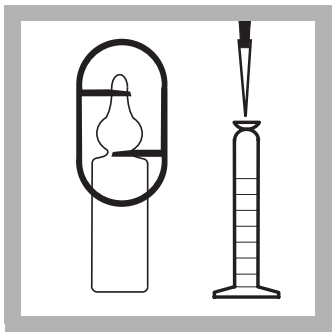


6. Press **OPTIONS>MORE>TIMER> GENERAL TIMER**.

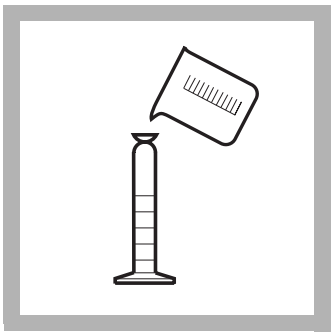
Set a 5-minute timer and press **OK**.



7. After the timer expires, break open one ampule of ULR Chlorine Buffer Solution. Using a TenSette Pipet and clean tip, transfer 1.0 mL of buffer from the ampule to a clean 50-mL mixing graduated cylinder.



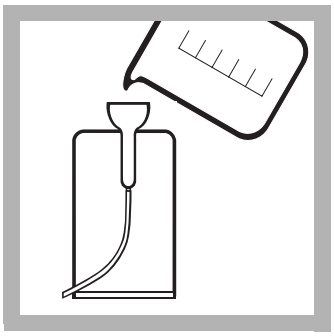
8. Break open one ampule of DPD Indicator Solution for Ultra Low Range Chlorine. Using a TenSette Pipet and a clean tip, transfer 1.0 mL of indicator from the ampule to the cylinder. Swirl to mix the reagents. Proceed to step 9 within one minute.



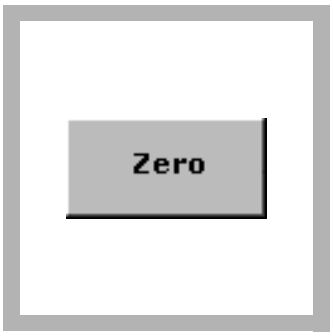
9. Fill the cylinder to the 50-mL mark with dechlorinated water from step 5. Cap and invert twice to mix. Save the remaining water for step 11.

10. Press **TIMER>OK**.

A three-minute reaction time will begin.

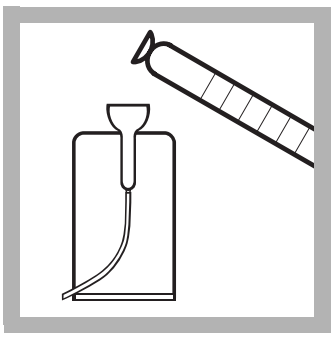


11. During the reaction period, flush the Pour-Thru Cell with the remainder of original dechlorinated water from step 9.

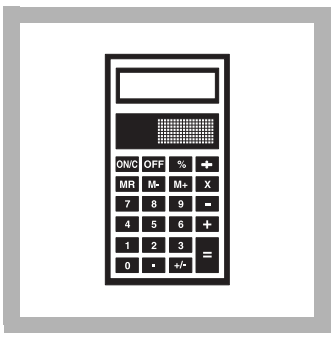


12. When the flow stops, press **ZERO**.

The display will show: 0 µg/L Cl_2.

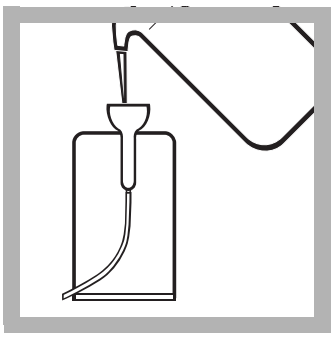


13. When the timer expires, introduce the contents of the cylinder into the Pour-Thru Cell. Press **READ**. Results are in µg/L chlorine.



14. Use this value to correct the sample result obtained in this procedure.

See the user manual for details on saving the reagent blank value.



15. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Copper, Cu^{2+}	Greater than 1000 µg/L
Iodine, I_2	Interferes at all levels.
Iron (Fe^{3+})	Greater than 1000 µg/L

Table 1 Interfering Substances and Levels (continued)

<table>
<tr><th>Interfering Substance</th><th>Interference Levels and Treatments</th></tr>
<tr><td>Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})</td><td>1. Adjust sample pH to 6–7 with 1.000 N Sulfuric Acid[1].
2. Add 9 drops Potassium Iodide (30 g/L)[1] to an 80-mL sample.
3. Mix and wait 1 minute.
4. Add 9 drops Sodium Arsenite[1, 2] (5 g/L) and mix.
5. Analyze the treated sample as described in the procedure above.
6. Subtract the result of this test from the original analysis to obtain the correct concentration.</td></tr>
<tr><td>Nitrite, NO_2^- (uncommon in clean waters)</td><td><table><tr><th>mg/L nitrite</th><th>Apparent µg/L chlorine</th></tr><tr><td>2.0 mg/L</td><td>3 µg/L</td></tr><tr><td>5.0 mg/L</td><td>5 µg/L</td></tr><tr><td>10.0 mg/L</td><td>7 µg/L</td></tr><tr><td>15.0 mg/L</td><td>16 µg/L</td></tr><tr><td>20.0 mg/L</td><td>18 µg/L</td></tr></table></td></tr>
<tr><td>Ozone</td><td>Interferes at all levels</td></tr>
<tr><td>Peroxides</td><td>May interfere</td></tr>
<tr><td>Extreme sample pH or highly buffered samples</td><td>Adjust to pH 6–7</td></tr>
</table>

[1] See Optional Reagents and Apparatus on page 8.
[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for safe handling and disposal instructions.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine immediately after collection. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (0.5 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the chlorine analysis immediately.

Treating Analysis Labware

Glassware used in this test must be chlorine demand-free. Fill the 100-mL mixing cylinder and sample container with a dilute solution of chlorine bleach prepared by adding 1 mL of commercial bleach to 1 liter of water. Soak in this solution at least one hour. After soaking, rinse thoroughly with deionized water and allow to dry before use.

Treat the Pour-Thru Cell similarly with dilute bleach and let stand for several minutes. Rinse several times with deionized water.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored reaction products, especially if the reacted solutions are allowed to remain in the cell for long periods after measurement. Remove the buildup by rinsing the cell with 5.25 N Sulfuric Acid* followed by several rinsings with deionized water.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the chlorine voluette ampules. Multiply the mg/L concentration on the label by 1000 to enter this value as µg/L. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **Edit** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the top off a Low Range Chlorine Voluette® Ampule Standard Solution, 25 to 30-mg/L (25,000 to 30,000 µg/L) Cl_2.

5. Prepare three sample spikes. Use the TenSette® Pipet to add 0.1, 0.2 and 0.3 mL of standard to three 50-mL samples, respectively. Swirl gently to mix.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Summary of Method

This method is designed for clean water, low in color and turbidity. The main applications include monitoring for trace chlorine break-through of activated carbon beds and feedwater to reverse osmosis membranes or ion-exchange resins.

Several modifications to the normal DPD chlorine method are necessary to measure trace levels of chlorine. The Pour-Thru Cell must be used in the spectrophotometer. Liquid reagents are also required. The reproducible optics of the Pour-Thru Cell give more stable readings than is possible with movable sample cells, resulting in more stable measurements.

The reagents are packaged in ampules and sealed under argon gas to ensure stability. Use of liquid reagents eliminates any slight turbidity that might be caused by using powdered reagents. Due to the possible oxidation of the reagents (which could give a positive chlorine reading in the blank), a reagent blank must be determined at least once a day for each lot of reagent used. This reagent blank value is subtracted from the sample result and the corrected value is the actual chlorine concentration. Test results are measured at 515 nm.

* See Optional Reagents and Apparatus on page 8.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
ULR Chlorine Reagent Set (approximately 20 tests), includes:			25630-00
ULR Chlorine Buffer Solution, 1.5-mL ampules	1 mL	20/pkg	24931-20
DPD Indicator Solution for ULR Chlorine, 1.5-mL ampules	1 mL	20/pkg	24932-20
Blanking Reagent for ULR Chlorine	1 mL	29 mL	24930-23

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Beaker, 250-mL	1	each	500-46H
Cylinder, graduated mixing, 50-mL	1	each	1896-41
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg	21856-96
Pour-Thru Cell Module Kit	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, Voluette® Ampule, 25–30 mg/L, 2-mL	20/pkg	26300-20

Optional Reagents and Apparatus

Description	Cat. No.
Potassium Iodide, 30 g/L 100 mL	343-32
Sodium Arsenite, 5 g/L 100 mL	1047-32
Sulfuric Acid, 1 N 100 mL	1270-32
Sulfuric Acid, 5.25 N 1000 mL	2449-53

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Updated November 2005 Edition 1

Chlorine, Total

★Method 10014

DPD Method[1]

Pour-Thru Cell and OriFlo™ Filtration

ULR (2 to 500 µg/L as Cl_2)

Scope and Application: For detecting trace levels of chlorine and chloramines in clean waters relatively free of color and turbidity; USEPA accepted for reporting for wastewater analysis[2].

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

[2] U.S. Patent 5,362,650 covers the procedure. U.S. Patent 5,549,816 covers the OriFlo™ Filtration System.

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Test Preparation

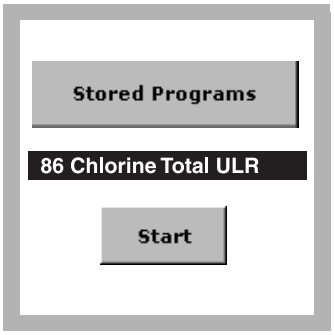
Before starting the test:

Analyze samples immediately. Samples containing chlorine cannot be preserved for later analysis.
A reagent blank value for a combined lot of indicator/buffer reagent solutions should be determined at least once a day. If sample color or turbidity fluctuates frequently during the day, determine a reagent blank for each sample. Refer to Treating Analysis Labware on page 7.
The reagent blank value is normally less than 5 µg/L. If the value is greater than 5 µg/L, an interfering substance may be present in the blanking water or the DPD Indicator may be degrading. If there is doubt about the reagents, repeat the reagent blank determination using chlorine-demand-free water for the sample. Blanks up to 5 µg/L may be used.
Use a new filter for each test. Using an unspecified filter may give low analysis results or inability to filter the required volume.
Ampules contain more than 1.0 mL of solution for ease of transfer. Discard excess reagent in the ampule.
Refer to the instrument User Manual for Pour-Thru cell and module assembly and installation.
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel.

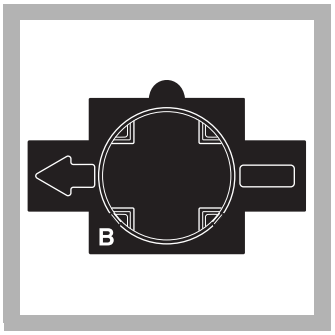
Collect the following items:	**Quantity**
ULR Chlorine Buffer Solution, 1.5-mL ampules	1 mL
DPD Indicator Solution for ULR Chlorine, 1.5-mL ampules	1 mL
Blanking Reagent for ULR Chlorine	1 mL
Membrane Filters, 3-micron, 25-mm	1
OriFlo Assembly	1
Beaker, 250 mL	1
Cylinder, graduated mixing, 50-mL.	1
Pipet, TenSette®, 0.1 to 1.0 mL	1
Pipet Tips	2
Pour-Thru Module and cell	1

Note: *Reorder information for consumables and replacement items is on page 9.*

Pour-Thru Cell — Method 8370

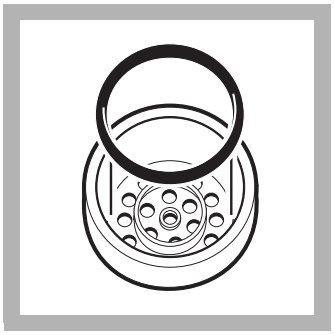


1. Select the test.

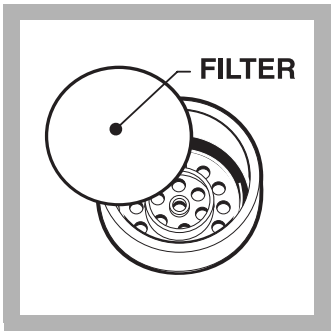


2. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch (round) path in line with the adapter arrow.

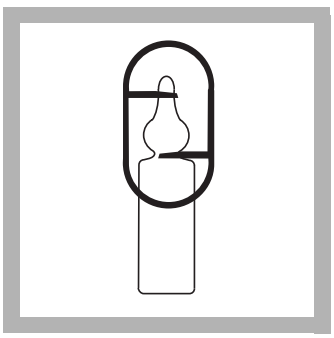
Flush the Pour-Thru cell with 50 mL of deionized water.



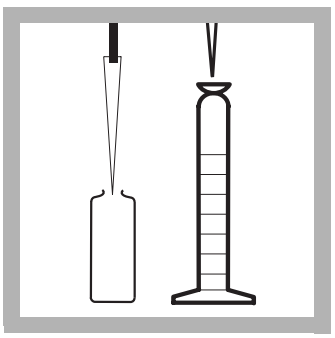
3. Unscrew the cap from the OriFlo™ plunger assembly. Be sure that the O-ring is properly seated in the cap.



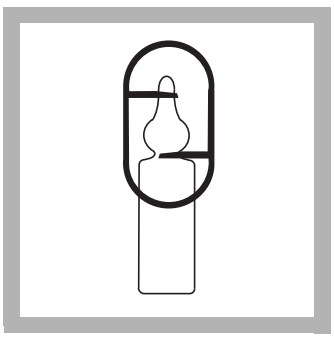
4. Install a new, 3-micron filter into the cap well. Wet the filter with a few drops of deionized water. Reassemble and hand-tighten the cap onto the plunger.



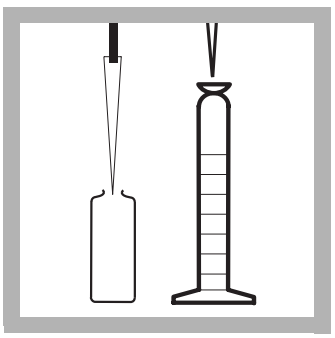
5. Break open one ULR Chlorine Buffer Solution Ampule.



6. Using a TenSette® Pipet and a clean tip, transfer 1.0 mL of buffer from the ampule to a clean, treated 50-mL graduated mixing cylinder.

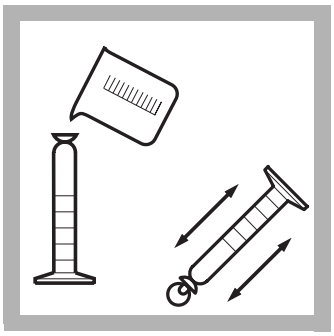


7. Break open one ampule of DPD Indicator Solution for Ultra Low Range Chlorine.



8. Using a TenSette Pipet and a clean tip, transfer 1.0 mL of indicator from the ampule to the graduated mixing cylinder. Swirl to mix.

Proceed to step 9 within one minute.

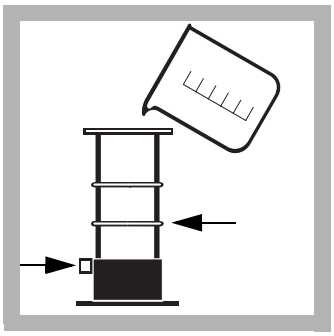


9. Prepared Sample: Avoiding extra agitation, carefully fill the cylinder to the 50-mL mark with sample. Stopper the cylinder. Gently invert it twice to mix.

10. Press **TIMER>OK**.

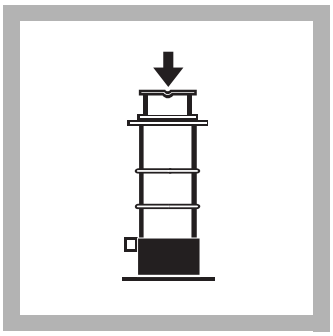
A three-minute reaction time will begin. Perform steps 11–16 during this period.

Measure the reacted sample 3–6 minutes after mixing the sample and reagents. If less than three minutes elapses, the reaction with chloramines may be incomplete. A reading after six minutes may result in higher reagent blank values.

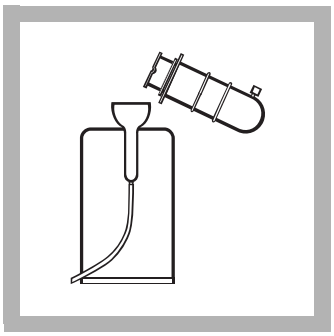


11. Push the valve button on the OriFlo™ barrel assembly to the “closed” position. Place the barrel assembly into its stand. Pour approximately 50 mL of the original sample into the barrel.

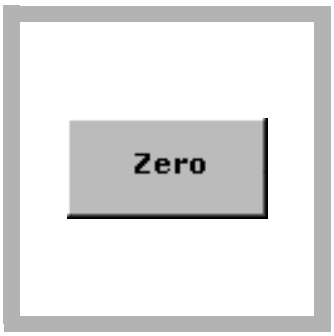
The lower ring on the barrel assembly represents about a 50-mL volume.



12. Insert the plunger into the barrel and slowly push the plunger down with even pressure, until the plunger is fully seated.

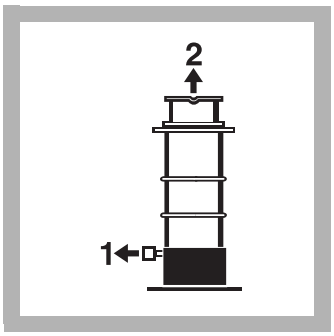


13. Pour the filtered sample from the plunger reservoir into the Pour-Thru Cell.



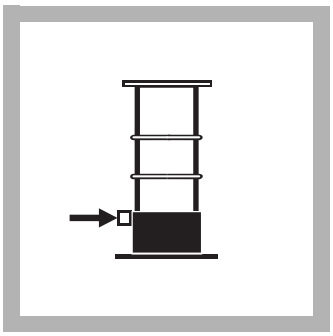
14. When the flow stops, press **ZERO**.

The display will show: 0 µg/L Cl_2.

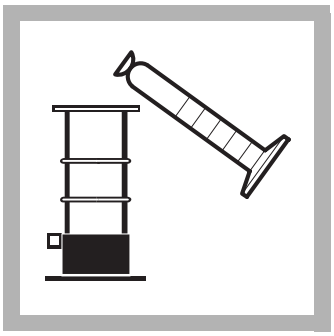


15. Pull the barrel valve button out to the “open” position. Pull the plunger up to separate it from the barrel assembly. Discard the remaining unfiltered sample.

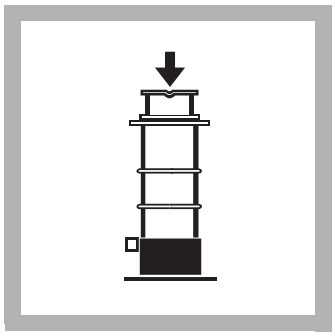
A new membrane may be required for very turbid samples. Alternatively, use a second Quick Filter unit with a new membrane filter installed.



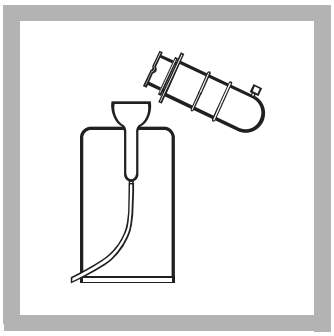
16. Push the barrel valve button to the “closed” position. Place the barrel assembly into its stand.



17. When the timer expires, pour the contents of the mixing cylinder into the barrel.



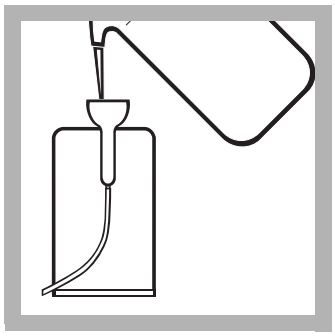
18. Insert the plunger into the barrel and slowly push the plunger down with even pressure, until the plunger is fully seated.



19. Pour the filtered, reacted sample from the plunger reservoir into the Pour-Thru Cell.

Press **READ**. Results are in µg/L chlorine.

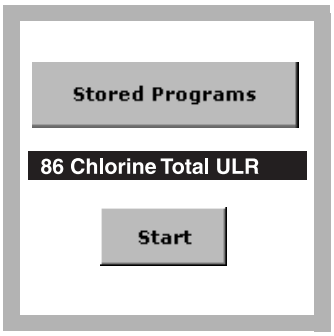
If a dechlorinating agent (e.g., sulfite or sulfur dioxide) is present, the sample result, corrected for the reagent blank, will read "0" or a slightly negative value.



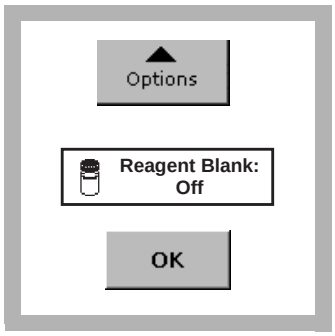
20. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Subtract the reagent blank value (Determining the Reagent Blank Value) from the sample value obtained in step 19.

Determining the Reagent Blank Value

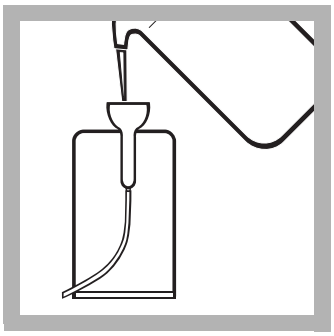


1. Select the test.



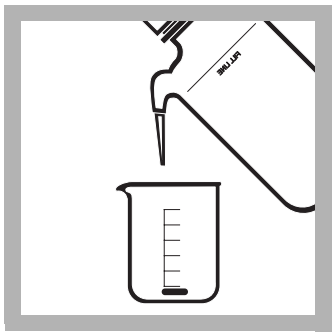
2. Make sure that the reagent blank setting is off.

Press **OPTIONS>MORE> REAGENT BLANK>OFF.** See the user manual for information.

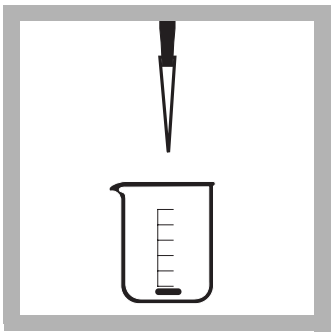


3. Insert Adapter B. Install the Pour-Thru module and cell with the 1-inch (round) path in line with the arrow on the adapter.

Flush the Pour-Thru cell with 50 mL of deionized water.

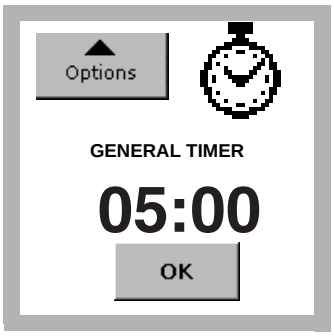


4. Collect about 100 mL of deionized or tap water in a clean, 250-mL beaker.



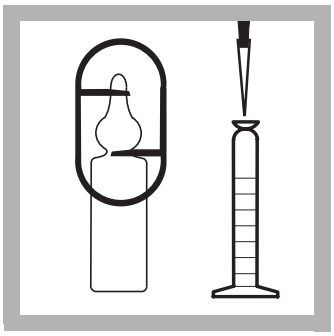
5. Using a TenSette Pipet, add 1.0 mL of Blanking Reagent to the beaker. Swirl several times to mix.

The Blanking Reagent removes chlorine and chloramines from the water.

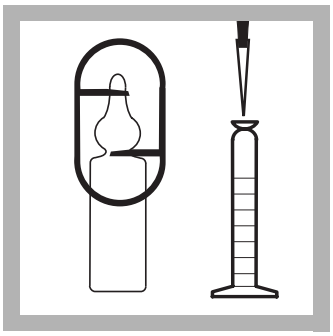


6. Press **OPTIONS>MORE>TIMER> GENERAL TIMER**.

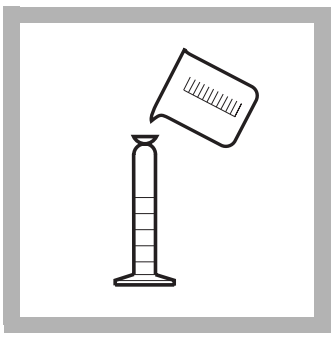
Set a 5-minute timer and press **OK**. A five-minute dechlorination period will begin.



7. After the timer expires, break open one ampule of ULR Chlorine Buffer Solution. Using a TenSette Pipet and clean tip, transfer 1.0 mL of buffer from the ampule to a clean 50-mL mixing graduated cylinder.



8. Break open one ampule of DPD Indicator Solution for Ultra Low Range Chlorine. Using a TenSette Pipet and a clean tip, transfer 1.0 mL of indicator from the ampule to the cylinder. Swirl to mix the reagents. Proceed to step 9 within one minute.

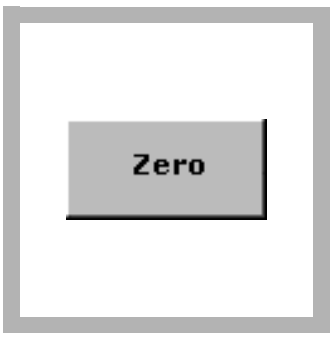


9. Fill the cylinder to the 50-mL mark with dechlorinated water from step 5. Cap and invert twice to mix. Save the remaining water for step 11.

10. Press **TIMER>OK**.

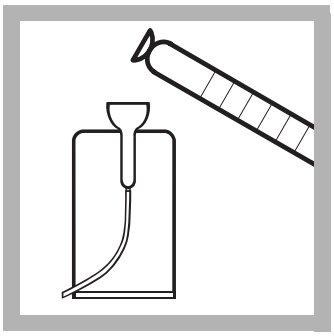
A three-minute reaction time will begin.

11. During the reaction period, flush the Pour-Thru Cell with the remainder of original dechlorinated water from step 9.

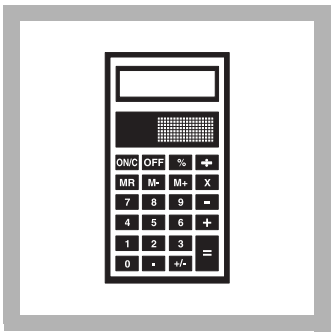


12. When the flow stops, press **ZERO**.

The display will show: 0 µg/L Cl_2.

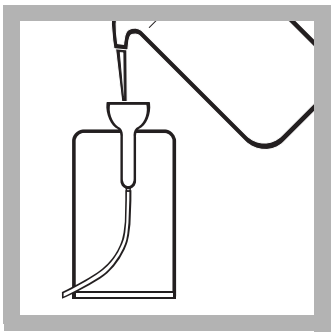


13. When the timer expires, introduce the contents of the cylinder into the Pour-Thru Cell. When the flow stops, press **READ**. Results are in µg/L chlorine.



14. Use this value to correct the sample result obtained in this procedure.

See the user manual for details on saving the reagent blank value.



15. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Bromine, Br_2	Interferes at all levels.
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Copper, Cu^{2+}	Greater than 1000 µg/L
Iodine, I_2	Interferes at all levels.
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7 with 1.000 N Sulfuric Acid[1]. **2.** Add 9 drops Potassium Iodide (30 g/L)[1] to an 80-mL sample. **3.** Mix and wait 1 minute. **4.** Add 9 drops Sodium Arsenite[1, 2] (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure above. **6.** Subtract the result of this test from the original analysis to obtain the correct concentration.
Nitrite, NO_2^- (uncommon in clean waters)	See nested table below

mg/L nitrite	Apparent µg/L chlorine
2.0 mg/L	3 µg/L
5.0 mg/L	5 µg/L
10.0 mg/L	7 µg/L
15.0 mg/L	16 µg/L
20.0 mg/L	18 µg/L

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Ozone	Interferes at all levels.
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust to pH 6–7

[1] See Optional Reagents and Apparatus on page 9.
[2] Samples treated with sodium arsenite for interferences will be hazardous waste as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for safe handling and disposal instructions.

Sample Collection, Storage, and Preservation

Analyze samples for chlorine immediately after collection. Chlorine is a strong oxidizing agent and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is not obtaining a representative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no head space (air) above the sample. Perform the chlorine analysis immediately.

Treating Analysis Labware

Glassware used in this test must be chlorine demand-free. Fill the 100-mL mixing cylinder and sample container with a dilute solution of chlorine bleach prepared by adding 1 mL of commercial bleach to 1 liter of water. Soak in this solution at least one hour. After soaking, rinse thoroughly with deionized water and allow to dry before use.

Treat the Pour-Thru Cell similarly with dilute bleach and let stand for several minutes. Rinse several times with deionized water.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored reaction products, especially if the reacted solutions are allowed to remain in the cell for long periods after measurement. Remove the buildup by rinsing the cell with 5.25 N Sulfuric Acid* followed by several rinsings with deionized water.

* See Optional Reagents and Apparatus on page 9.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A keypad will appear. Enter the average chlorine concentration shown on the certificate enclosed with the chlorine voluette ampules. Multiply the mg/L value from the label by 1000, to enter this as µg/L. Press **OK**.

3. A summary of the Standard Additions procedure will appear. Press **OK** to accept the values for standard concentration, sample volume, and spike volumes as shown. Press **Edit** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the top off a Low Range Chlorine Voluette® Ampule Standard Solution, 25 to 30-mg/L (25,000 to 30,000 µg/L).

5. Prepare three sample spikes. Use the TenSette® Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 50-mL samples, respectively. Swirl gently to mix.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Summary of Method

It is essential that interfering sample turbidity is removed using a 3-micron membrane filter. To avoid chlorine loss, the filtration is done after reacting the DPD with the chlorine in the sample. The filter used has been specifically selected to avoid retention of the colored product. Sample color is compensated by zeroing the spectrophotometer on a filtered sample.

Several modifications to the normal DPD chlorine method are necessary to measure trace levels of chlorine. The Pour-Thru Cell must be used in the spectrophotometer. Liquid reagents are also required. The reproducible optics of the Pour-Thru Cell give more stable readings than is possible with movable sample cells, resulting in more stable measurements.

The reagents are packaged in ampules and sealed under argon gas to ensure stability. Use of liquid reagents eliminates any slight turbidity that might be caused by using powdered reagents. Due to the possible oxidation of the reagents (which could give a positive chlorine reading in the blank), a reagent blank must be determined at least once a day for each lot of reagent used. This reagent blank value is subtracted from the sample result and the corrected value is the actual chlorine concentration. Test results are measured at 515 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
ULR Chlorine Reagent Set (approximately 20 tests), includes:	—	—	25630-00
ULR Chlorine Buffer Solution, 1.5-mL ampules	1 mL	20/pkg	24931-20
DPD Indicator Solution for ULR Chlorine, 1.5-mL ampules	1 mL	20/pkg	24932-20
Blanking Reagent for ULR Chlorine	1 mL	29 mL	24930-23

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
ULR Chlorine Apparatus Set, includes:	—	—	25956-00
Membrane Filters, 3-micron, 25-mm	1	25/pkg	25940-25
OriFlo™ Assembly	1	each	49660-00
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Beaker, 250-mL	1	each	500-46H
Cylinder, graduated mixing, 50-mL	1	each	1896-41
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg	21856-96
Pour-Thru Cell Module Kit	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Chlorine Standard Solution, Voluette® Ampule, 25–30 mg/L, 2-mL	20/pkg	26300-20

Optional Reagents and Apparatus

Description	Cat. No.
Potassium Iodide, 30 g/L 100 mL	343-32
Sodium Arsenite, 5 g/L 100 mL	1047-32
Sulfuric Acid, 1 N 100 mL	1270-32
Sulfuric Acid, 5.25 N 100 mL	2449-53

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Updated November 2005 Edition 1

Chromium, Hexavalent

★Method 8023 — 1,5-Diphenylcarbohydrazide Method[1]

Powder Pillows or AccuVac® Ampuls — (0.010 to 0.700 mg/L Cr^{6+})

Scope and Application: For water and wastewater;
USEPA accepted for reporting for wastewater analysis[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.
[2] Procedure is equivalent to USGS method 1-1230-85 for wastewater.

Test Preparation

Before starting the test:

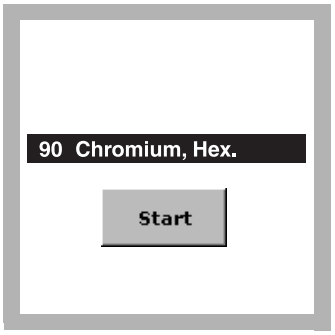
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.
At high chromium levels, a precipitate will form. Sample dilution may be necessary.
The final samples are highly acidic. Neutralize to pH 6–9 with Sodium Hydroxide Standard Solution and refer to reagent MSDS sheets for disposal information.

Collect the following items:	**Quantity**
Powder Pillow Test:	
ChromaVer® 3 Chromium Reagent Powder Pillows	1
Sample cells, 1-in. square, 10-mL	2
AccuVac Test:	
Collect at least 40 mL of sample in a 50-mL beaker	40 mL
ChromaVer® 3 AccuVac® Ampuls	1
Beaker, 50-mL (AccuVac test)	1
Sample cell, 10-mL round, with cap	1

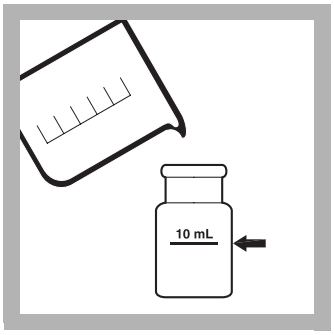
Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows — Method 8023

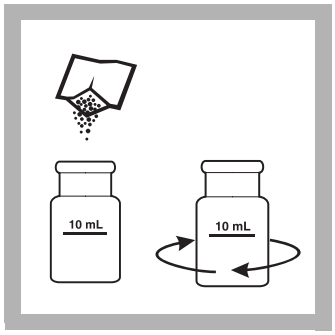
1. Press **STORED PROGRAMS**.



2. Select the test.



3. Fill a square sample cell with 10 mL of sample.

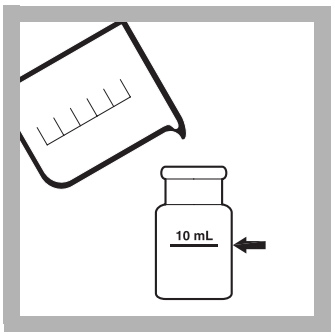


4. Prepared Sample: Add the contents of one ChromaVer® 3 Reagent Powder Pillow to the sample cell. Swirl to mix.

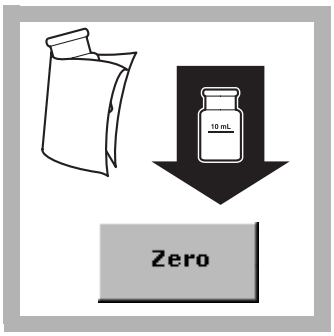
A purple color will form if hexavalent chromium is present.

5. Press **TIMER>OK**.

A five-minute reaction period will begin.



6. Blank Preparation: Fill a second square sample cell with 10 mL of sample.



7. When the timer expires, insert the blank into the cell holder with the fill line facing right.

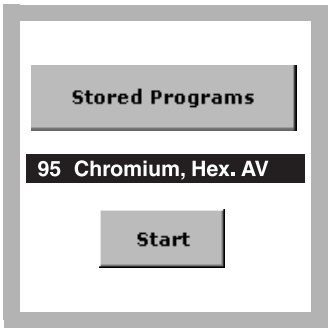
Press **ZERO**. The display will show:

0.000 mg/L Cr^{6+}

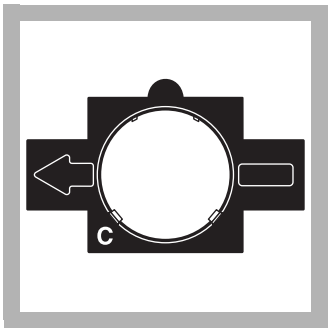
8. Insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Cr^{6+}.

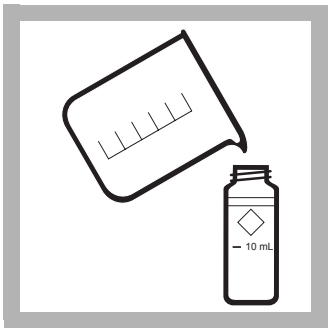
AccuVac Ampul® Method 8023



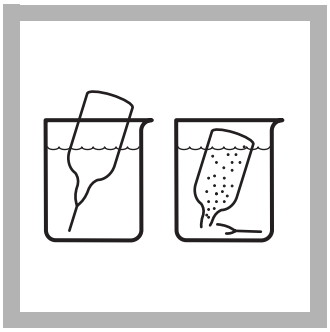
1. Select the test.



2. Insert Adapter C.



3. Blank Preparation: Fill a round sample cell with 10-mL of sample.

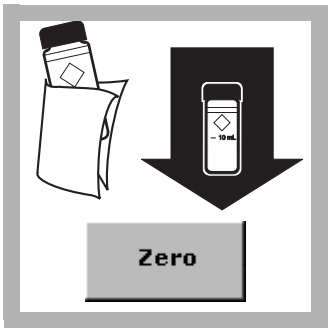


4. Prepared Sample: Fill a ChromaVer 3 Reagent AccuVac® Ampul with sample from the beaker. Keep the tip immersed while the Ampul fills completely.

5. Quickly invert the Ampul several times to mix. Wipe off any liquid or fingerprints.

6. Press **TIMER>OK**.

A five-minute reaction period will begin.



7. When the timer expires, insert the blank into the cell holder.

Press **ZERO**. The display will show:

0.000 mg/L Cr^{6+}

8. Insert the prepared sample into the cell holder.

Press **READ**. Results are in mg/L Cr^{6+}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Iron	May interfere above 1 mg/L
Mercurous & Mercuric Ions	Interfere slightly
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Vanadium	May interfere above 1 mg/L. Allow 10 minutes for the reaction period before reading.
Turbidity	For turbid samples, treat the blank with the contents of one Acid Reagent Powder Pillow[1]. This will ensure that any turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent will also be dissolved in the blank.

[1] See Optional Reagents and Apparatus on page 6

Sample Collection, Preservation, and Storage

Collect samples in a cleaned glass or plastic container. Store at 4 °C (39 °F) up to 24 hours. Samples must be analyzed within 24 hours.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell, or AccuVac Ampul (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Chromium Voluette® Ampule Standard, 12.5 mg/L Cr^{6+}.

5. For analysis using powder pillows, use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to three 25-mL samples and mix thoroughly. Transfer 10 mL of each solution into a 10-mL sample cell and analyze as described above.

Note: *For AccuVac Ampuls, fill three mixing cylinders* with 50-mL of sample and spike with 0.2 mL, 0.4 mL, and 0.6 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers. Analyze each standard addition sample as described in the procedure above.*

6. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

*See Optional Reagents and Apparatus on page 6.

Standard Solution Method
Prepare a 0.50-mg/L Cr^{6+} standard solution daily, as follows:

1. Using a 5.00 mL pipet transfer 5.00 mL of Hexavalent Chromium Standard Solution, 50 mg/L, into a Class A 500-mL volumetric flask.

2. Perform the hexavalent chromium procedure as described above.

3. Dilute to the mark with deionized water. Perform the test procedure as described above.

4. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

5. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Hexavalent chromium is determined by the 1,5-Diphenylcarbohydrazide method using a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains an acidic buffer combined with 1,5-Diphenylcarbohydrazide, which reacts to give a purple color when hexavalent chromium is present. Test results are measured at 540 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
ChromaVer® 3 Chromium Reagent Powder Pillows	1	100/pkg	12710-99
OR			
ChromaVer® 3 AccuVac® Ampuls	1	25/pkg	25050-25
Deionized Water	varies	4 L	272-56

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Chromium, Hexavalent Standard Solution, 10-mL Voluette® Ampules, 12.5-mg/L Cr^{6+}	16/pkg	14256-10
Chromium, Hexavalent Standard Solution, Chromium, Standard Solution, 50.0-mg/L Cr^{6+}	100 mL	810-42H

Optional Reagents and Apparatus

Description	Cat. No.
Acid Reagent Powder Pillow	2126-99
Flask, volumetric, Class A, 500-mL	14574-49
Pipet, 5.00 mL	14515-37
Sodium Hydroxide Standard Solution	2450-26

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Updated November 2005 Edition 1

Chromium, Total

Method 8024 — Alkaline Hypobromite Oxidation Method[1, 2]

Powder Pillows — (0.01 to 0.70 mg/L)

Scope and Application: For water and wastewater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

[2] This procedure is equivalent to Standard Method 3500-CRD for wastewater.

Test Preparation

Before starting the test:

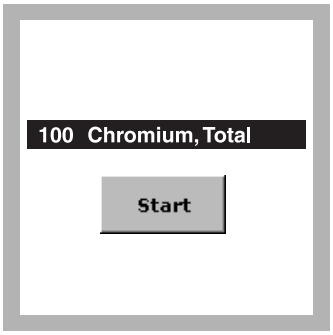
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Prepare a boiling water bath for step 5. Use finger cots to handle hot sample cells.

Collect the following items:	**Quantity**
Acid Reagent Powder Pillows	1
ChromaVer® 3 Chromium Reagent Powder Pillows	1
Chromium 1 Reagent Powder Pillows	1
Chromium 2 Reagent Powder Pillows	1
Hot Plate	1
Water Bath and Rack	1
Finger Cots	varies
Sample Cells, 1-inch square	2
Sample Cell, 1-inch round, 10–20–25 mL, with cap	1

Note: *Reorder information for consumables and replacement items is on page 5.*

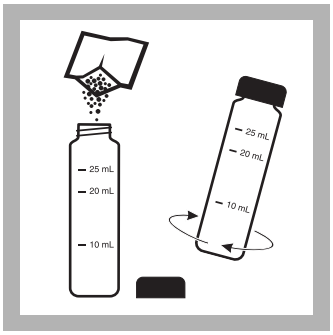
Powder Pillows — Method 8024

1. Press **STORED PROGRAMS**.

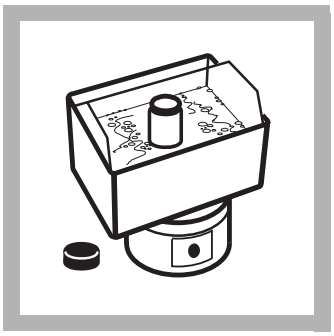


2. Select the test.

3. Fill a 25-mL sample cell with 25 mL of sample.



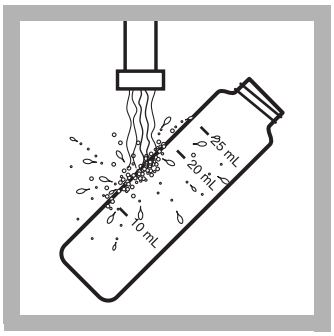
4. Prepared Sample: Add the contents of one Chromium 1 Reagent Powder Pillow. Swirl to mix.



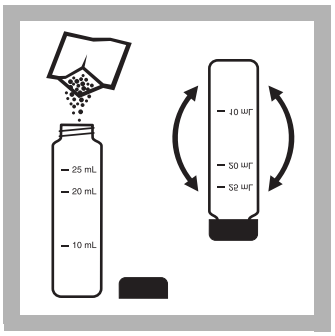
5. Insert the prepared sample into a boiling water bath.

6. Press **TIMER>OK**.

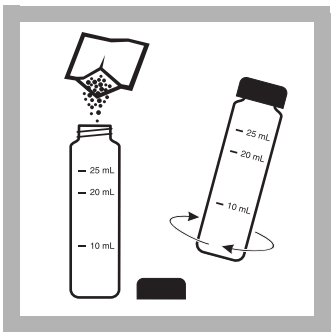
A five-minute reaction period will begin.



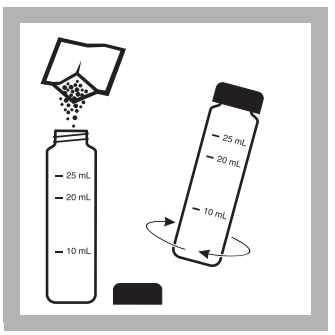
7. When the timer expires, remove the prepared sample. Using running water, cool the cell to 25 °C.



8. Remove cap and add the contents of one Chromium 2 Reagent Powder Pillow. Cap and invert to mix.



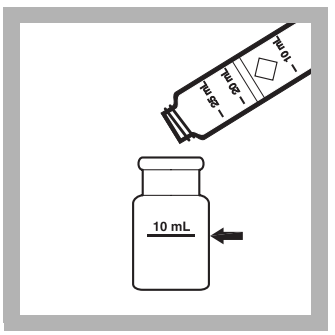
9. Add the contents of one Acid Reagent Powder Pillow. Swirl to mix.



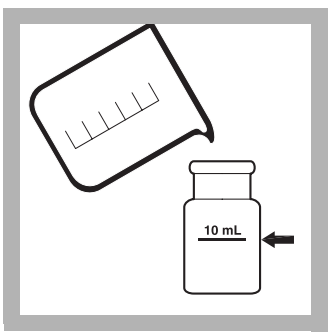
10. Add the contents of one ChromaVer 3 Chromium Reagent Powder Pillow. Swirl to mix.

11. Press **TIMER>OK**.

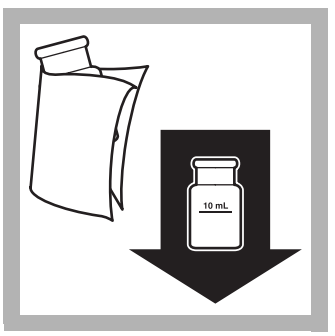
A five-minute reaction period will begin.



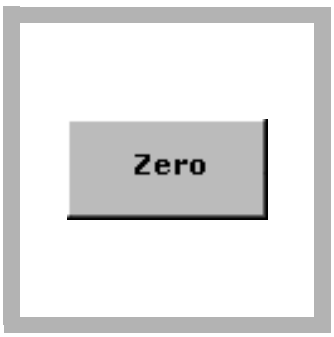
12. While the sample is reacting, pour 10 mL from the mixing bottle into a square sample cell.



13. Blank Preparation: When the timer expires, fill another sample cell with 10 mL of sample.



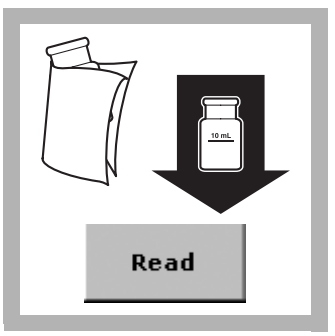
14. Wipe the blank and insert it into the cell holder with the fill line facing right.



15. Press **Zero**.

The display will show:

0.00 mg/L Cr



16. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Cr.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.
Organic material	May inhibit complete oxidation of trivalent chromium. If high levels of organic material are present, digestion may be required. Perform the analysis as described in this procedure on the digested sample.
Turbidity	For turbid samples, treat the 25-mL blank and the sample the same during steps 3–9.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed glass or plastic containers. To preserve samples, adjust the pH to 2 or less with nitric acid. This requires approximately 2 mL per liter of the acid. Store preserved samples at room temperature up to six months. Adjust the pH to about 4 with 5.0 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Trivalent Chromium Voluette® Ampule Standard, 12.5-mg/L as Cr^{3+}.

5. Prepare three sample spikes. Fill three mixing cylinders with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method
Prepare a 0.50-mg/L trivalent chromium standard as follows:

1. Dilute 5.00 mL of Trivalent Chromium Standard Solution, 50-mg/L as Cr^{3+}, to 500 mL with deionized water. Prepare this solution daily.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Trivalent chromium in the sample is oxidized to the hexavalent form by hypobromite ion under alkaline conditions. The sample is acidified. The total chromium content is determined by the 1,5-Diphenylcarbohydrazide method. Determine trivalent chromium by subtracting the results of a separate hexavalent chromium test from the results of the total chromium test.
Test results are measured at 540 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Total Chromium Reagent Set (100 tests), includes:	—	—	22425-00
Acid Reagent Powder Pillows	1	100/pkg	2126-99
ChromaVer® 3 Chromium Reagent Powder Pillows	1	100/pkg	12066-99
Chromium 1 Reagent Powder Pillows	1	100/pkg	2043-99
Chromium 2 Reagent Powder Pillows	1	100/pkg	2044-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Sample Cell, 10-20-25 mL, with cap	1	each	24019-06
Hot plate, 3½-inch diameter, 120 VAC, 50/60 Hz	1	each	12067-01
OR			
Hot Plate, 4-inch diameter, 240 VAC, 50/60 Hz	1	each	12067-02
Water Bath and Rack	1	each	1955-55

Recommended Standards

Description	Unit	Cat. No.
Chromium, Trivalent, Standard Solution, 50-mg/L Cr^{3+}	100 mL	14151-42
Chromium, Trivalent, Standard Sol., 12.5-mg/L Cr^{3+}, Voluette Ampule®, 10-mL	16/pkg	14257-10

Optional Reagents and Apparatus

Description	Cat. No.
Acid Reagent Powder Pillow	2126-99
Finger Cots	14647-02
Flask, volumetric, Class A, 500-mL	14574-49
PIpette, volumetric, Class A, 5.00 mL	14515-37

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Updated November 2005 Edition 1

Chromium, Total and Hexavalent

Method 10218 (Chromium, Hexavalent)
Method 10219 (Chromium, Total) **1,5-Diphenylcarbohydrazide Method**
TNTplus™ 854 **(0.03 to 1.00 mg/L Cr)**

Scope and Application: For wastewater and process analysis

Test Preparation

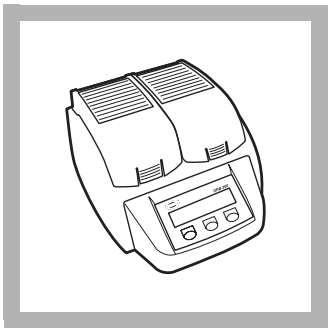
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Recommended sample pH is 3–9.
Recommended sample and reagent temperature is 15–35 °C (59–95 °F). Incorrect results may be obtained if test is not performed at the recommended temperature.
Recommended reagent storage is 2–8 °C (35.6–46.4 °F).
The concentration of trivalent chromium is obtained mathematically from the difference between chromium (total) and chromium VI (hexavalent).
TNTplus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

Collect the following items:	**Quantity**
Chromium, Total and Hexavalent, TNT854 Reagent set	1
Light Shield	1
Pipettor for 2.0 mL sample	1
Pipettor Tips	varies
Test Tube Rack	1

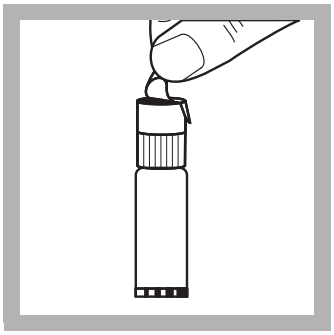
Note: *Reorder information for consumables and replacement items is on page 7.*

TNTplus Chromium, Total Method 10219

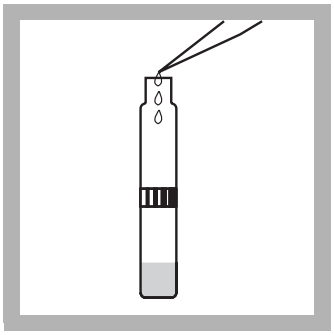


1. Turn on the DRB200 Reactor. Preheat to 100 °C.

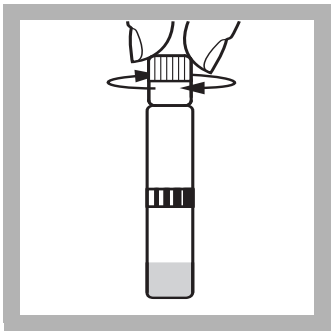
Note: *For DRB200 Reactors with 16-mm wells, insert a 16-mm to 13-mm adapter sleeve into each well* ***before*** *turning on the reactor.*



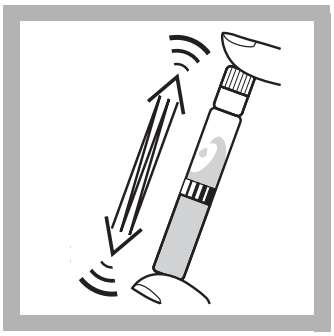
2. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



3. Pipet 2.0 mL of sample into the vial.

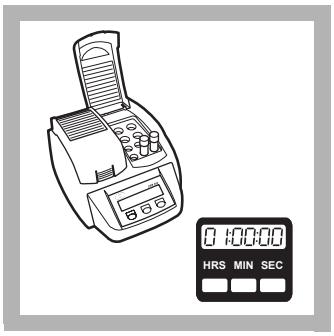


4. Flip the DosiCap ***Zip*** over so that the reagent side faces the vial. Screw the cap tightly onto the vial.



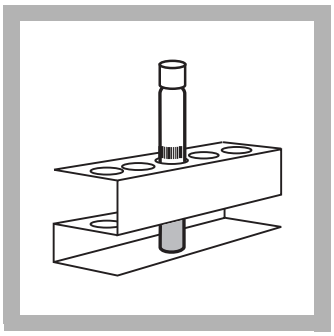
5. Shake the capped vial 2–3 times to dissolve the reagent in the cap.

Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

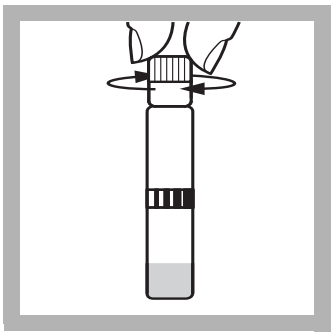


6. Heat the vials for one hour at 100 °C.

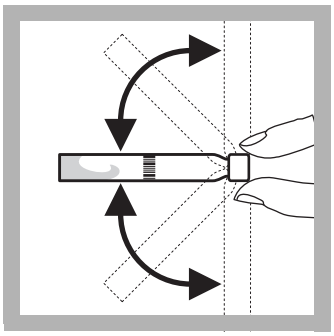
Install the Light Shield in Cell Compartment #2.



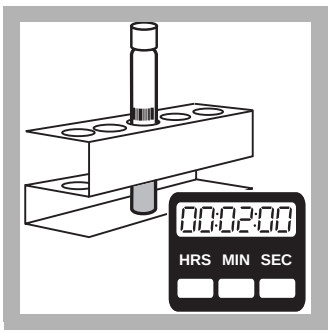
7. When the timer expires remove the hot vials from the reactor. Cool the vials to 15–35 °C. **Do not invert** the vial after digestion.



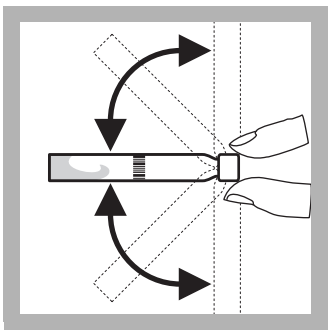
8. Screw an orange DosiCap B onto the cooled vial.



9. Invert the vial 2-3 times to mix.



10. After inverting the tube, allow the vial to sit undisturbed for 2–3 minutes.



11. After the timer expires, invert the vial again 2–3 times.

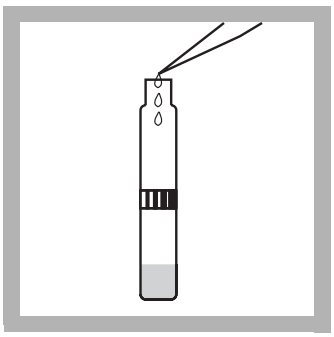
12. Thoroughly clean the outside of the vial.

Insert the prepared vial into the cell holder.

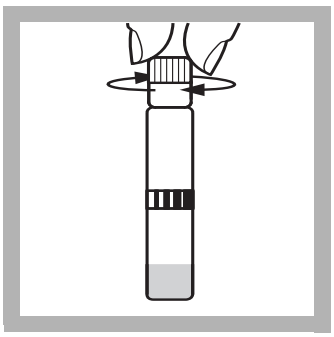
The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L Cr.

No instrument Zero is required.

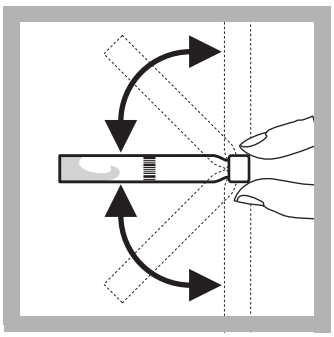
TNTplus Chromium, Hexavalent — Method 10218



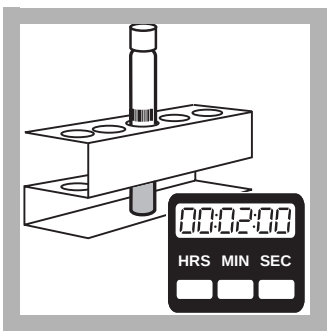
1. Pipet 2.0 mL of sample into the vial.



2. Screw an orange DosiCap B onto the cooled vial.

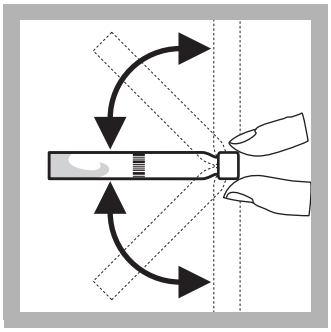


3. Invert the vial 2-3 times to mix.

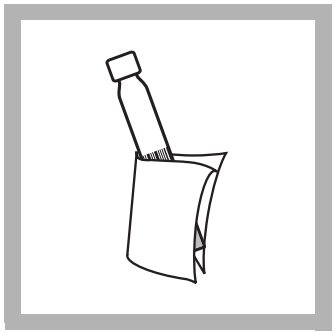


4. After inverting the tube, allow the vial to sit undisturbed for 2–3 minutes.

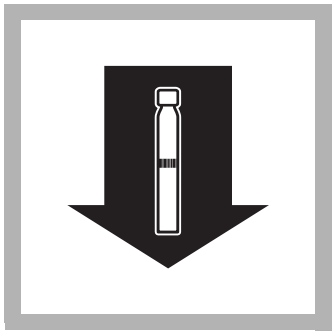
Install the Light Shield in Cell Compartment #2.



5. After the timer expires, invert the vial again 2–3 times.



6. Thoroughly clean the outside of the vial.



7. Insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L Cr.

No instrument Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 12 of the total chromium procedure or step 7 of the hexavalent chromium procedure. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. The digestion in the total chromium procedure usually destroys all color and turbidity and a sample blank is not required. To compensate for color or turbidity in the determination of hexavalent chromium, the procedure is repeated and the color forming reagent that is present in the DosiCap B is not added.

To determine the sample blank for hexavalent chromium:

1. Run the procedure as written, but do not add the DosiCap B Reagent in step 2.
2. Cap the vial with the original DosiCap ***Zip*** (do not remove the foil).
3. The value obtained in step 7 is subtracted from the value obtained on the original hexavalent chromium sample to give the corrected sample concentration.

Alternatively, hexavalent chromium samples that contain turbidity only may be filtered through a membrane filter and analyzed using the hexavalent procedure. Results are reported as dissolved hexavalent chromium. Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Larger amounts of iron, copper, and reducing and oxidizing agents give low-bias results. Lead, mercury and tin give high-bias results.

Important Note: *Undissolved chromium is not determined with the determination of chromium(VI). An analyte concentration greatly (above 20 mg/L) in excess of the stated range will adversely affect color formation, resulting in a false reading within the method range.*

Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
SO_4^{2-}, Na^+, K^+, NO_3^-	2000 mg/L
Cl^-	1000 mg/L
Ca^{2+}	125 mg/L
Mg^{2+}, NH^{4+}	100 mg/L
Zn^{2+}, Ni^{2+}, Co^{2+}, Cd^{2+}	50 mg/L
Pb^{2+}	25 mg/L
Cu^{2+}, Fe^{3+}	10 mg/L
Ag^+	5 mg/L
Sn^{2+}	1 mg/L

Sample Collection, Storage, and Preservation

Collect samples in acid-washed glass or plastic containers. To preserve samples for total chromium analysis, adjust the pH to 2 or less with nitric acid. This requires approximately 2 mL per liter of the acid. Store preserved samples at 4 °C (39.2 °F) for up to six months. Bring the sample temperature to 15–35 °C (59–95 °F) adjust the pH to about 4 with 5.0 N Sodium Hydroxide before analysis. To preserve samples for hexavalent chromium analysis, adjust the pH to 8 with 1N Sodium Hydroxide. Store at 4 °C (39.2 °F) for up to 24 hours. Bring sample to 15–35 °C (59–95 °F). No pH neutralization is required. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method (Total Chromium)
To check the accuracy of the total chromium method, prepare a 0.50-mg/L trivalent chromium standard as follows:

1. Dilute 5.00 mL of Trivalent Chromium Standard Solution, 50-mg/L as Cr^{3+}, to 500 mL with deionized water.

2. Use 2.0 mL of the trivalent chromium standard in place of the sample in step 3. Prepare this solution daily.

Standard Solution Method (Hexavalent Chromium)
To check the accuracy of the hexavalent chromium method, prepare a 0.50-mg/L hexavalent chromium standard as follows:

1. Dilute 5.00 mL of Hexavalent Chromium Standard Solution, 10 mg/L as Cr^{6+}, to 100 mL with deionized water.

2. Use 2.0 mL of this 0.50 mg/L standard in place of the sample in step 2. Prepare this solution daily.

Summary of Method

In the total chromium procedure, all chromium in the sample is oxidized to the hexavalent chromium (Cr^{6+}). The hexavalent chromium then reacts with 1,5-diphenylcarbazide to form 1,5-diphenylcarbazone. The amount of red color formed with hexavalent chromium is directly proportional to the amount of chromium present in the sample. Determine trivalent chromium by subtracting the results of a separate hexavalent chromium test from the results of the total chromium test.Test results are measured at 543 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Chromium, Total and Hexavalent TNTplus, TNT854 Reagent Set	1	25/pkg	TNT854

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 13x17 mm + 2x20 mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable volume, 1–5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	1	100/pkg	27952-00
Test Tube Cooling Rack	1–2	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
Chromium, Trivalent, Standard Solution, 50-mg/L Cr^{3+}	100 mL	14151-42
Chromium, Hexavalent Standard Solution, 10, 2 mL ampules	20/pkg	25572-20

Optional Reagents and Apparatus

Description	Cat. No.
Bottle, Sampling, low density poly, w/cap, 500 mL, 12/pkg	20870-79
DRB200 Reactor, 115 V, 21x13mm + 4x20 mm (dual block)	DRB200-02
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	DRB200-03
DRB200 Reactor, 115 V, 12x13mm + 8x20 mm (dual block)	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	DRB200-06
DRB200 Reactor, 230V, 15x13 mm + 15 x 13 mm (dual block)	DRB200-07
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	DRB200-08
Flask, filtering, glass, 1000-mL (SUVA)	546-53
Flask, volumetric, 100-mL	14574-42
Flask, volumetric, Class A, 500-mL	14574-49
Filter holder, glass for vacuum filtration (SUVA)	2340-00
Filter membrane, 47-mm, 0.45-micron hydrophilic polyethersulfone for SUVA	28947-00
Nitric Acid, ACS	152-49
pH paper, 0–14 units	26013-00
Pipette, volumetric, Class A, 5.00 mL	14515-37
Pipet, safety bulb	14561-00
Sodium Hydroxide,1.0 N	1045-32
Sodium Hydroxide, 5.0 N	2450-32
TNTplus Reactor Adapter Sleeves, 16-00 to 13-mm diameter, 5/pkg	28958-05
Tubing, rubber, 12 ft	560-19

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Updated November 2005 Edition 1

Cobalt

Method 8078 — 1-(2-Pyridylazo)-2-Naphthol (PAN) Method[1]

Powder Pillows — (0.01 to 2.00 mg/L)

Scope and Application: For water and wastewater; digestion is required for determining total recoverable cobalt; if EDTA is present, use the vigorous digestion.

[1] Adapted from Watanbe, H., *Talanta*, 21 295 (1974)

Test Preparation

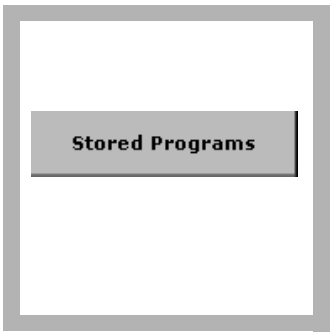
Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
If the sample is less than 10 °C (50 °F), warm to room temperature prior to analysis.
Nickel can be determined with the same sample prepared with this method. Use program Number 340. A reagent blank is necessary for the nickel procedure.

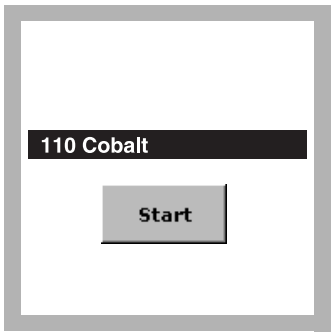
Collect the following items:	**Quantity**
Cobalt Reagent Set, 10-mL, includes:	
EDTA Reagent Powder Pillows	2
Phthalate-Phosphate Reagent Powder Pillows	2
PAN Indicator Solution	1 mL
Water, deionized	25 mL
Cylinder, graduated mixing, 25-mL	2
Sample Cells, 1-inch square glass, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 4.*

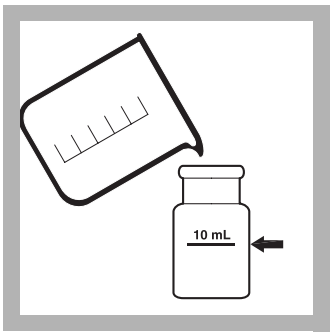
Powder Pillows — Method 8078



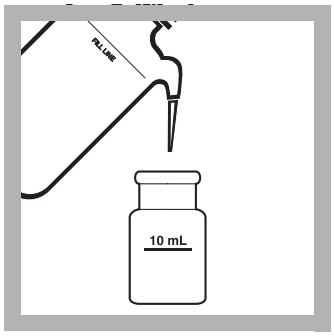
1. Press **STORED PROGRAMS**.



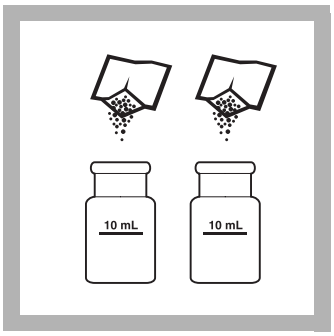
2. Select the test.



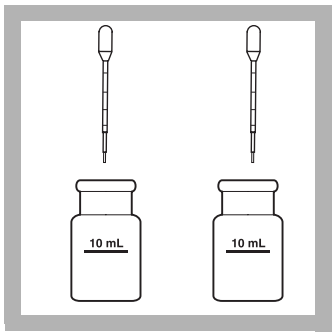
3. Prepared Sample: Fill a square sample cell to the 10-mL mark with sample.



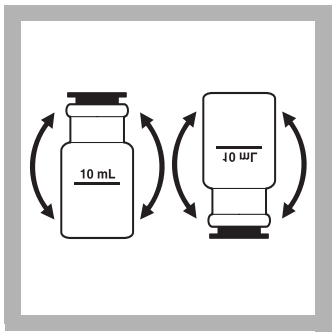
4. Blank Preparation: Fill a second square sample cell to the 10-mL mark with deionized water.



5. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell.



6. Use the plastic dropper provided to add 0.5 mL of 0.3% PAN Indicator Solution to each cell. Stopper each each cell. Invert several times to mix.

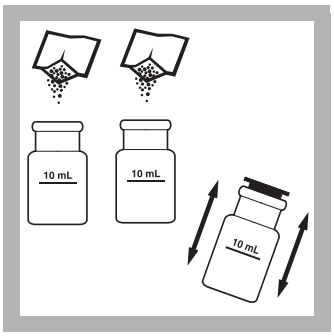


7. Stopper the cells. Invert several times to mix.

8. Press **TIMER>OK**.

A three-minute reaction period will begin.

During the reaction period, the sample solution may vary from green to dark red, depending on the chemical make-up of the sample. The deionized water blank should be yellow.

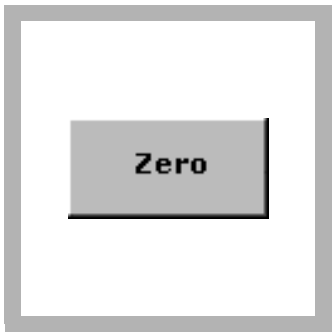


9. When the timer expires, add the contents of one EDTA Reagent Powder Pillow to each cylinder.

Stopper the cells.

Shake to dissolve.

10. Wipe the blank and insert it into the cell holder with the fill line facing right.



11. Press **ZERO**.

The display will show:

0.00 mg/L Co

12. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Co.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Al^{3+}	32 mg/L
Ca^{2+}	1000 mg/L as $CaCO_3$
Cd^{2+}	20 mg/L
Cl^{-}	8000 mg/L
Cr^{3+}	20 mg/L
Cr^{6+}	40 mg/L
Cu^{2+}	15 mg/L
F^{-}	20 mg/L
Fe^{2+}	Interferes directly and must not be present
Fe^{3+}	10 mg/L
K^{+}	500 mg/L
Mg^{2+}	400 mg/L
Mn^{2+}	25 mg/L
Mo^{6+}	60 mg/L
Na^{+}	5000 mg/L
Pb^{2+}	20 mg/L
Zn^{2+}	30 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid* (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the sample pH between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution* just before analysis. Do not exceed pH 8 as this may cause some loss of cobalt as a precipitate. Correct test results for volume additions.

Accuracy Check

Standard Solution Method
Prepare a 1.0 mg/L cobalt standard solution as follows:

1. Dilute 10.0 mL of a 10-mg/L working stock solution to 100 mL in a volumetric flask. Prepare the 10-mg/L working stock solution daily by diluting 10.00 mL of Cobalt Standard Solution, 1000-mg/L as Co, to 1000 mL with deionized water.

2. To adjust the calibration curve using the reading obtained with the 1.0-mg/L standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 4.

Summary of Method

After buffering the sample and masking any Fe^{3+} with pyrophosphate, the cobalt is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator. The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt, which can both be determined using the same sample. Test results are measured at 620 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Cobalt Reagent Set, 10-mL (100 tests), includes:	—	—	26516-00
(2) EDTA Reagent Powder Pillows	2	100/pkg	7005-99
(2) Phthalate-Phosphate Reagent Powder Pillows	2	100/pkg	26151-99
(1) PAN Indicator Solution, 0.3%	1 mL	100 mL	21502-32
Water, deionized	25 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated mixing, 25-mL	2	each	20886-40
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper, rubber	2	6/pkg	1731-06

Recommended Standards

Description	Unit	Cat. No.
Cobalt Standard Solution, 1000-mg/L Co	100 mL	21503-42

Optional Reagents and Apparatus

Description	Cat. No.
Nitric Acid, 1:1	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	2450-32

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Updated November 2005 Edition 1

Color, True and Apparent

Method 8025 **Platinum-Cobalt Standard Method[1, 2] (15 to 500 units)**

Scope and Application: For water, wastewater, and seawater; equivalent to NCASI method 253 for pulp and paper effluent using 465 nm (requires pH adjustment)

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater* and *NCASI, Technical Bulletin No. 253*, Dec. 1971

[2] Adapted from *Wat. Res.* Vol. 30, No. 11, pp. 2771–2775, 1996

Test Preparation

Before starting the test:

NCASI procedure requires pH adjustment. Adjust the pH to 7.6 with 1.0 N HCl or 1.0 N NaOH. When adjusting the pH, if overall volume change is greater than 1%, start over and use a stronger acid or base. Use Program 125 when performing the NCASI procedure.

To test for **apparent color**, omit steps 3–6 and step 8. Use unfiltered deionized water in step 7 and unfiltered sample in step 9.

Collect the following items:	Quantity
Hydrochloric Acid Solution, 1.0 N (Program 125)	varies
Sodium Hydroxide, 1.00 N (Program 125)	varies
Water, deionized	50 mL
FIlter Apparatus: membrane filter, filter holder, filter flask, and aspirator	1
Sample Cells, 1-inch square, 10 mL, matched pair	2
Stopper, rubber, one hole, No. 7	1
Tubing, rubber	1

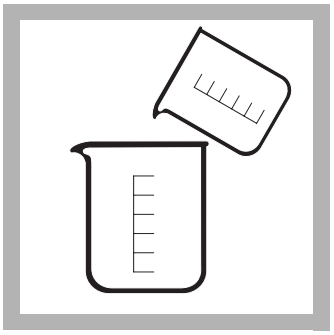
Note: *Reorder information for consumables and replacement items is on page 4.*

Platinum-Cobalt Method 8025

1. Press **STORED PROGRAMS**.

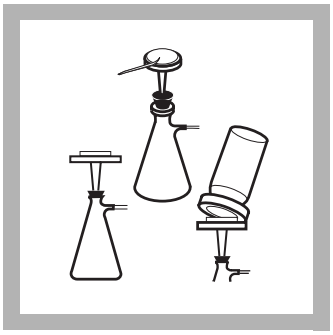
1. Select the test.

NCASI: Use Program 125 for the NCASI test.



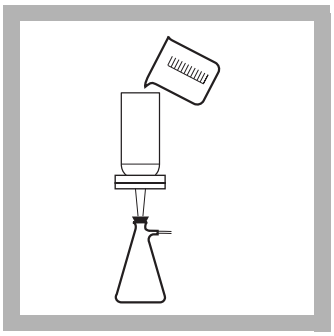
2. Collect 200 mL of sample in a 400-mL beaker.

NCASI: Adjust the pH as described in Test Preparation.

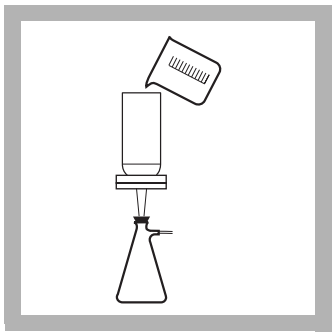


3. Assemble the filtering apparatus (0.45 micron membrane filter, filter holder, filter flask, and aspirator).

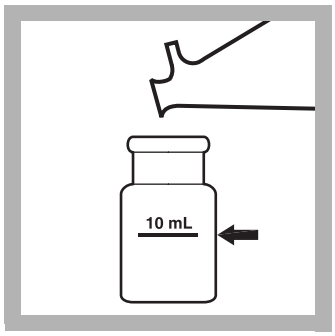
NCASI: Test prescribes a 0.8-micron filter.



4. Rinse the filter by pouring about 50 mL of deionized water through the filter. Discard the rinse water.

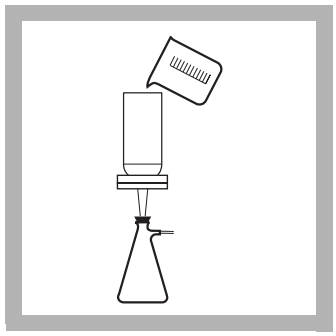


5. Pour another 50 mL of deionized water through the filter.

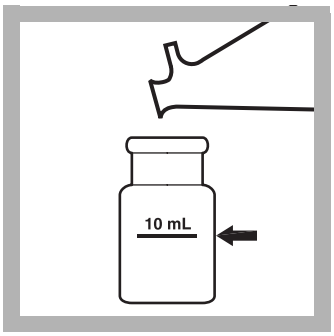


6. Blank Preparation: Fill a square sample cell with 10 mL of filtered deionized water from step 5.

Discard the excess water in the flask.

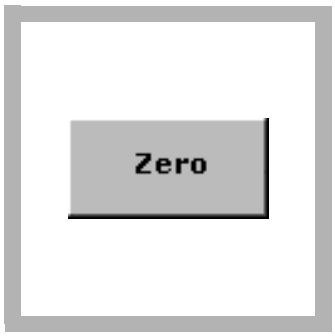


7. Pour about 50 mL of sample through the filter.



8. Prepared Sample: Fill a second square sample cell with 10 mL of filtered sample.

9. Wipe the blank and insert it into the cell holder with the fill line facing right.



10. Press **ZERO**.

The display will show:

0 units PtCo

11. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L PtCo.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, fill bottles completely and cap tightly. Avoid excessive agitation or prolonged contact with air. Samples can be stored for 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before analysis.

Accuracy Check

Standard Solution Method
Prepare a 250 platinum-cobalt units standard as follows:

1. Using Class A glassware, pipet 50.00 mL of a 500 Platinum-Cobalt Units Standard Solution into a 100-mL volumetric flask. Dilute to the 100 mL mark with deionized water.

2. To adjust the calibration curve using the reading obtained with the 250 platinum-cobalt units standard, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected units). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Color may be expressed as “apparent” or “true” color. The apparent color includes that from dissolved materials plus that from suspended matter. By filtering or centrifuging out the suspended materials, the true color can be determined. The procedure describes true color analysis. If apparent color is desired, it can be determined by measuring an unfiltered water sample. The stored program is used for both forms of color.

The stored program is calibrated in color units based on the APHA-recommended standard of 1 color unit being equal to 1 mg/L platinum as chloroplatinate ion. Test results for Programs 120 and 125 are measured at 455 and 465 nm, respectively.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Hydrochloric Acid Solution, 1.0 N	varies	1 L	23213-53
Sodium Hydroxide, 1.00 N	varies	900 mL	1045-53
Water, deionized	50 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Aspirator, Nalgene vacuum pump	1	each	2131-00
Filter, membrane, 47-mm, 0.8-microns	1	100/pkg	26408-00
Filter, membrane, 47-mm, 0.45-microns	1	100/pkg	13530-00
Flask, filtering, 500-mL	1	each	546-49
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper, rubber, one hole, No. 7	1	6/pkg	2119-07
Tubing, rubber	1 ft	12 ft	560-19

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Color Standard Solution, 500 platinum-cobalt units	1L	1414-53
Color Standard Solution, 15 platinum-cobalt units	1 L	26028-53
Color Standard Solution, 500 platinum-cobalt units, 10-mL Voluette® Ampules	16/pkg	1414-10
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, volumetric, Class A, 50.00 mL	each	14515-41
Pipet Filler, safety bulb	each	14651-00

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Updated November 2005 Edition 1

Copper

Method 8143
Powder Pillows

Porphyrin Method[1]
LR (1 to 210 µg/L)

Scope and Application: For water, wastewater, and sea water

[1] Adapted from Ishii and Koh, *Bunseki Kagaku*, 28 (473), 1979

Test Preparation

Before starting the test:

Digestion is required for determining total copper.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Wash all glassware with detergent. Rinse with tap water. Rinse again with 1:1 Nitric Acid Solution. Rinse a third time with copper-free, deionized water.
If samples contain high levels of metals, a slight metallic deposit or yellow buildup may form in the sample cell. Wash the cell as described above.

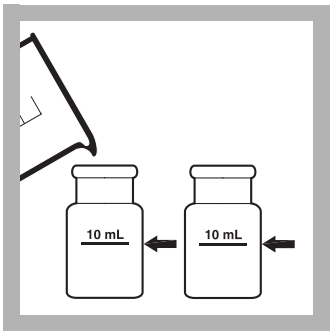
Collect the following items:	**Quantity**
Copper Masking Reagent Powder Pillows	1
Porphyrin 1 Reagent Powder Pillows	2
Porphyrin 2 Reagent Powder Pillows	2
Nitric Acid Solution, 1:1	varies
Sample Cells, 1-inch square	2

Note: *Reorder information for consumables and replacement items is on page 5.*

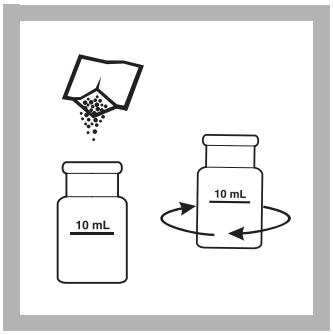
Powder Pillows — Method 8143

1. Press **STORED PROGRAMS**.

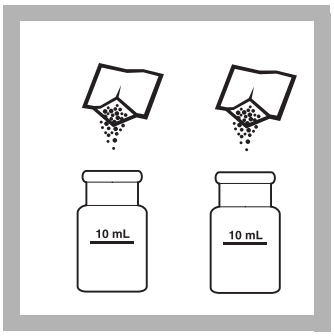
2. Select the test.



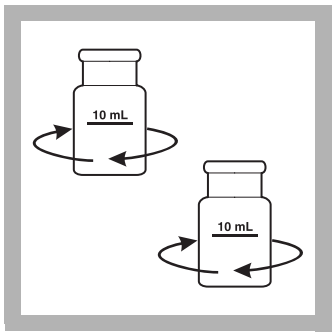
3. Fill two square sample cells with 10 mL of sample.



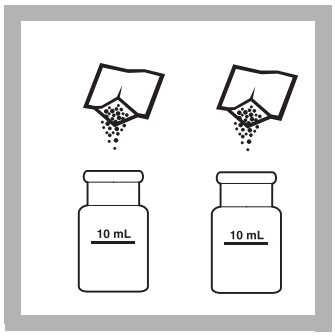
4. Blank Preparation: Add the contents of one Copper Masking Reagent Powder Pillow to one of the sample cells. Swirl to dissolve. This cell will be the blank.



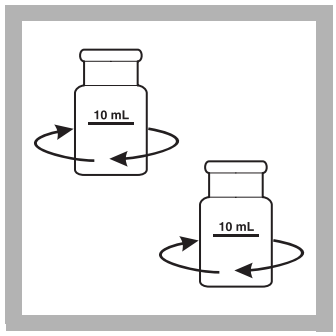
5. Add the contents of one Porphyrin 1 Reagent Powder Pillow to each sample cell.



6. Swirl to dissolve.



7. Add the contents of one Porphyrin 2 Reagent Powder Pillow to each sample cell.



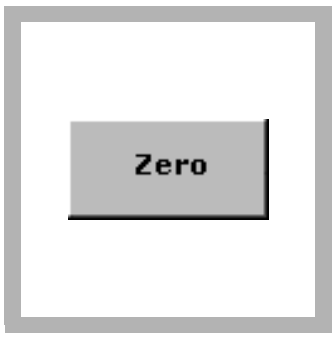
8. Swirl to dissolve.

If copper is present, the sample will momentarily turn blue, then return to yellow.

9. Press **TIMER>OK**.

A 3-minute reaction period will begin.

10. When the timer expires, insert the blank into the cell holder with the fill line facing right.



11. Press **ZERO**.

The display will show:

0 µg/L Cu

12. Insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in µg/L Cu.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum, Al^{3+}	60 mg/L
Cadmium, Cd^{2+}	10 mg/L
Calcium, Ca^{2+}	1500 mg/L
Chelating agents	Interfere at all levels unless either the Digesdahl or vigorous digestion is performed
Chloride, Cl^-	90,000 mg/L
Chromium, Cr^{6+}	110 mg/L
Cobalt, Co^{2+}	100 mg/L
Fluoride, F^-	30,000 mg/L
Iron, Fe^{2+}	6 mg/L
Lead, Pb^{2+}	3 mg/L
Magnesium	10,000 mg/L
Manganese	140 mg/L
Mercury, Hg^{2+}	3 mg/L
Molybdenum	11 mg/L
Nickel, Ni^{2+}	60 mg/L
Potassium, K^+	60,000 mg/L
Sodium, Na^+	90,000 mg/L
Zinc, Zn^{2+}	9 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed plastic bottles. To preserve, adjust the pH to 2 or less with nitric acid (about 5 mL per liter). Store preserved samples up to six months at room temperature. Before testing, adjust the pH of the preserved sample to between 2 and 6. If the sample is too acidic, adjust the pH with 5.0 N Sodium Hydroxide Standard Solution*. Correct test results for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. Prepare a 4000-µg/L copper standard by adding 4.00 mL Copper Standard Solution, 100-mg/L, to a 100-mL volumetric flask. Dilute to 100 mL with copper-free deionized water.

2. After reading test results, leave the sample cell (unspiked sample) in the instrument.

3. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

* See Optional Reagents and Apparatus on page 5.

4. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

5. Fill eight sample cells with 10 mL of sample. Use the TenSette® Pipet to add 0.1 mL of Copper Standard Solution, 4000-µg/L Cu, to two of the sample cells. Then pipet 0.2 mL of the standard solution into two more cells. Finally, pipet 0.3 mL of the standard solution into two more cells.

6. Analyze each standard addition sample as described in the procedure above, using one of the two spiked samples in each set as the blank. Accept each standard additions reading by pressing **READ**. The copper concentration reading should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Standard Solutions Method

1. To assure the accuracy of the test, prepare a 150-µg/L copper standard by pipetting 15.00 mL of Copper Standard Solution, 10.0-mg/L Cu, into a 1000-mL volumetric flask.

2. Dilute to the mark with copper-free, reagent-grade water. Prepare this solution daily. Perform the copper procedure as described above.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The porphyrin method is very sensitive to trace amounts of free copper. The method is free from most interferences and does not require any sample extraction or concentration before analysis. Interferences from other metals are eliminated by the copper masking reagent. The porphyrin indicator forms an intense, yellow-colored complex with any free copper present in sample. Test results are measured at 425 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Copper Reagent Set (100 tests), includes:	—	—	26033-00
(1) Copper Masking Reagent Powder Pillows	1	100/pkg	26034-49
(2) Porphyin 1 Reagent Powder Pillows	2	100/pkg	26035-49
(2) Porphyrin 2 Reagent Powder Pillows	2	100/pkg	26036-49
Nitric Acid Solution, 1:1	varies	500 mL	2540-49

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Copper Standard Solution, 100-mg/L Cu	100 mL	128-42
Copper Standard Solution, 10-mg/L Cu	100 mL	129-32
Water, deionized	4L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Sodium Hydroxide Standard Solution, 5.0 N	2450-32

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Updated November 2005 Edition 1

Copper

★Method 8506 and Method 8026
Powder Pillows or AccuVac® Ampuls

Bicinchoninate Method[1]
(0.04 to 5.00 mg/L)

Scope and Application: For water, wastewater and seawater[2]; Method 8506 USEPA approved for reporting wastewater analysis (digestion required)[3]

[1] Adapted from Nakano, S., *Yakugaku Zasshi*, 82 486-491 (1962) [*Chemical Abstracts,* 58 3390e (1963)]
[2] Pretreatment required; see *Interferences (Using Powder Pillows)*
[3] *Federal Register*, 45 (105) 36166 (May 29, 1980)

Test Preparation

Before starting the test:

Digestion is required for determining total copper.
Adjust the pH of acid-preserved samples to 4–6 with 8 N KOH before analysis.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

Collect the following items:	**Quantity**
Powder Pillow Test:	
CuVer® 1 Copper Reagent powder pillow	1
Sample Cells, 1-in. square, 10-mL (Powder Pillow Test)	2
AccuVac Test:	
CuVer® 2 Copper Reagent AccuVac® Ampul	1
Beaker, 50-mL (AccuVac test)	1
Sample Cell, 10-mL (AccuVac test)	1

Note: *Reorder information for consumables and replacement items is on page 6.*

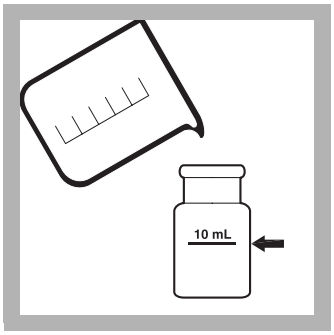
Note: *If copper is present, the sample will turn purple when it mixes with the reagent powder.*

Note: *Accuracy is not affected by undissolved powder.*

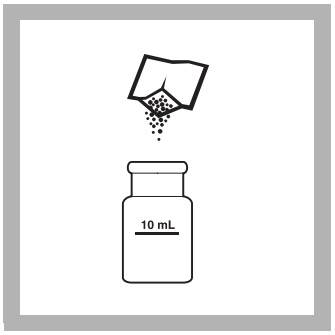
Powder Pillows — Method 8506

1. Press **STORED PROGRAMS**.

2. Select the test.



3. Prepared Sample: Fill a square sample cell with 10 mL of sample.

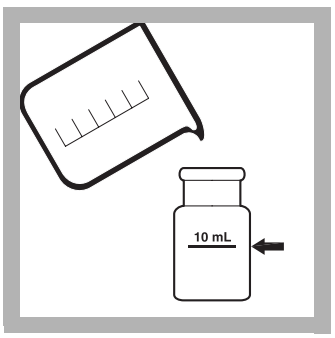


4. Add the contents of one CuVer® 1 Copper Reagent Powder Pillow to the sample cell (the prepared sample). Twirl sample cell to mix.

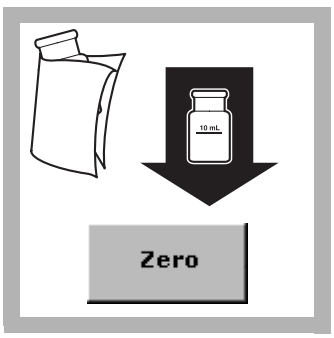
Use a CuVer 2 Copper Reagent Pillow for samples containing high levels of aluminum, iron, and hardness. A 25-mL sample cell is required. See Table 1.

5. Press **TIMER>OK**.

A 2-minute reaction period will begin.



6. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of sample.

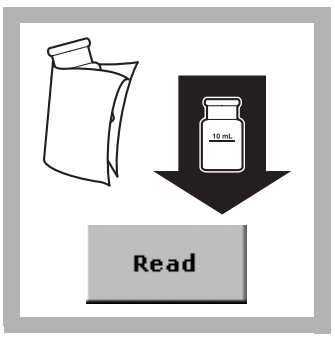


7. Insert the blank into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

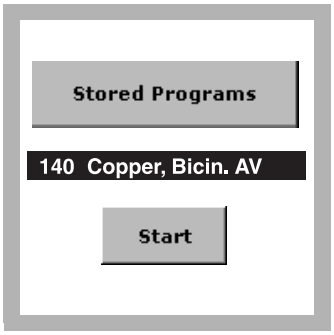
0.00 mg/L Cu



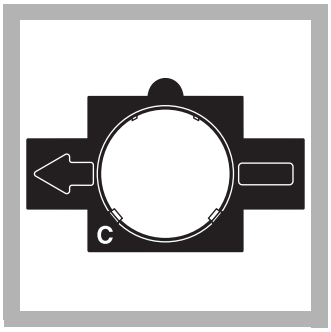
8. Within 30 minutes after the timer expires, insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Cu.

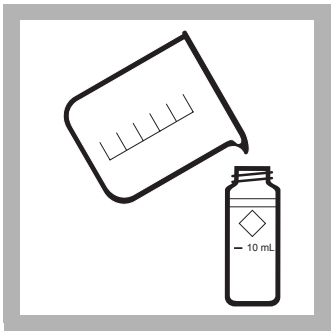
AccuVac® Ampul Method 8026



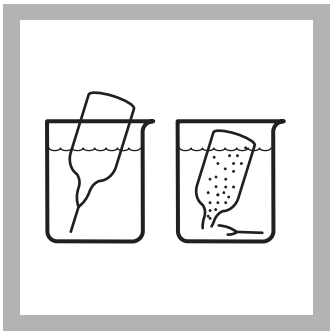
1. Select the test.



2. Insert Adapter C.

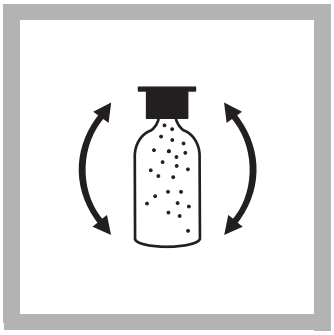


3. Blank Preparation: Fill a round sample cell with 10-mL of sample.



4. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a CuVer 2 AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.



5. Quickly invert the Ampul several times to mix. Wipe off any liquid or fingerprints with cloth or soft paper towel.

6. Press **TIMER>OK**

A 2-minute reaction period will begin.

7. When the timer expires, insert the blank into the cell holder. Close the cover.

Press **ZERO**.

The display will show:

0.00 mg/L Cu

8. Within 30 minutes after the timer expires, insert the AccuVac Ampul into the cell holder.

Press **READ**. Results are in mg/L Cu.

Interferences

Table 1 suggests treatments for powder pillows. Table 2 suggests treatments for AccuVac Ampuls. To differentiate free copper from that complexed to EDTA or other complexing agents, use a 25-mL sample cell and Free Copper Reagent Powder Pillow instead of the CuVer 1 Powder Pillow in step 4. Results in step 8 will be free copper only. Add a Hydrosulfite Reagent Powder Pillow to the same sample and re-read the result. This result will include the total dissolved copper (free and complexed). Unlike CuVer 1 Reagent, CuVer 2 Reagent Powder Pillows and AccuVac Ampuls react directly with copper, which is complexed by chelants such as EDTA.

Table 1 Interfering Substances and Suggested Treatments for Powder Pillows

Interfering Substance	Interference Levels and Treatments
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution drop-wise until sample pH is above 4. Continue with step 3.
Aluminum, Al^{3+}	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in step 4. Results obtained will include total dissolved copper (free and complexed). Requires a 25-mL sample volume.
Cyanide, CN^-	Prevents full color development. Before adding the CuVer 1 Powder Pillow Reagent, add 0.2 mL of formaldehyde to the 10-mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in step 4. Results obtained will include total dissolved copper (free and complexed). Requires a 25-mL sample volume.
Iron, Fe^{3+}	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in step 4. Results obtained will include total dissolved copper (free and complexed). Requires a 25-mL sample volume.
Silver, Ag^+	If a turbidity remains and turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

Table 2 Interfering Substances and Suggested Treatments for AccuVac® Ampuls

Interfering Substance	Interference Levels and Treatments
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution drop-wise until sample pH is above 4. Continue with step 3.
Aluminum, Al^{3+}	Reagents accommodate high levels.
Cyanide, CN^-	Prevents full color development. Add 0.5 mL of formaldehyde per 25-mL of sample before using CuVer 2 Reagent AccuVac Ampul. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Reagents accommodate high levels.
Iron, Fe^{3+}	Reagents accommodate high levels.
Silver, Ag^+	If a turbidity remains and turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

Sample Collection, Storage, and Preservation

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with concentrated nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Before analysis, adjust the pH to 4–6 with 8 N Potassium Hydroxide. Do not exceed pH 6, as copper may precipitate. Correct the test result for volume additions. If only dissolved copper is to be determined, filter the sample before acid addition.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Copper Voluette® Ampule Standard, 12.5-mg/L Cu.

5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to the unspiked sample. Press the timer icon. After the timer expires, read the result.

6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Press the timer icon. After the timer expires, read the result.

7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Press the timer icon. After the timer expires, read the result. Each addition should reflect approximately 100% recovery.

Note: *For AccuVac Ampuls, fill three mixing cylinders with 50-mL of sample and spike with 0.2 mL, 0.4 mL, and 0.6 mL of Copper Voluette Ampule Standard, 75-mg/L Cu. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solutions Method
Prepare a 4.00-mg/L Standard as follows:

1. Using Class A glassware, pipet 4.00 mL of Copper Standard Solution, 100-mg/L as Cu, into a 100-mL volumetric flask. Dilute to volume with deionized water, stopper and invert to mix. Perform the procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Copper in the sample reacts with a salt of bicinchoninic acid contained in CuVer 1 or CuVer 2 Copper Reagent to form a purple colored complex in proportion to the copper concentration. Test results are measured at 560 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
CuVer® 1 Copper Reagent Powder Pillows	1	100/pkg	21058-69
OR			
CuVer® 2 Copper Reagent AccuVac® Ampuls	1	25/pkg	25040-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Copper Standard Solution, 100-mg/L as Cu	100 mL	128-42
Copper Voluette® Ampule Standard, 12.5-mg/L as Cu	16/pkg	21126-10
Copper Voluette® Ampule Standard, 75-mg/L as Cu, 2-mL	10/pkg	14247-10
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	28337-49
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL/L	28336-49

Optional Reagents and Apparatus

Description	Cat. No.
Beakers, 50-mL	500-41H
CuVer 2 Copper Reagent Powder Pillow	21882-99
Cylinders, mixing	1896-41
Formaldehyde	2059-32
Nitric Acid, concentrated	152-49
Potassium Chloride Solution	765-42
Potassium Hydroxide Standard Solution, 8 N	282-32H
Reagent Set for Free and Total Copper, includes:	24392-00
Hydrosulfite Reagent Powder Pillows	21188-69
Free Copper Reagent Powder Pillows	21823-69
Sample Cells, 25 mL, with stoppers, 2/pkg	26126-02

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Updated November 2005 Edition 1

Cyanide

Method 8027

Pyridine-Pyrazalone Method[1]

Powder Pillows

(0.002 to 0.240 mg/L CN^-)

Scope and Application: For water, wastewater, and seawater

[1] Adapted from Epstein, Joseph, *Anal. Chem.* 19(4), 272 (1947)

Test Preparation

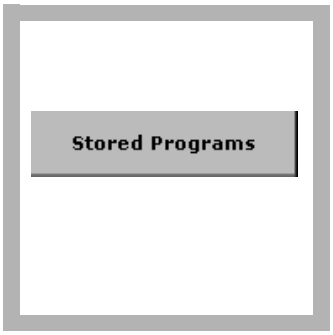
Before starting the test:

Use a water bath to maintain the optimum temperature for the reaction in this test (25 °C). Samples at less than 23 °C require longer reaction times, and samples at greater than 25 °C yield low results.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
The timing for steps 3–10 is critical. You may find it useful to open the necessary reagents before starting this sequence.
All samples to be analyzed for cyanide should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation. See Acid Distillation on page 5.
See Pollution Prevention and Waste Management on page 3 for proper disposal of solutions containing cyanide.

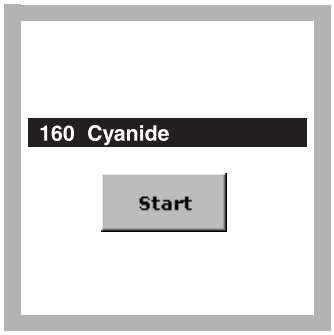
Collect the following items	Quantity
CyaniVer® Cyanide 3 Reagent Powder Pillow	1
CyaniVer® Cyanide 4 Reagent Powder Pillow	1
CyaniVer® Cyanide 5 Reagent Powder Pillow	1
Cylinder, graduated, 10-mL	1
Sample Cells, 1-inch square glass	2

Note: *Reorder information for consumables and replacement items is on page 7.*

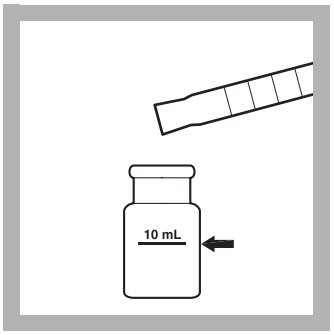
Powder Pillows — Method 8027



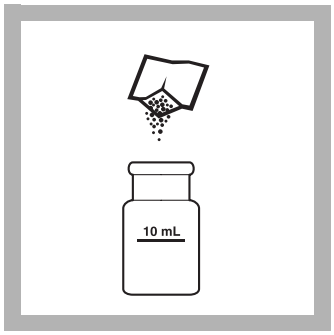
1. Press **STORED PROGRAMS**.



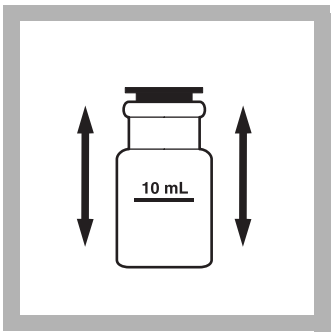
2. Select the test.



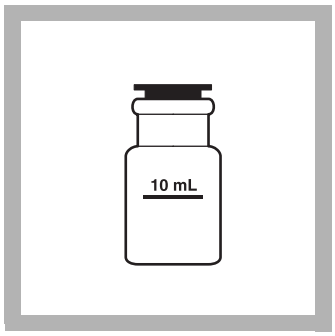
3. Using a graduated cylinder, fill a square sample cell with a 10 mL of sample.



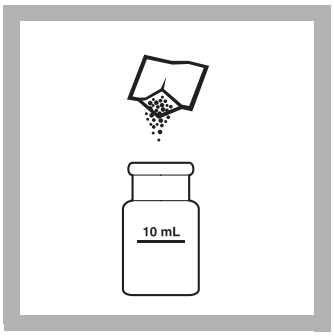
4. Prepared Sample: Add the contents of one CyaniVer 3 Cyanide Reagent Powder Pillow. Cap the cell.



5. Shake the sample cell for 30 seconds.

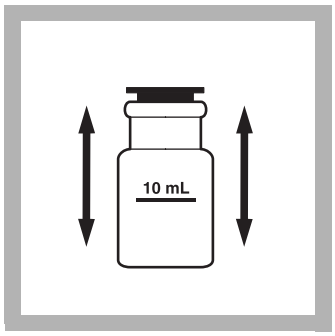


6. Leave the sample cell undisturbed for an additional 30 seconds.

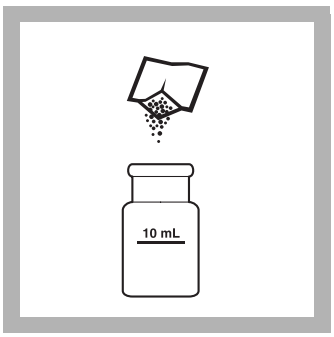


7. Add the contents of one CyaniVer 4 Cyanide Reagent Powder Pillow.

Cap the sample cell.

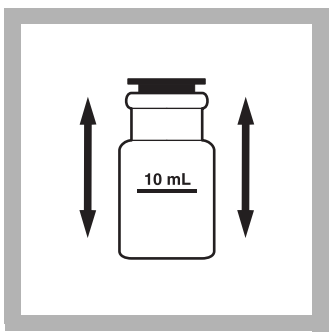


8. Shake the sample for 10 seconds. Immediately proceed to *step* 9. (Delaying the addition of the CyaniVer 5 will produce low test results.)



9. Add the contents of one CyaniVer 5 Cyanide Reagent Powder Pillow.

Cap the sample cell.



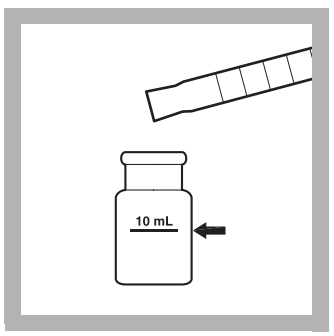
10. Shake the cell vigorously.

If cyanide is present, a pink color will develop.

11. Press **TIMER>OK**.

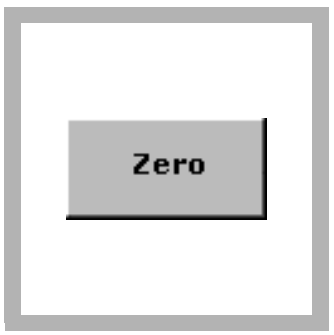
A 30-minute reaction period will begin.

The solution will turn from pink to blue.



12. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of sample.

13. Wipe the blank and insert it into the cell holder with the fill line facing right.



14. Press **ZERO**.

The display will show:

0.000 mg/L CN^-

15. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

16. Press **READ**.

Results are in mg/L CN^-.

Pollution Prevention and Waste Management

Special Considerations for Cyanide Containing Materials
Samples analyzed by this procedure may contain cyanide, which is regulated as reactive (D003) waste by the federal RCRA. It is imperative these materials be handled safely to prevent the release of hydrogen cyanide gas (an extremely toxic material with the smell of almonds). Most cyanide compounds are stable and can be safely stored for disposal in highly alkaline solutions (pH >11) such as 2 N sodium hydroxide. Never mix these wastes with other laboratory wastes which may contain lower pH materials such as acids or even water.

In the event of a spill or release, special precautions must be taken to prevent exposure to hydrogen cyanide gas. The following steps may be taken to destroy the cyanide compounds in the event of an emergency:

- Use a fume hood or supplied air or self contained breathing apparatus.
- While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).
- Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
- Neutralize and flush the solution down the drain with a large excess of water.

Note: *If the solution contains other regulated materials such as chloroform or heavy metals, it may still need to be collected for hazardous waste disposal. Never flush hazardous wastes down the drain.*

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chlorine	Large amounts of chlorine in the sample will cause a milky white precipitate after the addition of the CyaniVer® 5 Reagent. If chlorine or other oxidizing agents are known to be present, pretreat the sample before testing using the procedure in this table for oxidizing agents.
Metals	Nickel or cobalt in concentrations up to 1 mg/L do not interfere. Eliminate the interference from up to 20 mg/L copper and 5 mg/L iron by adding the contents of one HexaVer Chelating Reagent Powder Pillow[1] to the sample and then mixing before adding the CyaniVer 3 Cyanide Reagent Powder Pillow in step 4. Prepare a reagent blank of deionized water and reagents to zero the instrument in step 14.
Oxidizing Agents	**1.** Adjust a 25-mL portion of the alkaline sample to pH 7–9 with 2.5 N Hydrochloric Acid Standard Solution[1]. Count the number of drops of acid added. **2.** Add two drops of Potassium Iodide Solution[1] and two drops of Starch Indicator Solution[1] to the sample. Swirl to mix. The sample will turn blue if oxidizing agents are present. **3.** Add Sodium Arsenite Solution[1] drop-wise until the sample turns colorless. Swirl the sample thoroughly after each drop. Count the number of drops. **4.** Take another 25-mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in step a. **5.** Subtract one drop from the amount of Sodium Arsenite Solution added in step c. Add this amount to the sample and mix thoroughly. Continue with step 3 of the cyanide procedure.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Reducing Agents	**1.** Adjust a 25-mL portion of the alkaline sample to pH 7–9 with 2.5 N Hydrochloric Acid Standard Solution[1]. Count the number of drops added. **2.** Add four drops of Potassium Iodide Solution[1] (Cat. No. 343-32) and four drops of Starch Indicator Solution to the sample. Swirl to mix. The sample should be colorless. **3.** Add Bromine Water[1] drop-wise until a blue color appears. Swirl the sample thoroughly after each addition. Count the number of drops. **4.** Take another 25-mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in step a. **5.** Add the total number of drops of Bromine Water counted in *step c* to the sample and mix thoroughly. **6.** Continue with step 3 of the cyanide procedure.
Turbidity	Large amounts of turbidity will cause high readings. Use filter paper[1] and a funnel[1] to filter highly turbid water samples before use in steps 3 and 12. The test results should then be recorded as soluble cyanide.

[1] See Optional Reagents and Apparatus on page 8.

Sample Collection, Storage, and Preservation

Collect samples in glass or plastic bottles and analyze as quickly as possible.

The presence of oxidizing agents, sulfides and fatty acids can cause the loss of cyanide during sample storage. Samples containing these substances must be pretreated as described below before preservation with sodium hydroxide. If the sample contains sulfide and is not pretreated, it must be analyzed within 24 hours.

Preserve the sample by adding 4.0 mL of 5.0 N Sodium Hydroxide Standard Solution* to each liter (or quart) of sample, using a glass serological pipet and pipet filler. Check the sample pH; 4-mL of sodium hydroxide is usually enough to raise the pH of most water and wastewater samples to 12. Add more 5.0 N Sodium Hydroxide if necessary. Store the samples at 4 °C (39 °F) or less. Samples preserved in this manner can be stored for 14 days.

Before testing, samples preserved with 5.0 N Sodium Hydroxide or samples that are highly alkaline due to chlorination treatment processes or sample distillation procedures should be adjusted to approximately pH 7 with 2.5 N Hydrochloric Acid Standard Solution. Where significant amounts of preservative are used, a volume correction should be made.

Oxidizing Agents
Oxidizing agents such as chlorine decompose cyanides during storage. To test for their presence and to eliminate their effect, pretreat the sample as follows:

1. Take a 25-mL portion of the sample and add one drop of 10-g/L m-Nitrophenol Indicator Solution*. Swirl to mix.
2. Add 2.5 N Hydrochloric Acid Standard Solution drop-wise until the color changes from yellow to colorless. Swirl the sample thoroughly after the addition of each drop.
3. Add two drops of Potassium Iodide Solution*, 30-g/L, and two drops of Starch Indicator Solution, to the sample. Swirl to mix. The solution will turn blue if oxidizing agents are present.

* See Optional Reagents and Apparatus on page 8.

4. If step 3 suggests the presence of oxidizing agents, add two level, 1-g measuring spoonfuls of Ascorbic Acid* per liter of sample.
5. Withdraw a 25-mL portion of sample treated with ascorbic acid and repeat *steps 1* to *3*. If the sample turns blue, repeat *steps 4* and *5*.
6. If the 25-mL sample remains colorless, preserve the remaining sample to pH 12 for storage with 5 N Sodium Hydroxide Standard Solution* (usually 4-mg/L).
7. Perform the procedure given under Interfering Substances and Levels, Reducing Agents, to eliminate the effect of excess ascorbic acid, before following the cyanide procedure.

Sulfides
Sulfides will quickly convert cyanide to thiocyanate (SCN^-). To test for the presence of sulfide and eliminate its effect, pretreat the sample as follows:

1. Place a drop of sample on a disc of Hydrogen Sulfide Test Paper* that has been wetted with pH 4 Buffer Solution*.
2. If the test paper darkens, add a 1-g measuring spoon of Lead Acetate to the sample. Repeat *step a*.
3. If the test paper continues to turn dark, keep adding Lead Acetate* until the sample tests negative for sulfide.
4. Filter the lead sulfide precipitate through Filter Paper* and a Funnel*. Preserve the sample for storage with 5 N Sodium Hydroxide Standard Solution* or neutralize to a pH of 7 for analysis.

Fatty Acids
Caution
Perform this operation under a ventilation hood and as quickly as possible.

When distilled, fatty acids will pass over with cyanide and, under the alkaline conditions of the absorber, will form soaps. If the presence of fatty acid is suspected, use the following pretreatment before preserving samples with sodium hydroxide.

1. Acidify 500 mL of sample to pH 6 or 7 with a 4:1 dilution of glacial Acetic Acid*.
2. Pour the sample into a 1000-mL separation funnel and add 50 mL of Hexane*.
3. Stopper the funnel and shake for one minute. Allow the layers to separate.
4. Drain off the lower, sample layer into a 600-mL beaker. If the sample is to be stored, add enough 5 N Sodium Hydroxide Standard Solution* to raise the pH above 12.

Acid Distillation

All samples to be analyzed for cyanide should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation. With most compounds, a one-hour reflux is adequate.

If thiocyanate is present in the original sample, a distillation step is absolutely necessary as thiocyanate causes a positive interference. High concentrations of thiocyanate can yield a substantial quantity of sulfide in the distillate. The “rotten egg” smell of hydrogen sulfide will accompany the distillate when sulfide is present. The sulfide must be removed from the distillate prior to testing.

* See Optional Reagents and Apparatus on page 8.

If cyanide is not present, the amount of thiocyanate can be determined. The sample is not distilled and the final reading is multiplied by 2.2. The result is mg/L SCN^-.

The distillate can be tested and treated for sulfide after the last step of the distillation procedure by using the following lead acetate treatment procedure.

1. Place a drop of the distillate (already diluted to 250 mL) on a disc of Hydrogen Sulfide Test Paper* that has been wetted with pH 4.0 Buffer Solution*.
2. If the test paper darkens, add 2.5 N Hydrochloric Acid Standard Solution* drop-wise to the distillate until a neutral pH is obtained.
3. Add a 1-g measuring spoon of lead acetate* to the distillate and mix. Repeat step 1.
4. If the test paper continues to turn dark, keep adding lead acetate until the distillate tests negative for sulfide.
5. Filter the black lead sulfide precipitate through filter paper* and a funnel*. Neutralize the liquid filtrate to pH 7 and immediately analyze for cyanide.

Distillation Procedure
The following steps describe the distillation process using distillation apparatus* and cyanide glassware* offered by the manufacturer:

1. Set up the distillation apparatus for cyanide recovery, leaving off the thistle tube. Refer to the *Distillation Apparatus Manual*. Turn on the water and make certain it is flowing steadily through the condenser.
2. Fill the distillation apparatus cylinder to the 50-mL mark with 0.25 N Sodium Hydroxide Standard Solution*.
3. Fill a clean 250-mL graduated cylinder to the 250-mL mark with sample and pour it into the distillation flask. Place a stirring bar into the flask and attach the thistle tube.
4. Arrange the vacuum system as shown in the Distillation Apparatus Manual, but do not connect the vacuum tubing to the gas bubbler. Turn on the water to the aspirator to full flow and adjust the flow meter to 0.5 SCFH.
5. Connect the vacuum tubing to the gas bubbler, making certain that air flow is maintained (check the flow meter) and that air is bubbling from the thistle tube and the gas bubbler.
6. Turn the power switch on and set the stir control to 5. Using a 50-mL graduated cylinder, pour 50 mL of 19.2 N Sulfuric Acid Standard Solution* through the thistle tube and into the distillation flask.
7. Using a water bottle, rinse the thistle tube with a small amount of deionized water.
8. Allow the solution to mix for three minutes; then add 20 mL Magnesium Chloride Reagent* through the thistle tube and rinse again. Allow the solution to mix for 3 more minutes.
9. Verify that there is a constant flow of water through the condenser.
10. Turn the heat control to 10.
11. Carefully monitor the distillation flask at this point in the procedure. Once the sample begins to boil, slowly lower the air flow to 0.3 SCFH. If the contents of the distillation flask begin to back up through the thistle tube, increase the air flow by adjusting the flow meter until the contents do not back up through the thistle tube. Boil the sample for one hour.
12. After one hour, turn off the still, but maintain the air flow for 15 minutes more.

* See Optional Reagents and Apparatus on page 8.

13. After 15 minutes, remove the rubber stopper on the 500-mL vacuum flask to break the vacuum and turn off the water to the aspirator. Turn off the water to the condenser.

14. Remove the gas bubbler/cylinder assembly from the distillation apparatus. Separate the gas bubbler from the cylinder and pour the contents of the cylinder into a 250-mL, Class A volumetric flask. Rinse the gas bubbler, cylinder and J-tube connector with deionized water and add the washings to the volumetric flask.

15. Fill the flask to the mark with deionized water and mix thoroughly. Neutralize the contents of the flask and analyze for cyanide.

Accuracy Check

Standard Solutions Method
CAUTION
Cyanides and their solutions, and the hydrogen cyanide liberated by acids, are very poisonous. Both the solutions and the gas can be absorbed through the skin.

Prepare a 100 mg/L cyanide stock solution weekly as follows:

1. Dissolve 0.2503 grams of potassium cyanide in deionized water and dilute to 1000 mL.

2. Immediately before use, prepare a 0.200 mg/L cyanide working solution by diluting 2.00 mL of the 100 mg/L stock solution to 1000 mL using deionized water.

3. To adjust the calibration curve using the reading obtained with the 0.200 mg/L standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected units). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The Pyridine-Pyrazalone method used for measuring cyanide gives an intense blue color with free cyanide. A sample distillation is required to determine cyanide from transition and heavy metal cyanide complexes. Test results are measured at 612 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Cyanide Reagent Set, includes:	—	—	24302-00
(1) CyaniVer® 3 Cyanide Reagent Powder Pillow	1	100/pkg	21068-69
(1) CyaniVer® 4 Cyanide Reagent Powder Pillow	1	100/pkg	21069-69
(1) CyaniVer® 5 Cyanide Reagent Powder Pillow	1	100/pkg	21070-69

Consumables and Replacement Items, continued

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 10-mL	1	each	508-38
Sample Cells, 1-inch square, 10-mL, matched pair	2	2/pkg	24954-02
Stopper, poly, hollow	—	6/pkg	14480-00

Recommended Standards

Description	Unit	Cat. No.
Potassium Cyanide, ACS	125 g	767-14
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Acetic Acid	100-49
Ascorbic Acid	6138-26
Balance, analytical	28014-01
Bromine Water	2211-20
Buffer Solution, pH 4	12223-49
Filter Paper	1894-57
Funnel	1083-67
Hexane Solution	14478-49
HexaVer Chelating Reagent Powder Pillow	243-99
Hydrochloric Acid Standard Solution, 2.5 N	1418-32
Hydrogen Sulfide Test Paper	25377-33
m-Nitrophenol Indicator Solution, 10-g/L	2476-32
Magnesium Chloride Reagent	14762-53
Potassium Iodide Solution, 30-g/L	343-32
Sodium Arsenite Solution, 5-g/L	1047-32
Sodium Hydroxide Standard Solution, 0.25 N	14763-53
Sodium Hydroxide Standard Solution, 5.0 N	2450-53
Starch Indicator Solution	349-32
Sulfuric Acid Standard Solution, 19.2 N	2038-49
Cyanide Glassware	22658-00
Distillation Apparatus, 115 VAC	22744-00
Distillation Apparatus, 230 VAC	22744-02
Distillation Apparatus Set	22653-00

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Updated November 2005 Edition 1

Cyanuric Acid

Method 8139
Powder Pillows

Turbidimetric Method
(5 to 50 mg/L)

Scope and Application: For water

Test Preparation

Before starting the test:

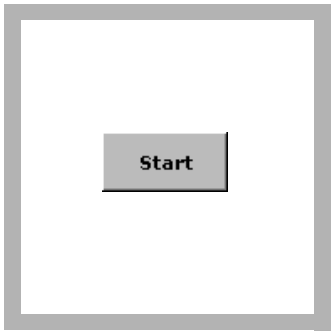
Filter highly turbid samples with filter paper and a funnel.
Clean sample cells with soap, water, and a brush soon after each test to avoid a build-up of film on the sample cell.

Collect the following items:	**Quantity**
Bottle, mixing, square, glass	1
Cyanuric Acid 2 Reagent Powder Pillow	1
Sample cells, 1-inch square, 10 mL	2

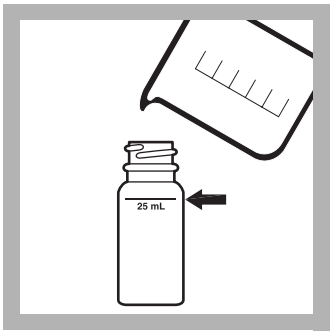
Note: *Reorder information for consumables and replacement items is on page 3.*

Powder Pillows Method 8139

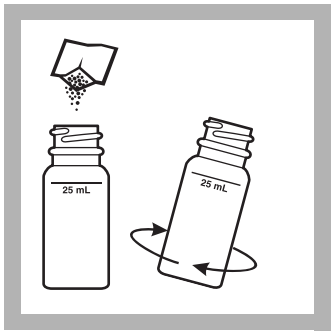
1. Press **STORED PROGRAMS**.



2. Select the test.



3. Fill a mixing bottle with 25 mL of sample.

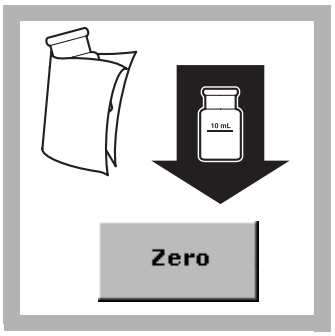


4. Prepared Sample: Add the contents of one Cyanuric Acid 2 Reagent Powder Pillow. Swirl to mix.

After adding the reagent, a white turbidity will form if cyanuric acid is present.

5. Press **TIMER>OK**.

A 3-minute reaction period will begin.

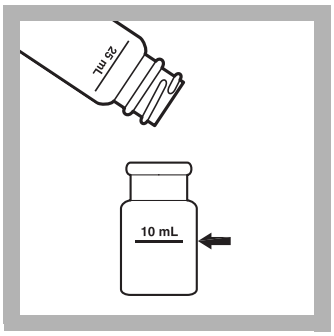


6. Blank Preparation: Fill a square sample cell with 10 mL of sample and insert it in the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

0 mg/L Cyan Acid



7. When the timer expires, fill a second square sample cell with 10 mL of prepared sample.

8. Within seven minutes after the timer expires, place the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Cyan Acid.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Samples must be analyzed within 24 hours.

Accuracy Check

Standard Solution Method

1. Dissolve 1.000 gram of cyanuric acid in l liter of deionized water to make a 1000-mg/L solution. Cyanuric acid is difficult to dissolve; it may take several hours to completely dissolve. This solution is stable for several weeks.

2. Dilute 3.00 mL of the 1000-mg/L solution to 100 mL with deionized water to make a 30-mg/L solution. Prepare fresh daily. Testing the 30-mg/L solution should give test results of about 30 mg/L cyanuric acid.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The test for Cyanuric Acid uses the turbidimetric method. Cyanuric Acid 2 Reagent precipitates any Cyanuric Acid present and holds it in suspension. The amount of turbidity caused by the suspended particles is directly proportional to the amount of cyanuric acid present. Test results are measured at 480 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Cyanuric Acid 2 Reagent Powder Pillow	1	50/pkg	2460-66

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Bottle, mixing, square, with 25 mL mark	1	each	17042-00
Sample Cell, 1-inch square, 10-mL matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Cyanuric Acid	25 g	7179-24
Water, deionized	4 L	272-56

Optional Reagents

Description	Cat. No.
Filter, paper for funnel	1894-57
Funnel	1083-67

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Updated November 2005 Edition 1

Fluoride

★Method 8029 — SPADNS Method[1]

Reagent Solution or AccuVac® Ampuls — (0.02 to 2.00 mg/L F⁻)

Scope and Application: For water, wastewater and seawater; USEPA accepted for reporting for drinking and wastewater analyses (distillation required; see *Distillation on page 4*)[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater, 4500-F B & D*

[2] Procedure is equivalent to USEPA method 340.1 for drinking water and wastewater.

Test Preparation

Before starting the test:

The sample and deionized water should be at the same temperature (±1 °C). Temperature adjustments may be made before or after reagent addition.
SPADNS Reagent is toxic and corrosive. Use care while handling the reagent.
For best results, measure the volume of SPADNS Reagent as accurately as possible.
If the instrument displays Over Measure Range!, dilute a fresh sample with an equal volume of deionized water and repeat the test, using this solution in step 3. Multiply the result by 2.

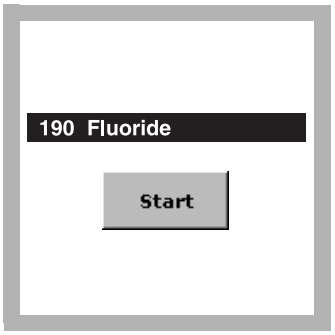
Collect the following items:	**Quantity**
Solution Test:	
SPADNS Reagent Solution	4 mL
Deionized Water	10 mL
Pipet, volumetric, 2-mL	1
Pipet, volumetric, 10-mL	1
Pipet Filler Bulb	1
Sample cells, 1-in. square, 10-mL	2
Thermometer, –10 to 110 °C	1
AccuVac Test:	
SPADNS Fluoride Reagent AccuVac® Ampuls	2
Deionized Water	40 mL
Beaker, 50-mL	1

Note: *Reorder information for consumables and replacement items is on page 5.*

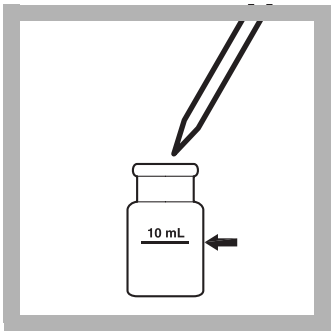
Important Note: *SPADNS Reagent contains sodium arsenite. Final solutions will contain arsenic (D004) in sufficient concentration to be regulated as a hazardous waste for Federal RCRA. Refer to the MSDS for disposal instructions.*

Using SPADNS Reagent Method 8029

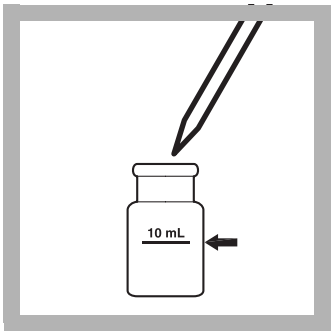
1. Press **STORED PROGRAMS**.



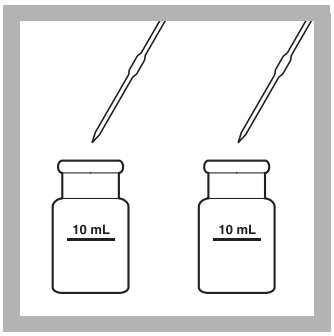
2. Select the test.



3. Prepared Sample: Pipet 10.0 mL of sample into a dry square sample cell.



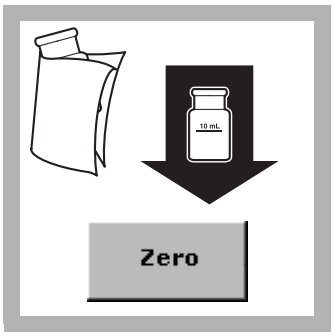
4. Blank Preparation: Pipet 10.0 mL of deionized water into a second dry square sample cell.



5. Carefully pipet 2.0 mL of SPADNS Reagent into each cell. Swirl to mix.

6. Press **TIMER>OK**.

A one-minute reaction period will begin.

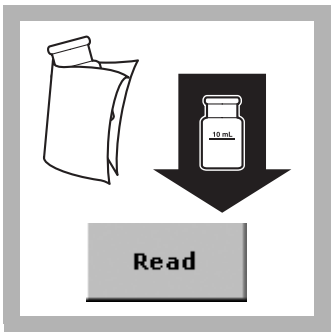


7. When the timer expires, insert the blank into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

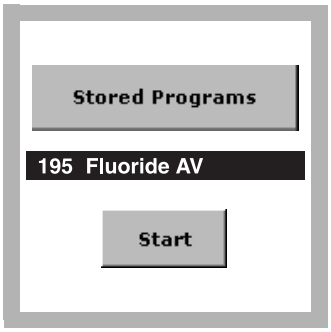
0.00 mg/L F⁻



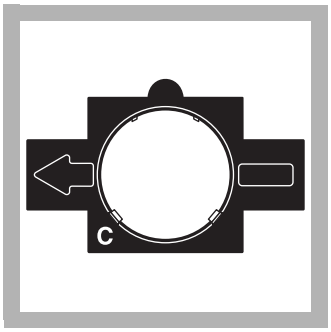
8. Insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L F⁻.

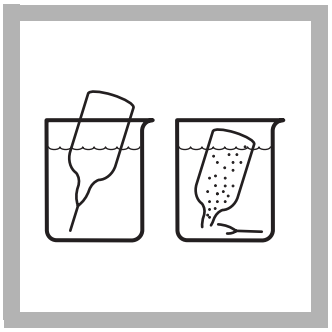
AccuVac® Ampul — Method 8029



1. Select the test.

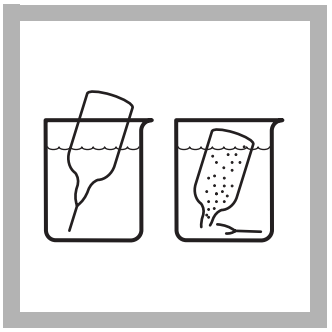


2. Insert Adapter C.



3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill one SPADNS Fluoride Reagent AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.



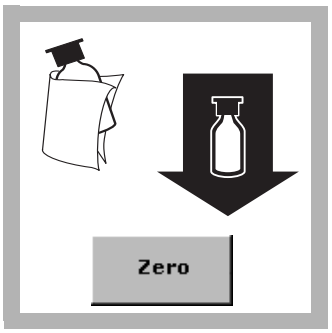
4. Blank Preparation: Pour at least 40 mL of deionized water into a second beaker.

Fill a second Ampul with deionized water. Keep the tip immersed while the Ampul fills completely.

5. Quickly invert both Ampuls several times to mix.

6. Press **TIMER>OK**.

A one-minute reaction period will begin.



7. When the timer expires, insert the blank into the cell holder. Press **ZERO**.

The display will show:

0.00 mg/L F⁻

8. Insert the AccuVac Ampul that contains the sample into the cell holder.

Press **READ**. Results are in mg/L F⁻.

Interferences

This test is sensitive to small amounts of interference. Glassware must be very clean (acid rinse before each use). Repeat the test with the same glassware to ensure that results are accurate.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Alkalinity (as $CaCO_3$)	At 5000 mg/L it causes a –0.1 mg/L F^- error.
Aluminum	At 0.1 mg/L it causes a –0.1 mg/L F^- error. To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting 2 hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.
Chloride	At 7000 mg/L it causes a +0.1 mg/L F^- error.
Chlorine	SPADNS Reagent contains enough arsenite to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, add one drop of Sodium Arsenite Solution[1] to 25 mL of sample for each 2 mg/L of Chlorine.
Iron, ferric	At 10 mg/L it causes a –0.1 mg/L F^- error.
Phosphate, ortho	At 16 mg/L it causes a +0.1 mg/L F^- error.
Sodium Hexametaphosphate	At 1.0 mg/L it causes a +0.1 mg/L F^- error.
Sulfate	At 200 mg/L it causes a +0.1 mg/L F^- error.

[1] See Optional Reagents and Apparatus on page 6.

Distillation

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

1. Set up the distillation apparatus for general purpose distillation. Refer to the Distillation Apparatus manual for proper assembly. Use a 125-mL Erlenmeyer flask to collect the distillate.
2. Turn on the water and maintain a steady flow through the condenser.
3. Measure 100 mL of sample into the distillation flask using a 100-mL graduated cylinder. Add a magnetic stir bar and 5 glass beads.
4. Turn the stirrer power switch on. Turn the stir control to 5.
5. Using a 250-mL graduated cylinder, carefully add 150 mL of StillVer® Distillation Solution into the flask. (StillVer Distillation Solution is a 2:1 mixture of concentrated sulfuric acid and water.)

Note: *When distilling samples with high amounts of chloride, add 5 mg of Silver Sulfate* to the sample for every mg/L of chloride in the sample.*

6. With the thermometer in place, turn the heat control to 10. The yellow pilot lamp indicates the heater is on.
7. When the temperature reaches 180 °C or when 100 mL of distillate has been collected, turn the still off (requires about 1 hour).
8. Dilute the distillate to a volume of 100 mL, if necessary. The distillate may now be analyzed by the SPADNS or the fluoride ion-selective electrode method.

* See Optional Reagents and Apparatus on page 6.

Sample Collection, Storage, and Preservation

Samples may be stored in glass or plastic bottles for at least seven days when cooled to 4 °C (39 °F) or lower. Warm samples to room temperature before analysis.

Accuracy Check

Standard Solution Method
A variety of standard solutions covering the entire range of the test is available. Use these instead of sample to verify technique.

Minor variations between lots of reagent become measurable above 1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

To adjust the calibration curve using the reading obtained with a standard solution:

1. Press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST>OFF**.

2. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected units). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The SPADNS Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. This method is accepted by the EPA for NPDES and NPDWR reporting purposes when the samples have been distilled. Seawater and wastewater samples require distillation. Test results are measured at 580 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
SPADNS Reagent Solution	4 mL	500 mL	444-49
OR			
SPADNS Fluoride Reagent AccuVac® Ampuls	2	25/pkg	25060-25
Water, deionized	10 mL	4 L	272-56

Required Apparatus (Solution)

Description	Quantity/Test	Unit	Cat. No.
Pipet Filler, safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 2.00-mL	1	each	14515-36
Pipet, volumetric, Class A, 10.00-mL.	1	each	14515-38
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Thermometer, –10 to 110 °C	1	each	1877-01

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H

Recommended Standards

Description	Unit	Cat. No.
Fluoride Standard Solution, 0.2-mg/L F^-	500 mL	405-02
Fluoride Standard Solution, 0.5-mg/L F^-	500 mL	405-05
Fluoride Standard Solution, 0.8-mg/L F^-	500 mL	405-08
Fluoride Standard Solution, 1.0-mg/L F^-	1000 mL	291-53
Fluoride Standard Solution, 1.0-mg/L F^-	500 mL	291-49
Fluoride Standard Solution, 1.2-mg/L F^-	500 mL	405-12
Fluoride Standard Solution, 1.5-mg/L F^-	500 mL	405-15
Fluoride Standard Solution, 2.0-mg/L F^-	500 mL	405-20
Fluoride Standard Solution, 100-mg/L F^-	500 mL	232-49
Standard, Drinking Water, Mixed Parameter, Inorganic for F^-, NO_3, PO_4, SO_4	500 mL	28330-49

Consumables and Replacement Items (continued)

Distillation Reagents and Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 100-mL	1	each	508-42
Cylinder, graduated, 250-mL	1	each	508-46
Distillation Heater and Support Apparatus Set,115 VAC, 50/60 Hz	1	each	22744-00
AND			
Distillation Heater and Support Apparatus Set,230 VAC, 50/60 Hz	1	each	22744-02
OR			
Distillation Apparatus Set, General Purpose	1	each	22653-00
Flask, Erlenmeyer, 125-mL	1	each	20897-43
Glass Beads	1	100/pkg	2596-00
StillVer® Distillation Solution	varies	500 mL	446-49
Stir Bar, magnetic	1	each	10764-16

Optional Reagents and Apparatus

Description	Cat. No.
Silver Sulfate	334-14
Sodium Arsenite Solution, 0.5 g/L	1047-32

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Updated November 2005 Edition 1

Formaldehyde

Method 8110

MBTH Method[1]

Powder Pillows

(3 to 500 µg/L)

Scope and Application: For water

[1] Adapted from Matthews, T.G. and Howell, T.C., *Journal of the Air Pollution Control Association*, 31 (11) 1181-1184 (1981).

Test Preparation

Before starting the test:

Analyze samples immediately. Do not preserve for later analysis.
Wash glassware with Chromic Acid Cleaning Solution[1] to remove trace contaminants.
Time and temperature are very important in this test. The sample should be 25 ±1°C, and the times specified in steps must be followed precisely. A temperature-controlled water bath is recommended for best accuracy
Obtain formaldehyde-free water by distilling water from alkaline permanganate (4 g Sodium Hydroxide[1], 2 g Potassium Permanganate[1] per 500 mL of water). Discard the first 50–100 mL of distillate

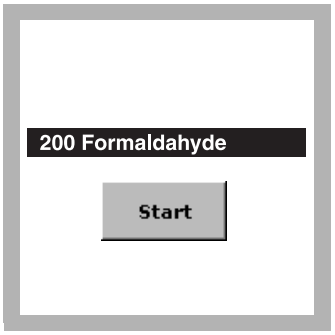
[1] See Optional Reagents and Apparatus on page 5.

Collect the following items:	Quantity
Developing Solution for LR Formaldehyde	5 mL
MBTH Powder Pillows	2
Clippers	1
Cylinder, graduated mixing, 50-mL	2
Pipet, serological, 5-mL	1
Pipet Filler	1
Sample Cells, 1-inch square glass, 10-mL	2

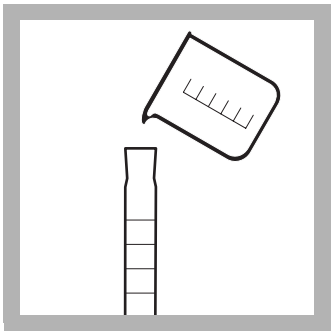
Note: *Reorder information for consumables and replacement items is on page 5.*

Powder Pillows — Method 8110

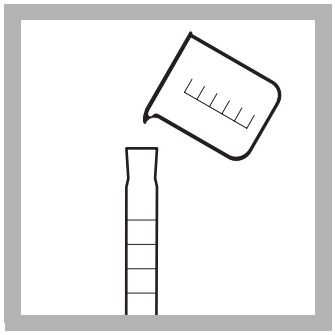
1. Press **STORED PROGRAMS**.



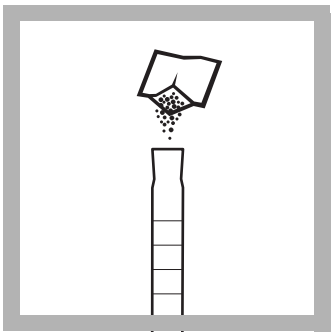
2. Select the test.



3. Prepared Sample: Accurately measure 25 mL of sample in a 50-mL mixing cylinder.



4. Blank Preparation: Accurately measure 25 mL of formaldehyde-free water in a second 50-mL mixing cylinder.

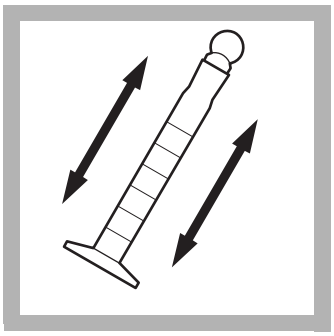


5. Add the contents of one MBTH Powder Pillow to the blank. Stopper the cylinder.

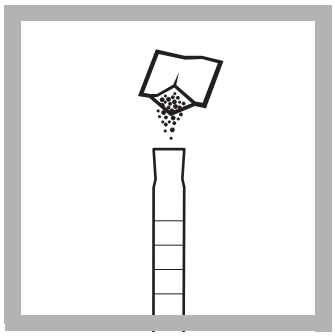
6. Press **TIMER>OK**.

A 17-minute reaction period will begin. Proceed with step 7 immediately after the timer starts.

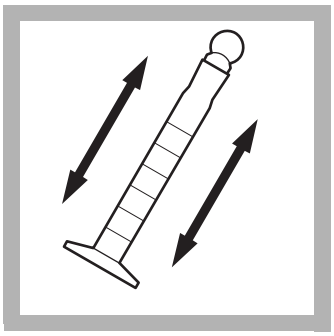
Complete steps 7–12 during the reaction period, at the times specified.



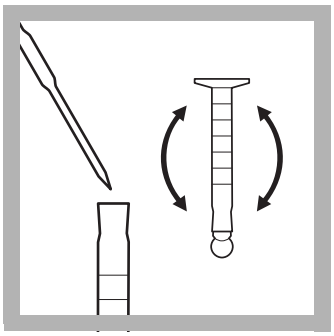
7. Immediately after the reaction period starts, shake the blank sample cylinder vigorously for 20 seconds. **Do not wait for the timer to expire.**



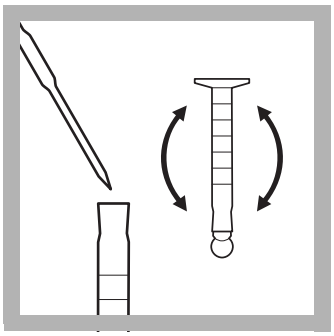
8. Add the contents of one MBTH Powder Pillow to the prepared sample when the timer displays **15:00**.



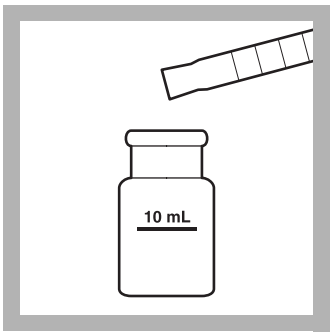
9. Stopper the cylinder and shake vigorously for 20 seconds.



10. Add 2.5 mL of Developing Solution for Low Range Formaldehyde to the blank when the timer shows **12:00**. Stopper and invert to mix.

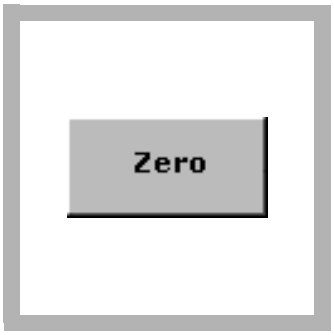


11. Add 2.5 mL of Developing Solution for Low Range Formaldehyde to the prepared sample when the timer shows **10:00**. Stopper and invert to mix.



12. Just before the timer shows **2:00**, pour at least 10 mL of the blank into the square sample cell. Pour the solution slowly to avoid bubble formation on the cell walls. If bubbles form, swirl to dislodge them.

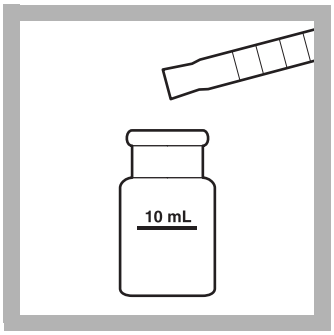
13. Immediately wipe the blank and insert it into the cell holder with the fill line facing right.



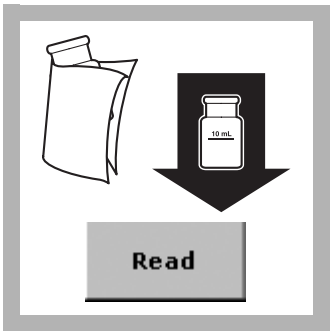
14. When the timer shows **2:00**, press **ZERO**.

The display will show:

0 µg/L CH_2O



15. Pour at least 10 mL of the prepared sample into a sample cell.



16. Wipe the cell and insert it into the cell holder with the fill line facing right.

When the timer expires, press **READ**. Note the results in µg/L CH_2O.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acetate	Greater than 1000 mg/L
Aldehydes (other)	Positive interference at all levels
Ammonium (as N)	Greater than 10 mg/L
Aniline	Greater than 10 mg/L
Bicarbonate	Greater than 1000 mg/L
Calcium	Greater than 3500 mg/L
Carbonate	Greater than 500 mg/L
Chloride	Greater than 5000 mg/L
Copper	Greater than 1.6 mg/L

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Cyclohexylamine	Greater than 250 mg/L
Ethanolamine	Greater than 33 mg/L
Ethylenediamine	Greater than 1.5 mg/L
Glucose	Greater than 1000 mg/L
Glycine	Greater than 1000 mg/L
Iron (Fe^{3+})	Greater than 12 mg/L
Lead	Greater than 100 mg/L
Manganese	Greater than 500 mg/L
Mercury	Greater than 70 mg/L
Morpholine	Greater than 0.36 mg/L
Nitrate	Greater than 1000 mg/L
Nitrite	Greater than 8 mg/L
Phenol	Greater than 1050 mg/L
Phosphate	Greater than 200 mg/L
Silica	Greater than 40 mg/L
Sulfate	Greater than 10,000 mg/L
Urea	Greater than 1000 mg/L
Zinc	Greater than 1000 mg/L

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Formaldehyde Voluette® Ampule Standard, 4000-mg/L CH_2O.

5. Use a TenSette® Pipet to add 0.2 mL of the standard to a 100-mL volumetric Class A flask. Dilute to volume with formaldehyde-free water and mix well. Prepare daily. This is an 8000-µg/L (8-mg/L) formaldehyde standard.

6. Prepare three sample spikes. Fill three 50-mL mixing cylinders* with 25 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of 8000-µg/L standard, respectively, to each sample and mix thoroughly.

7. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

* See Optional Reagents and Apparatus on page 5.

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery

Standard Solution Method
Prepare a 320-µg/L Formaldehyde Standard Solution by pipetting 1.0 mL of the 8000-µg/L solution into a 50-mL mixing cylinder. Dilute to 25.0 mL with formaldehyde-free water. Run the test directly on this sample.

Summary of Method

Formaldehyde reacts with MBTH (3-methyl-2-benzothiazoline hydrazone) and a developing solution to form a blue color in proportion to the formaldehyde concentration. Test results are measured at 630 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Formaldehyde Reagent Set (100 tests), includes:	—	—	22577-00
Developing Solution for LR Formaldehyde	5 mL	500 mL	22572-49
MBTH Powder Pillows	2	100/pkg	22571-69

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers	1	each	968-00
Cylinder, graduated mixing, 50-mL	2	each	1896-41
Pipet, serological, 5-mL	1	each	532-37
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Formaldehyde Standard Solution, 10-mL Voluette® Ampule, 4000-mg/L	16/pkg	22573-10

Optional Reagents and Apparatus

Description	Cat. No.
Chromic Acid Cleaning Solution, 500 mL	1233-49
Potassium Permanganate, 5 lb (2.27 kg)	769-05
Sodium Hydroxide ACS 500g	187-34

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Updated November 2005 Edition 1

Method 8030 Calcium and Magnesium; Calmagite Colorimetric Method (0.05 to 4.00 mg/L Ca and Mg as $CaCO_3$)

Scope and Application: For water, wastewater, and seawater

Test Preparation

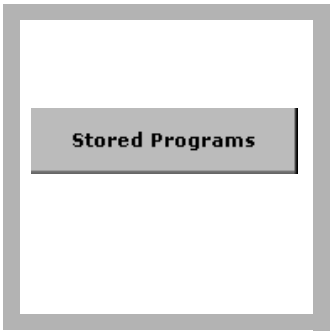
Before starting the test:

For the most accurate magnesium test results, keep the sample temperature between 21–29 °C (70–84 °F).
The test will detect any calcium or magnesium contamination in the mixing cylinder, measuring droppers, or sample cells. To test cleanliness, repeat the test until results are consistent.
Total hardness in mg/L equals mg/L Ca as $CaCO_3$ plus mg/L Mg as $CaCO_3$.
Remaining traces of EDTA or EGTA from previous tests will give erroneous results. Rinse sample cells thoroughly before using.

Collect the following items:	Quantity
Alkali Solution for Calcium and Magnesium test	1 mL
Calcium and Magnesium Indicator Solution	1 mL
EDTA Solution, 1 M	1 drop
EGTA Solution	1 drop
Cylinder, 100-mL, graduated mixing	1
Dropper, measuring, 0.5 and 1.0 mL	2
Sample Cells, 1-inch square	3

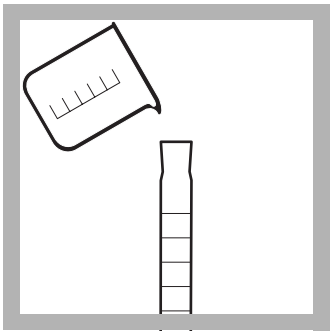
Note: *Reorder information for consumables and replacement items is on page 4.*

Calmagite Method 8030

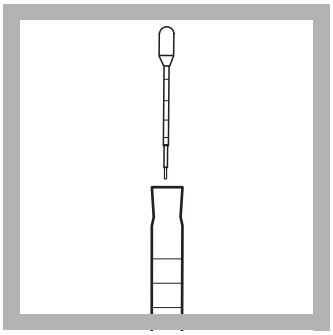


1. Press **STORED PROGRAMS**.

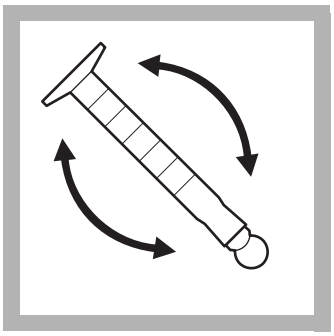
2. Select the test.



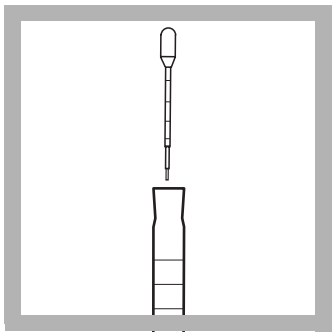
3. Pour 100 mL of sample into a 100-mL graduated mixing cylinder.



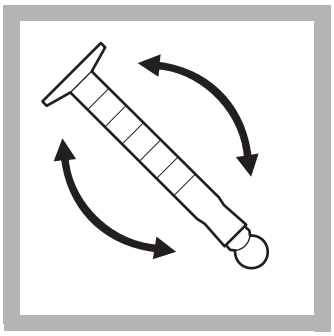
4. Add 1.0 mL of Calcium and Magnesium Indicator solution using a 1.0 mL measuring dropper.



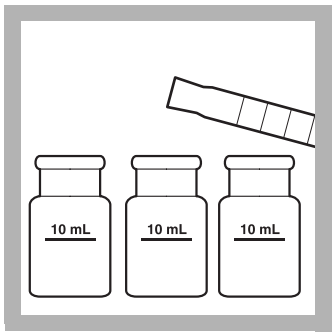
5. Stopper the cylinder and invert it several times.



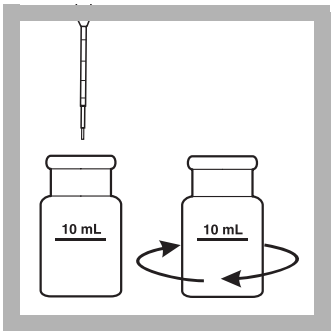
6. Add 1.0 mL of Alkali Solution for Calcium and Magnesium Test using a 1.0 mL measuring dropper.



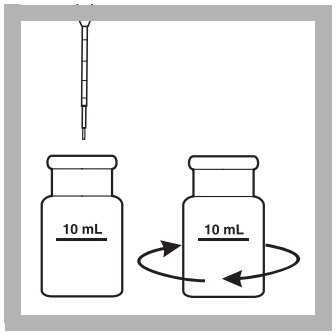
7. Stopper the cylinder and invert it several times.



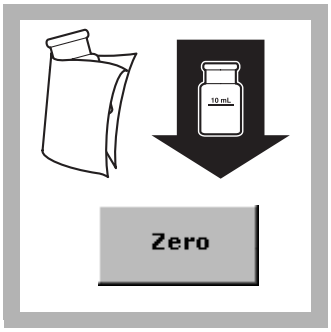
8. Pour 10 mL of the solution into each of **three** square sample cells.



9. Blank Preparation: Add one drop of 1 M EDTA Solution to the **first** cell. Swirl to mix.



10. Magnesium Sample: Add one drop of EGTA Solution to the **second** cell. Swirl to mix.



11. Insert the **blank** (first cell) into the cell holder with the fill line facing right.

Press **ZERO**.

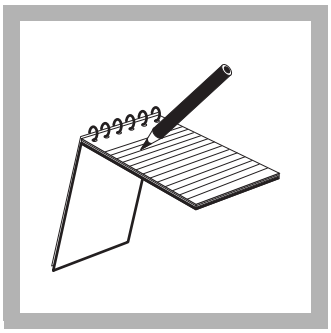
The display will show:

0.00 mg/L Mg $CaCO_3$

12. Insert the **magnesium sample** (second cell) into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L magnesium as calcium carbonate.

This value is the amount of magnesium in the sample expressed as $CaCO_3$.

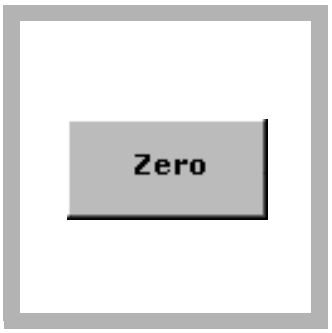


13. Do not remove the cell from the instrument. Record or store the magnesium results before proceeding with step 14.

14. Press **EXIT.**

Select the test.

Press **START**.

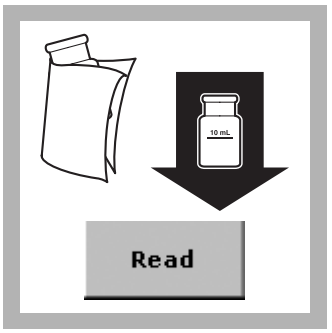


15. Press **ZERO**.

The display will show:

0.00 mg/L Ca $CaCO_3$

Remove the second cell.



16. Calcium Sample: Insert the **third** cell into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L calcium as calcium carbonate.

This value is the amount of calcium in the sample expressed as $CaCO_3$.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chromium ($^{3+}$)	Above 0.25 mg/L
Copper ($^{2+}$)	Above 0.75 mg/L
EDTA, chelated	Above 0.2 mg/L as $CaCO_3$
EDTA or EGTA	Traces remaining in sample cells from previous tests will give erroneous results. Rinse cells thoroughly before using.
Iron ($^{2+}$)	Above 1.4 mg/L
Iron ($^{3+}$)	Above 2.0 mg/L
Manganese ($^{2+}$)	Above 0.20 mg/L
Zinc ($^{2+}$)	Above 0.050 mg/L
Ca >1.0 mg/L; Mg >0.25 mg/L	For the most accurate calcium test result, rerun the test on a diluted sample if the calcium is over 1.0 and the magnesium is over 0.25 mg/L as $CaCO_3$. No retesting is needed if either is below those respective concentrations.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with Nitric Acid* (about 5 mL per liter). Cool samples to 4 °C. Preserved samples can be stored up to six months. Before analysis, adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution*. Correct the test results for volume additions.

* See Optional Reagents and Apparatus on page 4.

Summary of Method

The colorimetric method for measuring hardness supplements the conventional titrimetric method because the colorimetric method can measure very low levels of calcium and magnesium. Also, some metals (those listed the table above) that interfere in the titrimetric method may be inconsequential when diluting the sample to bring it within the range of this test. The indicator dye is calmagite, which forms a purplish-blue color in a strongly alkaline solution and changes to red when it reacts with free calcium or magnesium. Calcium and magnesium determinations are made by chelating calcium with EGTA to destroy any red color due to calcium and then chelating the calcium and magnesium with EDTA to destroy the red color due to both calcium and magnesium. By measuring the red color in the different states, calcium and magnesium concentrations are determined. Test results are measured at 522 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Hardness Reagent Set (100 tests), includes:	—	—	23199-00
Alkali Solution for Calcium and Magnesium test	1 mL	100 mL MDB	22417-32
Calcium and Magnesium Indicator Solution	1 mL	100 mL MDB	22418-32
EDTA Solution, 1 M	1 drop	50 mL SCDB	22419-26
EGTA Solution	1 drop	50 mL SCDB	22297-26

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, 100-mL, graduated mixing	1	each	1896-42
Dropper, measuring, 0.5 and 1.0 mL	2	20/pkg	21247-20
Sample Cells, 1-inch square, 10 mL, matched pair	3	2/pkg	24954-02

Optional Reagents and Apparatus

Description	Cat. No.
Nitric Acid	152-49
Sodium Hydroxide Standard Solution, 5.0 N	2450-32

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Updated November 2005 Edition 1

Hardness

Method 8374

Calcium and Magnesium; Chlorophosphonazo Colorimetric Method (8 to 1000 µg/L Ca and Mg as $CaCO_3$)

Scope and Application: For water, wastewater, and seawater

Test Preparation

Before starting the test:

For the most accurate magnesium test results, keep the sample temperature between 21–29 °C (70–84 °F).
The test will detect any calcium or magnesium contamination in the mixing cylinder, measuring droppers, or sample cells. To test cleanliness, repeat the test until results are consistent.
One mL of Chlorphosphonazo Solution* may be used instead of the solution pillow in step 5.
Total hardness in mg/L equals mg/L Ca as $CaCO_3$ plus mg/L Mg as $CaCO_3$.

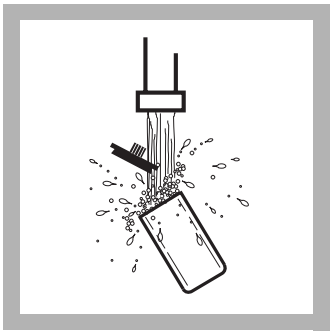
Collect the following items:	Quantity
ULR Hardness Reagent Set	1
Chlorophosphonazo Indicator Solution Pillow	1
CDTA Solution	1 drop
Shears for opening powder pillow	1
Sample Cells, 1-inch square polystyrene, with cap	1

Note: *Reorder information for consumables and replacement items is on page 4.*

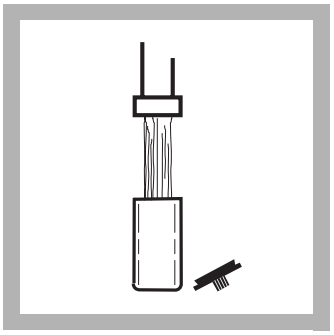
Chlorophosphonazo Method 8374

1. Press **STORED PROGRAMS**.

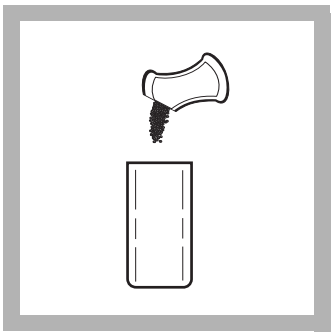
2. Select the test.



3. Rinse a plastic sample cell and the cap three times with the water to be tested. Do not allow the underside of the cap to come in contact with surfaces that may contaminate it.



4. Fill the plastic sample cell to the 25-mL mark with sample.

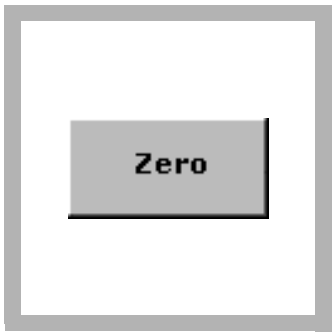


5. Add the contents of one Chlorophosphonazo Solution Pillow to the sample cell.

A small amount of solution may remain in the pillow. This will not affect results.

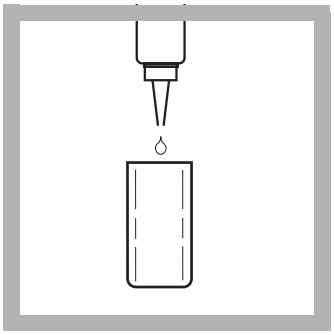
6. Cap the cell and swirl to mix.

7. Insert the cell into the cell holder.



8. Press **ZERO**.

The display will show: 0 µg/L $CaCO_3$.



9. Remove the cell from the holder. Add one drop of CDTA Reagent for Ultra Low Range Hardness.

Complete steps 10-11 within 1-2 minutes.

10. Cap the cell and swirl to mix.

11. Insert the cell into the holder.

12. Press **READ**.

Results are in µg/L $CaCO_3$.

Interferences

Interference studies were conducted at various hardness levels between 0 and 500 µg/L as $CaCO_3$. Various cations and anions were evaluated at levels in the range appropriate for ultra pure water applications. An ion is said to interfere when the resulting concentration is changed by ± 10%.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Negative interference above 150 µg/L
Ammonium	No interference at or below 1000 µg/L
Copper	Positive interference above 250 µg/L
Formaldehyde	No interference at or below 47,000 µg/L
Nitrate	Positive interference above 250 µg/L
Potassium	No interference at or below 1000 µg/L

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Silicon	Positive interference above 1000 µg/L
Sodium	Negative interference above 79,000 µg/L

Sample Collection, Storage, and Preservation

Do not use glass containers. Collect samples in clean plastic containers, preferably with screw-type closures. Rinse containers several times with the water to be analyzed before collecting the final sample. Seal to avoid contamination during transport. Analyze as soon as possible.

Accuracy Check

Standard Additions Method

1. Leave the unspiked sample in the sample cell compartment. Verify that the units displayed are in µg/L. Select standard additions mode by pressing **OPTIONS>MORE** and then **STANDARD ADDITIONS**.

2. Press **ENTER** to accept the default sample volume (mL), 25.

3. Obtain a Calcium Chloride Standard Solution, 20,000-µg/L as $CaCO_3$.

4. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to three 25-mL samples and mix each thoroughly.

5. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

6. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Using the 0.50-mg/L (500-µg/L as $CaCO_3$) Calcium Chloride Standard Solution, perform the procedure using the standard in place of the sample.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Calcium and magnesium combine equivalently with the Chlorophosphonazo III indicator to form a colored complex which absorbs light very strongly at 669 nm. One drop of the CDTA reagent breaks up this complex, and the resultant decrease in color is proportional to the amount of calcium and magnesium in the sample (as CaCO3). Test results are measured at 669 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
ULR Hardness Reagent Set (100 tests), includes:	—	—	26031-00
(1) 25895-99, (1) 25896-36, (1) 24102-01, (1) 24102-02			
OR			
ULR Hardness Reagent Set (500 tests), includes:	—	—	26031-01
(1) 25895-49, (2) 25896-36, (1) 24102-01, (1) 24102-02			
Chlorophosphonazo Indicator Solution Pillows	1	100/pkg	25895-99
CDTA Solution	1 drop	10 mL SCDB	25896-36

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Shears	1	each	23694-00
Sample Cells, 1-inch polystyrene with cap	1	12/pkg	24102-12

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Calcium Chloride Standard Solution, 20,000-µg/L as $CaCO_3$	946 mL	21246-16
Calcium Chloride Standard Solution, 500-µg/L as $CaCO_3$	946 mL	20580-16
Chlorophosphonazo Indicator Solution	500 mL	25895-49

Optional Reagents and Apparatus

Description	Cat. No.
Dispenser, 1.0-mL, Repipet Jr.	21113-02
Pipet, TenSette®, 0.1 to 1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01, 50/pkg	21856-96

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Updated November 2005 Edition 1

Hardness, Total

Method 8374 Calcium and Magnesium; Chlorophosphonazo Rapid Liquid Method
Pour-Thru Cell ULR (4 to 1000 µg/L Ca & Mg as $CaCO_3$)

Scope and Application: For boiler, cooling, and ultra pure water

Test Preparation

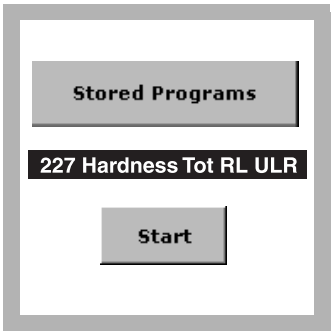
Before starting the test:

Pre-clean the Pour-Thru Cell and all labware as specified in Treating Analysis Labware on page 4.
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel.
If the sample concentration is greater than 750 µg/L, a 1:1 dilution of the sample is recommended for greatest accuracy. Use ultra-pure (aldehyde-free) water for the dilution. Repeat the analysis on the diluted sample and multiply the resulting concentration by two.
Alternate forms should only be used when the sample is known to contain only Mg or Ca. This method does not distinguish between the two forms.
Refer to the instrument User Manual for Pour-Thru cell and module assembly and installation.
Use dedicated plasticware for this analysis.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

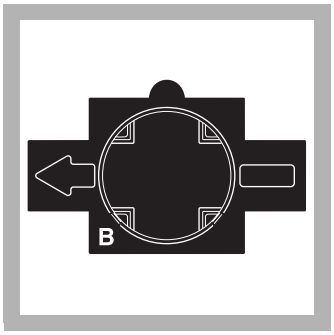
Collect the following items:	**Quantity**
Chlorophosphonazo Indicator Solution	2 mL
CDTA Reagent for Ultra Low Range Hardness	1 drop
Cylinder, graduated, 50-mL, poly	1
Dispenser, fixed-volume, 2.0-mL, Repipet Jr.	1
Flask, Erlenmeyer, PMP w/cap, 125-mL	1
Pour-Thru Cell Module	1

Note: *Reorder information for consumables and replacement items is on page 6.*

Pour-Thru Cell — Method 8374



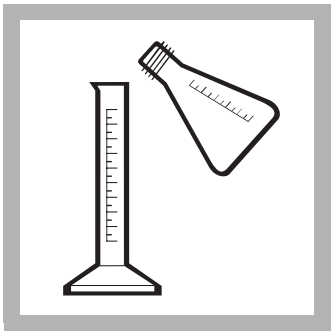
1. Select the test.



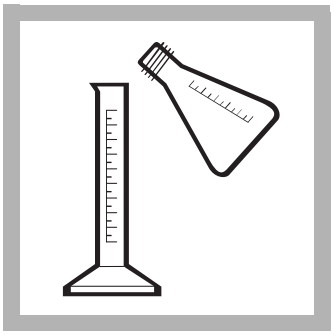
2. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch round path in line with the adapter arrow. Flush the Pour-Thru cell with 50 mL of ultra-pure water.

3. Fill a clean, 125-mL plastic Erlenmeyer flask to overflowing with sample.

Collect sample directly in the flask if possible.

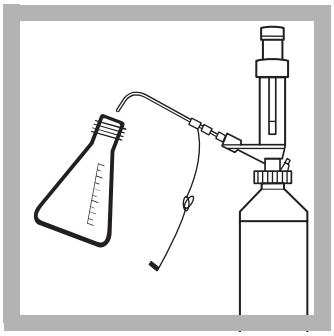


4. Rinse a clean, 50-mL plastic graduated cylinder three times with sample.

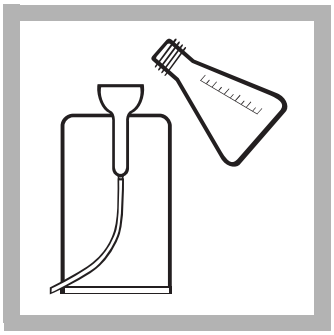


5. Fill the rinsed cylinder to the 50-mL mark with sample from the flask. Discard remaining contents of the flask.

6. Pour the contents of the 50-mL cylinder back into the flask.

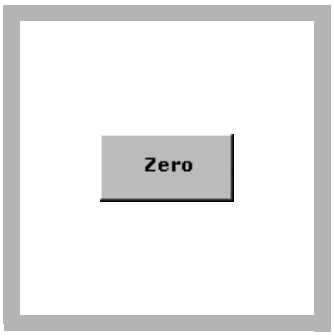


7. Add 2.0 mL of Chlorophosphonazo Reagent to the sample with the Repipet Jr. Dispenser. Swirl to mix.



8. Pour approximately half (25 mL) of the sample into the Pour-Thru Cell.

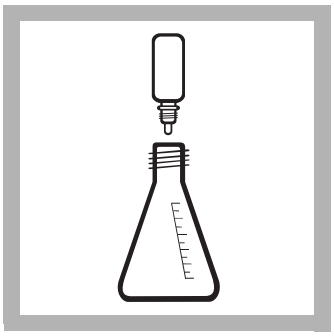
Use a clean, dry, plastic 25-mL graduated cylinder to measure the sample.



9. After the flow stops, press **ZERO**.

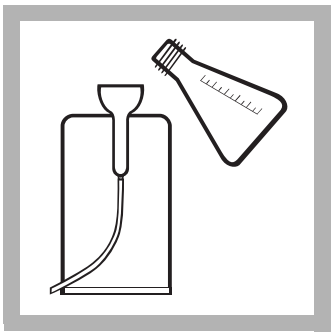
The display will show:

0 µg/L $CaCO_3$

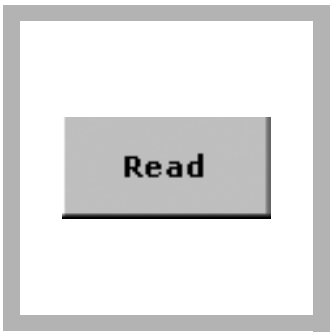


10. Add one drop of CDTA Reagent for Ultra Low Range Hardness to the remaining sample in the flask. Swirl to mix.

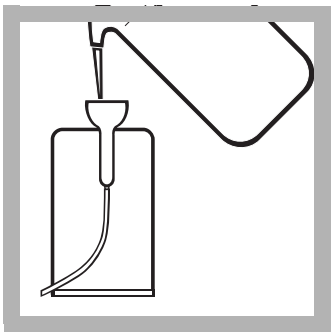
Complete steps 11 and 12 within one to two minutes.



11. Pour the remaining sample into the Pour-Thru Cell.

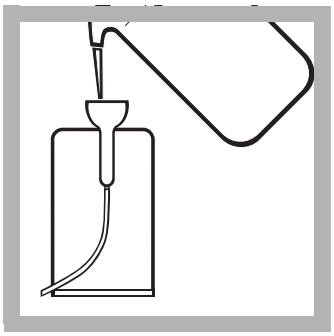


12. After the flow stops, press **READ**. Results are in µg/L $CaCO_3$.



13. Using a wash bottle, rinse the Pour-Thru Cell with ultra-pure water immediately after use.

Rinse the flask with ultra-pure water. Cap when finished.



14. Flush the Pour-Thru cell with an additional 50-mL of ultra-pure water.

Interferences

Interference studies were conducted at various hardness levels between 0 and 500 µg/L as $CaCO_3$. Various cations and anions were evaluated at levels in the range appropriate to ultra pure water applications. An ion is said to interfere when the resulting concentration is changed by ± 10%.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Negative interference above 150 µg/L
Ammonium	No interference at or below 1000 µg/L
Copper	Positive interference above 250 µg/L
Formaldehyde	No interference at or below 47,000 µg/L
Potassium	No interference at or below 1000 µg/L
Silicon	Positive interference above 1000 µg/L
Sodium	Negative interference above 79,000 µg/L

Treating Analysis Labware

Clean all containers used in this test thoroughly to remove any traces of calcium or magnesium. If possible, use plastic containers for all analysis and storage. Clean containers by normal means, then rinse with ultra-pure (aldehyde-free) water. Fill and soak for 10 minutes with a 1:25 dilution of Chlorophosphonazo Reagent in ultra-pure water. Rinse well with ultra-pure water. Keep containers tightly closed and dedicate them for ULR Hardness only. If containers are rinsed and capped after each use, only occasional soaking is necessary. Fill the Pour-Thru cell with this same mixture of chlorophosphonazo and water and let stand for several minutes. Rinse with ultra-pure water.

Avoid contamination of the Chlorophosphonazo Reagent bottle when placing the Repipet dispenser on the bottle. Rinse the inlet tubing and inside of the dispenser cap with copious amounts of ultra-pure water using a wash bottle. Place the inlet tubing into a beaker of ultra-pure water and depress the plunger 10–15 times to rinse the inside of the dispenser. (For best results, pour a small amount of reagent into the beaker of rinse water.) Remove the dispenser from the water and depress the plunger until all of the water has been expelled. Shake off any excess water on the dispenser, place the dispenser on the bottle, and tighten.

Sampling and Storage

Do not use glass containers. Collect samples in clean plastic containers, preferably with screw-type closures. Rinse containers several times with the water to be analyzed before collecting the final sample. Seal to avoid contamination during transport. Analyze as soon as possible.

Accuracy Check

Standard Additions Method (Sample Spike)

1. Leave the unspiked sample in the sample cell compartment. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

2. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

3. Obtain a Calcium Chloride Standard Solution, 20-mg/L (20,000-µg/L) as $CaCO_3$.

4. Prepare three sample spikes. Use a TenSette® Pipet to add 0.2 mL, 0.4 mL, and 0.6 mL, of a 20-mg/L as $CaCO_3$ Calcium Chloride Standard to three 50-mL samples, respectively.

5. Analyze each sample spike as described in the procedure above, starting with the 0.2 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

6. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery..

Standard Solution Method

1. Using a 0.50-mg/L (500-µg/L as $CaCO_3$) Calcium Chloride Standard Solution, perform the procedure using the standard in place of the sample.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternative concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Calcium and magnesium combine equivalently with the Chlorophosphonazo Indicator to form a colored complex which absorbs light very strongly at 669 nm. One drop of the CDTA reagent breaks up this complex, and the resultant decrease in color is proportional to the amount of calcium and magnesium (as $CaCO_3$) in the sample. Test results are measured at 669 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Chlorophosphonazo Indicator Solution	2 mL	500 mL	25895-49
CDTA Reagent for Ultra Low Range Hardness	1 drop	10 mL SCDB	25896-36

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 50-mL, poly	1	each	1081-41
Dispenser, fixed-volume, 2.0-mL, Repipet Jr.	1	each	22307-01
Flask, Erlenmeyer, PMP w/cap, 125-mL	1	each	20898-43
Pour-Thru Cell Kit	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Calcium Standard Solution, 20-mg/L as $CaCO_3$	946 mL	21246-16
Calcium Standard Solution, 0.50-mg/L as $CaCO_3$	946 mL	20580-16
Water, ultra-pure (aldehyde-free)	500 mL	25946-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Pipet, TenSette® 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	25/pkg	21856-96

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Updated November 2005 Edition 1

Hydrazine

Method 8141 — p-Dimethylaminobenzaldehyde Method[1]

Reagent Solution or AccuVac® Ampuls — (4 to 600 µg/L)

Scope and Application: For boiler water/feedwater

[1] Adapted from *ASTM Manual of Industrial Water*, D1385-78, 376 (1979)

Test Preparation

Before starting the test:

Samples cannot be preserved and must be analyzed immediately
Sample temperature should be 21 ± 4 °C (70 ± 7 °F).
After adding the HydraVer® 2 Hydrazine Reagent, a yellow color will develop in the sample if hydrazine is present. The blank may also have a faint yellow color.

Collect the following items:	**Quantity**
Solution Test:	
HydraVer 2 Reagent Solution	4 mL
Deionized Water	10 mL
Graduated Cylinder, 25-mL	1
AccuVac Test:	
HydraVer 2 Reagent AccuVac® Ampuls	2
Deionized Water	40 mL
Beaker, 50-mL	1

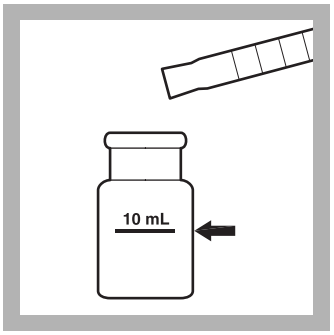
Note: *Reorder information for consumables and replacement items is on page 4.*

Important Note: *The final samples will have a pH less than 2, which is considered corrosive (0002) by the Federal RCRA. Refer to the MSDS for disposal instructions.*

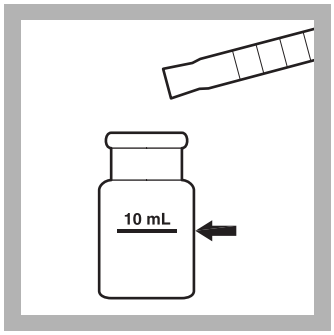
Reagent Solution — Method 8141

1. Select the test.

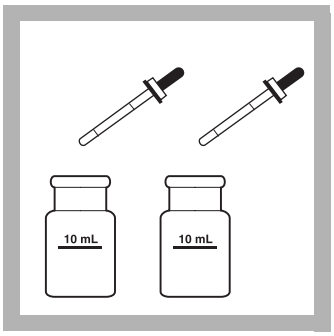
2. Select the test.



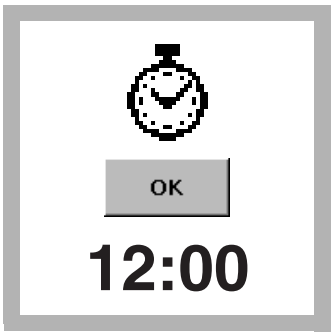
3. Blank Preparation: Use a graduated cylinder to pour 10 mL of deionized water into a square sample cell.



4. Prepared Sample: Use a graduated cylinder to pour 10 mL of sample into a second square sample cell.

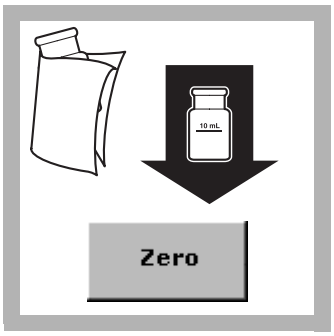


5. Add 0.5 mL of HydraVer 2 Hydrazine Reagent to each sample cell. Swirl to mix.



6. Press **TIMER>OK**.

A 12-minute reaction period will begin. Complete steps 7–8 during this period.



7. Insert the blank into the cell holder with the fill line facing right. Press **ZERO**.

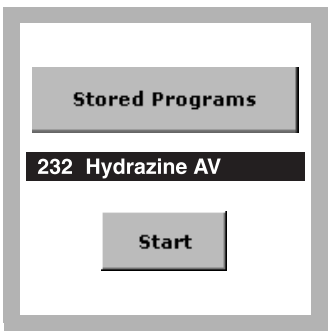
The display will show:

0 µg/L N_2H_4

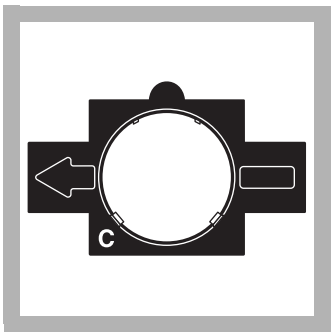
8. Insert the prepared sample into the cell holder. with the fill line facing right.

Immediately after the timer expires, press **READ.** Results are in µg/L N_2H_4.

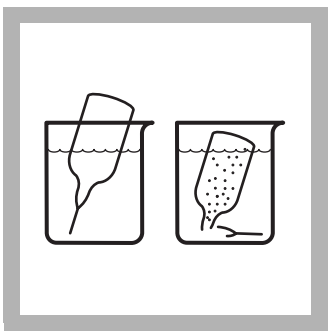
AccuVac® Ampul — Method 8141



1. Select the test.



2. Insert Adapter C.

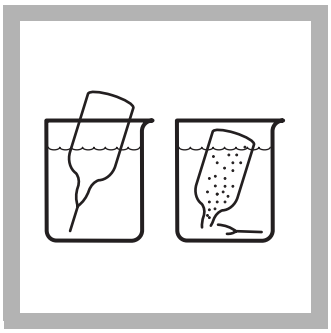


3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a HydraVer Hydrazine AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely.

4. Immediately press **TIMER>OK**.

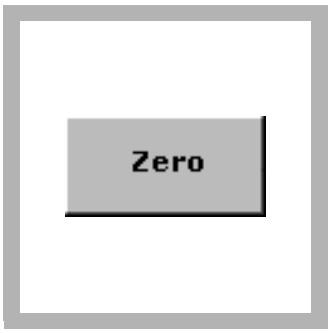
A 12-minute reaction period will begin. Complete steps 5–7 during this period.



5. Blank Preparation: Pour at least 40-mL of deionized water into a second beaker.

Fill a second Ampul with deionized water. Keep the tip immersed while the Ampul fills completely.

6. Insert the blank into the cell holder.



7. Press **ZERO**.

The display will show:

0 µg/L N_2H_4

8. Insert the prepared sample in the cell holder.

Immediately after the timer expires, press **READ**. Results are in µg/L N_2H_4.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Ammonia	No interference up to 10 mg/L. May cause a positive interference of up to 20% at 20 mg/L.
Highly colored or turbid samples	Prepare a blank by oxidizing the hydrazine in a portion of the sample with a 1:1 mixture of deionized water and household bleach. Add one drop of the mixture to 25 mL of sample in a graduated mixing cylinder and invert to mix. Use this solution in step 2, instead of deionized water, to prepare the blank.
Morpholine	No interference up to 10 mg/L.

Sample Collection, Storage, and Preservation

Samples collected in glass or plastic bottles should be filled completely and capped tightly. Avoid excessive agitation or exposure to air. Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

Accuracy Check

Standard Solutions Method

1. Prepare a 25-mg/L stock solution. Dissolve 0.1016 g of hydrazine sulfate in 1000 mL of oxygen-free deionized water. Prepare this stock solution daily.

2. Using Class A glassware, prepare a 0.25-mg/L (250-µg/L) hydrazine working solution by diluting 10.00 mL of the 25-mg/L stock solution to 1000 mL with deoxygenated deionized water. Prepare just before analysis. Perform either hydrazine procedure as described above.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Hydrazine in the sample reacts with the p-dimethylaminobenzaldehyde from the HydraVer 2 Reagent to form a yellow color which is proportional to the hydrazine concentration. Test results are measured at 455 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
HydraVer® 2 Hydrazine Reagent	1 mL	100 mL MDB	1790-32
OR			
HydraVer 2 Hydrazine Reagent AccuVac® Ampuls	2	25/pkg	25240-25
Water, deionized	10 mL	4 L	272-56

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 25-mL.	1	each	508-40
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H

Recommended Standards

Description	Unit	Cat. No.
Hydrazine Sulfate, ACS	100 g	742-26

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Updated November 2005 Edition 1

Iodine

Method 8031 — DPD Method[1]

Powder Pillows or AccuVac® Ampuls — (0.07 to 7.00 mg/L)

Scope and Application: For testing dissolved iodine residual used as disinfectant in process water, treated water, estuary water, and seawater

[1] Adapted from Palin, A.T., *Inst. Water Eng.*, 21 (6), 537-547 (1967).

Test Preparation

Before starting the test:

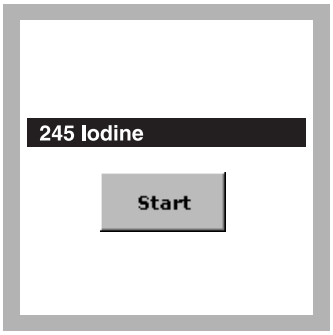
Analyze samples immediately. Do not preserve for later analysis
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
If the sample temporarily turns yellow after reagent addition, dilute a fresh sample. Repeat the test. A slight loss of iodine may occur due to the dilution. Apply the appropriate dilution factor.

Collect the following items:	**Quantity**
Powder Pillow Test:	
DPD Total Chlorine Reagent Powder Pillow	1
Sample cells, 1-inch square, 10-mL	2
AccuVac Test:	
DPD Total Chlorine Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cell, 10-mL, round	1
Stopper for 18 mm Tube	2

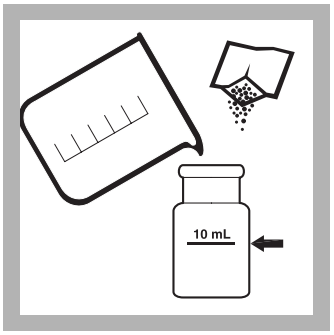
Note: *Reorder information for consumables and replacement items is on page 4.*

Powder Pillows — Method 8031

1. Press **STORED PROGRAMS**.

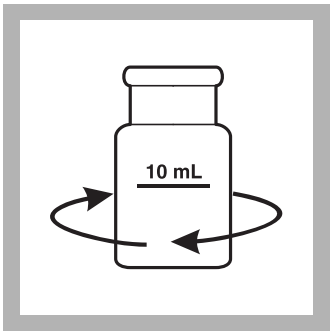


2. Select the test.



3. Prepared Sample: Fill a square sample cell with 10 mL of sample.

Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell.

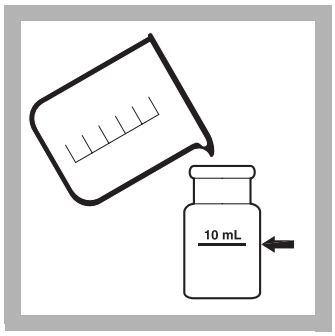


4. Swirl for about 20 seconds to mix.

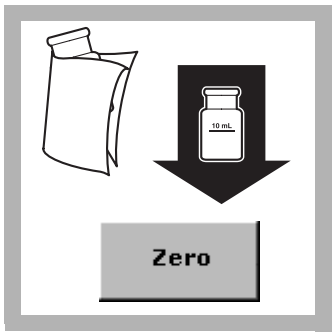
A pink color will develop if iodine is present.

5. Press **TIMER>OK**.

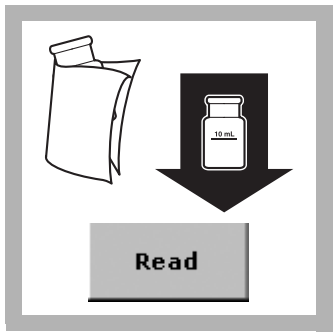
A three-minute reaction period will begin.



6. Blank Preparation: Fill a second square sample cell with 10 mL of sample.

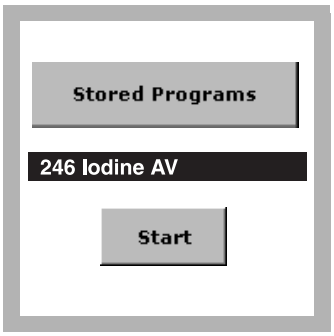


7. Wipe the blank and insert it into the cell holder with the fill line facing right. Close the cover.
Press **ZERO**. The display will show:
0.00 mg/L I_2

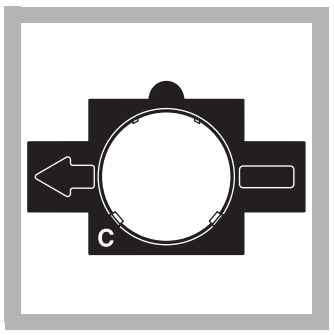


8. Within three minutes after the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing right.
Press **READ**. Results are in mg/L I_2.

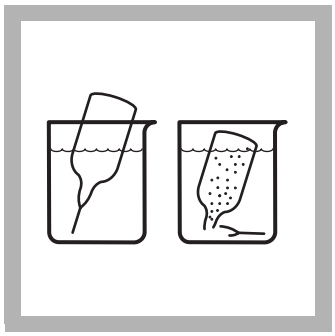
AccuVac Ampul — Method 8031



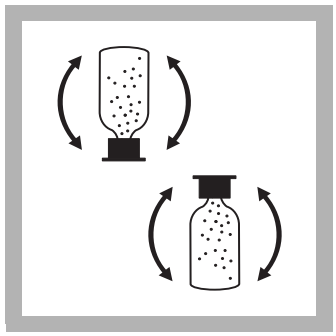
1. Select the test.



2. Insert Adapter C.



3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill a DPD Total Chlorine Reagent AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely.



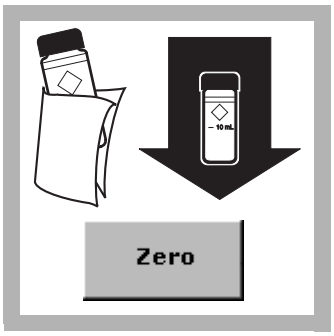
4. Quickly invert the Ampul several times to mix.

A pink color will develop if iodine is present.

5. Press **TIMER>OK**.

A three-minute reaction period will begin. Perform steps 6–7 during the reaction period.

6. Blank Preparation: Fill a round sample cell with 10 mL of sample.



7. Wipe the blank and insert it into the cell holder. Press **ZERO**. The display will show:

0.00 mg/L I_2

8. Within three minutes after the timer expires, wipe the prepared sample and insert it into the cell holder. Press **READ**. Results are in mg/L I_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sodium Hydroxide[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N Sulfuric Acid[1]. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine	Interferes at all levels
Chlorine and chloramines	Causes a positive interference at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as $CaCO_3$
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7. **2.** Add 3 drops Potassium Iodide[1] (30-g/L) to a 25-mL sample. **3.** Mix and wait 1 minute. **4.** Add 3 drops Sodium Arsenite[1, 2] (5-g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct iodine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust to pH 6–7.

[1] See Optional Reagents and Apparatus on page 4.

[2] Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by the Federal RCRA for arsenic (D004). Refer to the current MSDS for disposal information.

Sample Collection, Storage, and Preservation

Collect samples in clean, dry glass containers. If sampling from a tap, allow the water to flow at least 5 minutes to ensure a representative sample. Avoid excessive agitation and exposure to sunlight when sampling. Allow several volumes of water to overflow the container and cap the container so there is no headspace above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Proceed with the analysis immediately.

Summary of Method

Iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) to form a pink color, the intensity of which is proportional to the total iodine concentration. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1	100/pkg	21056-69
OR			
DPD Total Chlorine Reagent AccuVac® Ampuls	1	25/pkg	25030-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper for 18 mm tube	—	6/pkg	1731-06

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Optional Reagents and Apparatus

Description	Cat. No.
Potassium Iodide, 30 g/L, 100 mL	343-32
Sodium Arsenite, 5-g/L, 100 mL	1047-32
Sodium Hydroxide, 1 N, 100 mL	1045-32
Stopper for 18 mm tube, 25/pkg	1731-25
Sulfuric Acid, 1 N, 100 mL	1270-32

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Updated November 2005 Edition 1

Iron

Method 8147
FerroZine® Reagent Solution Pillows

FerroZine® Method[1]
(0.009 to 1.400 mg/L)

Scope and Application: For water and wastewater

[1] Adapted from Stookey, L.L., *Anal. Chem.*, 42(7), 779 (1970)

Test Preparation

Before starting the test:

Digestion is required for total iron determination.
Rinse glassware with a 1:1 hydrochloric acid solution. Rinse again with deionized water. These two steps will remove iron deposits that can cause slightly high results.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Instead of solution pillows, 0.5 mL of FerroZine® Iron Reagent Solution can be used.
If the sample contains rust, see Interferences on page 3.
Use clean clippers, free of rust, and wipe with a dry towel. Do not allow clippers to contact contents of the pillow.
FerroZine Iron Reagent may crystallize or precipitate when exposed to cold temperatures during shipment. Reagent quality is not affected. Place the reagent in warm water to redissolve.

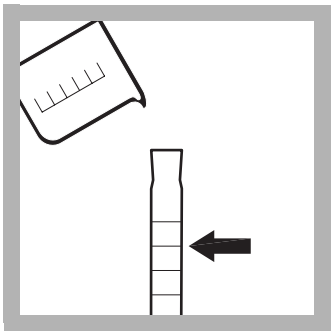
Collect the following items:	**Quantity**
FerroZine Iron Reagent Solution Pillows	1
OR	
FerroZine Iron Reagent Solution	0.5 mL
Cylinder, 25-mL graduated mixing, with stopper	1
Clippers for solution pillows	1
Sample Cells, 1-inch square, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 5.*

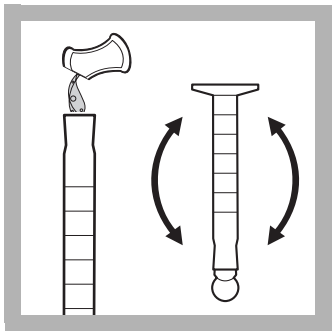
Solution Pillows — Method 8147

1. Press **STORED PROGRAMS**.

2. Select the test.



3. Fill a clean 25-mL graduated mixing cylinder to the 25-mL mark with sample.

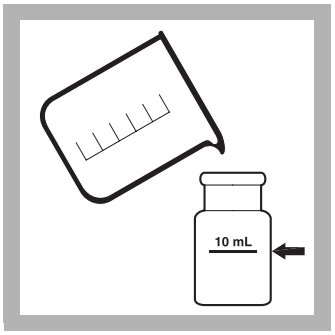


4. Prepared Sample: Add the contents of one FerroZine® Iron Reagent Solution Pillow to the mixing cylinder.

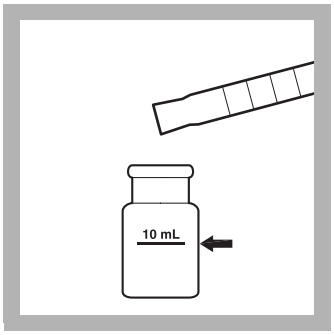
Stopper and invert to mix.

5. Press **TIMER>OK**.

A five-minute reaction period will begin. A purple color will develop if iron is present.

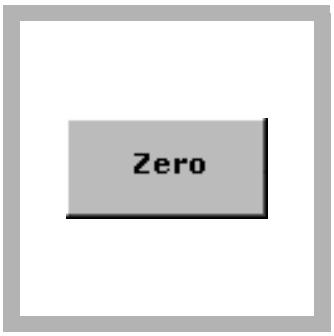


6. Blank Preparation: Fill a square sample cell with 10 mL of sample.



7. When the timer expires, pour 10 mL of the prepared sample into a second clean square sample cell.

8. Insert the blank into the cell holder with the fill line facing right.

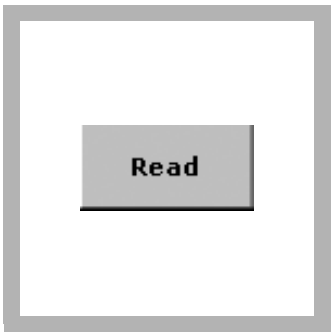


9. Press **ZERO**.

The display will show:

0.000 mg/L Fe

10. Insert the prepared sample into the cell holder with the fill line facing right.



11. Press **READ**.

Results are in mg/L Fe.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Strong chelants (EDTA)	Interfere at all levels. Use the FerroVer® or TPTZ methods for these samples. Use the TPTZ method for low iron concentrations.
Cobalt	May give slightly high results
Copper	May give slightly high results
Hydroxides	Boil the sample, with the FerroZine® Iron Reagent added to it from step 4, for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with step 5. Return the sample volume to 25 mL with deionized water.
Magnetite (black iron oxide) or Ferrites	**1.** Fill a 25-mL graduated cylinder with 25 mL of sample. **2.** Transfer this sample into a 125-mL Erlenmeyer flask. **3.** Add the contents of one FerroZine® Iron Reagent Solution Pillow and swirl to mix. **4.** Place the flask on a hot plate or over a flame and bring to a boil. **5.** Continue boiling gently for 20 to 30 minutes. ***Note:*** *Do not allow to boil dry.* ***Note:*** *A purple color will develop if iron is present.* **6.** Return the boiled sample to the 25-mL graduated cylinder. Rinse the Erlenmeyer flask with small amounts of deionized water and empty into the graduated cylinder. **7.** Return the sample volume to the 25-mL mark with deionized water. **8.** Pour this solution into a sample cell and swirl to mix. Proceed with steps 5–10.
Rust	Boil the sample, with the FerroZine Iron Reagent added to it from step 4, for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with step 5. Return the sample volume to 25 mL with deionized water.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with concentrated Nitric Acid, ACS* (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only reporting dissolved iron, filter the sample immediately after collection and before adding nitric acid.

Before testing, adjust the sample pH to 3–5 with Ammonium Hydroxide, ACS*. Do not exceed pH 5, or iron may precipitate. Correct test results for volume additions.

* See Optional Reagents and Apparatus on page 5.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off an Iron Voluette® Ampule Standard, 10-mg/L Fe.

5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to the unspiked sample. Press the timer icon. After the timer expires, read the result.

6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Press the timer icon. After the timer expires, read the result.

7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Press the timer icon. After the timer expires, read the result. Each addition should reflect approximately 100% recovery.

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Using Class A glassware, prepare a 1.0-mg/L Fe standard solution by pipetting 5.00 mL of Iron Standard Solution, 100-mg/L, into a 500-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the iron procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The FerroZine® Iron Reagent forms a purple-colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols and with digestion can be used to analyze samples containing magnetite (black iron oxide) or ferrites. Test results are measured at 562 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
FerroZine® Iron Reagent Solution, or	0.5 mL	500 mL	2301-49
FerroZine® Iron Reagent Solution Pillows	1	50/pkg	2301-66

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers for solution pillows	1	each	968-00
Cylinder, graduated mixing, 25-mL with stopper	1	each	20886-40
Sample Cells, 1-inch square, 10-mL, matched pair	2	2/pkg‘	24954-02

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Iron Standard Solution, 100-mg/L Fe	100 mL	14175-42
Iron Standard Solution, 10-mL Voluette® ampule, 10-mg/L Fe	16/pkg	14253-10
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	28337-49
Flask, volumetric, Class A, 500 mL	each	14574-49
Pipet, TenSette, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00

Optional Reagents and Apparatus

Description	Cat. No.
Ammonium Hydroxide, ACS, 58%	106-49
Hydrochloric Acid, 1:1, 6N	884-49
Nitric Acid, ACS, concentrated	152-49

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Updated November 2005 Edition 1

Iron, Ferrous

Method 8146 — 1, 10 Phenanthroline Method[1]

Powder Pillows or AccuVac® Ampuls — (0.02 to 3.00 mg/L)

Scope and Application: For water, wastewater, and seawater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*, 15th ed. 201 (1980)

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric iron, which is not determined.
If ferrous iron is present, an orange color will form after adding the reagent.

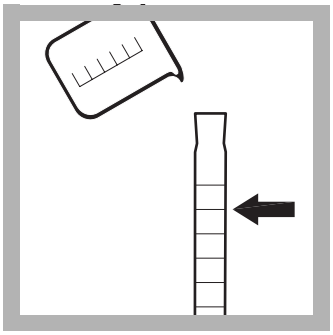
Collect the following items:	**Quantity**
Powder Pillow Test:	
Ferrous Iron Reagent Powder Pillows	1
Sample Cells, 1-inch square, 10-mL	2
AccuVac Test:	
Ferrous Iron Reagent AccuVac® Ampuls	1
Beaker, 50-mL (AccuVac test)	1
Sample Cell, 10-mL	1

Note: *Reorder information for consumables and replacement items is on page 5.*

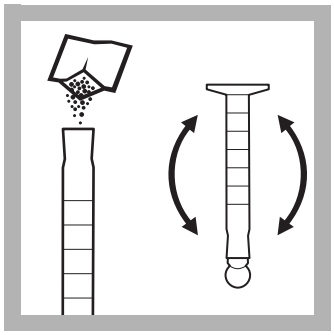
Powder Pillows — Method 8146

1. Press **STORED PROGRAMS**.

2. Select the test.



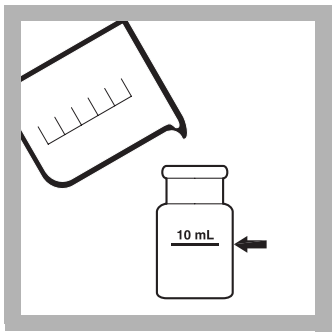
3. Fill a clean, mixed graduated cylinder with 25 mL of sample.



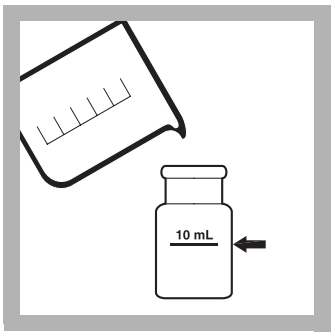
4. Prepared Sample: Add the contents of one Ferrous Iron Reagent Powder Pillow to the cylinder. Stopper and invert to mix.

5. Press **TIMER>OK**.

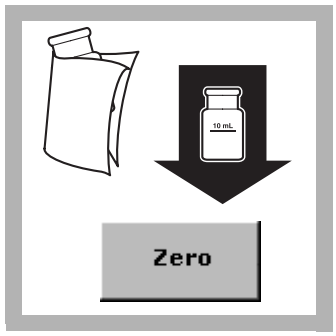
A three-minute reaction period will begin.



6. Blank Preparation: Fill a square cell with 10 mL of sample.



7. Fill a second square sample cell with the prepared sample from the mixing cylinder.

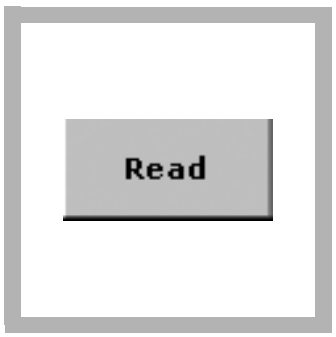


8. When the timer expires, insert the blank into the cell holder with the fill line facing right.

Press **ZERO**. The display will show:

0.00 mg/L Fe^{2+}

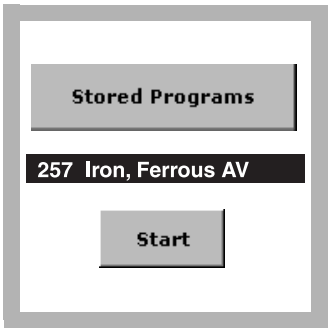
9. Insert the prepared sample into the cell holder with the fill line facing right.



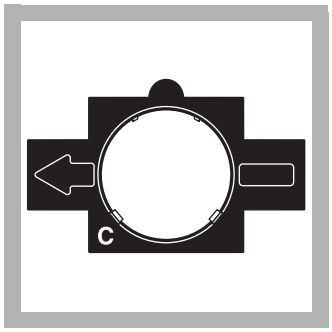
10. Press **READ**.

Results are in mg/L Fe^{2+}.

AccuVac Ampul — Method 8146

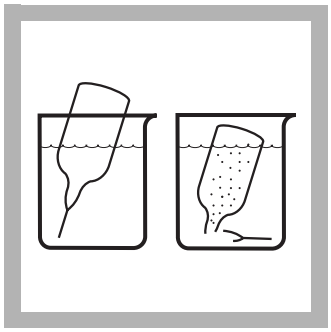


1. Select the test.



2. Insert Adapter C.

3. Blank Preparation: Fill a round sample cell with 10 mL of sample.

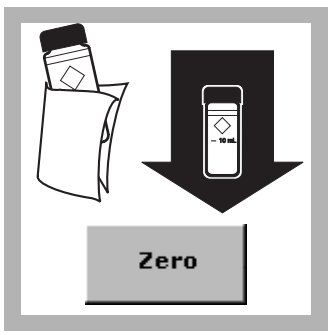


4. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill a Ferrous Iron AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely.

5. Quickly invert the Ampul several times to mix.

6. Press **TIMER>OK**.

A three-minute reaction period will begin.



7. When the timer expires, insert the blank into the cell holder.

Press **ZERO**.

The display will show:

0.00 mg/L Fe^{2+}

8. Insert the AccuVac Ampul into the cell holder.

Press **READ**. Results are in mg/L Fe^{2+}.

Sample Collection, Storage, and Preservation

Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection.

Accuracy Check

Standard Solution Method

1. Prepare a ferrous iron stock solution (100-mg/L Fe^{2+}) by dissolving 0.7022 grams of Ferrous Ammonium Sulfate, hexahydrate, in deionized water. Dilute to one liter in a Class A volumetric flask. In a 100-mL Class A volumetric flask, dilute 2.00 mL of this solution to 100 mL with deionized water to make a 2.0-mg/L standard solution. Prepare this solution immediately before use. Perform the iron procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The 1,10 phenanthroline indicator in the Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test. Test results are measured at 510 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Ferrous Iron Reagent Powder Pillows	1	100/pkg	1037-69
OR			
Ferrous Iron Reagent AccuVac® Ampuls	1	25/pkg	25140-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Balance, analytical	each	28014-01
Ferrous Ammonium Sulfate, hexahydrate, ACS	113 g	11256-14
Flask, volumetric, 1000 mL	each	14574-53
Pipet Bulb	each	14651-00
Pipet, volumetric, 2.00 mL	each	14515-35
Water, deionized	4 L	272-56

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Updated November 2005 Edition 1

Method 8147
Pour-Thru Cell

FerroZine® Rapid Liquid Method*
(0.009 to 1.400 mg/L Fe)

Scope and Application: For boiler, cooling, and natural waters[1]

[1] Adapted from Stookey, L.L., *Anal.Chem.,* 42 (7) 779 1970.

Test Preparation

Before starting the test:

If sample contains rust, see Interferences on page 3.
Digestion is required for total iron determination.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
If iron is present, a purple color will form after adding the reagent
Rinse glassware with a 1:1 HCl[1] solution. Rinse again with deionized water. This will remove residual iron that may interfere
Prepare the Pour-Thru cell by preparing a solution of 1 mL Ferrozine Reagent per 50 mL of deionized water. Pour this into the cell and allow to stand for approximately 5 minutes to react with any trace iron in the cell and cell tubing. Flush with iron free water.
FerroZine Iron Reagent may crystallize or precipitate when exposed to cold temperatures during shipment; reagent quality is not affected. Place the reagent in warm water to dissolve.

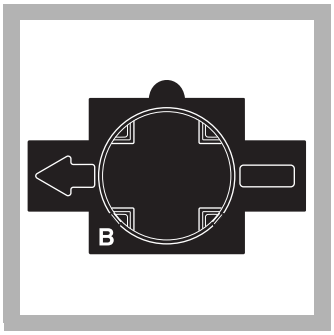
Collect the following items:	**Quantity**
FerroZine® Iron Reagent Solution	1.0 mL
Water, deionized	varies
Cylinder, graduated, 50-mL poly	1
Dispenser, fixed volume, 1.0-mL, Repipet Jr., with bottle	1
Flask, Erlenmeyer, PMP w/cap, 125-mL	2
Pour-Thru Cell Module	1

Note: *Reorder information for consumables and replacement items is on page 6.*

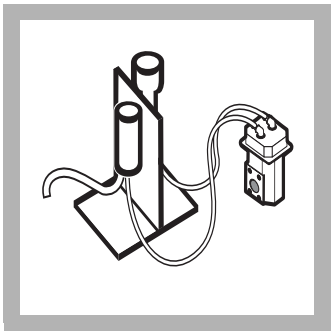
[1] See Optional Reagents and Apparatus on page 6.

Pour-Thru Cell — Method 8147

1. Select the test.

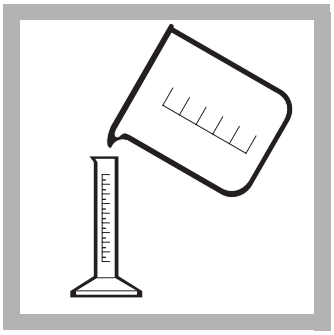


2. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch pathlength in line with the adapter arrow.

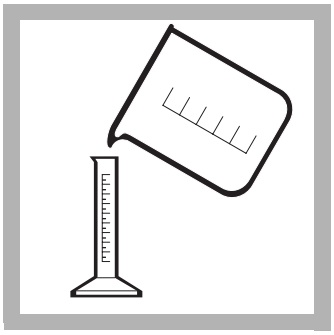


3. Flush the Pour-Thru Cell with 50 mL of deionized water.

4. Rinse two clean 125-mL Erlenmeyer flasks with the sample three times.

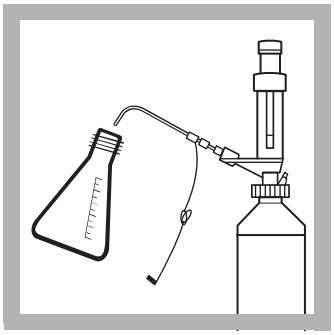


5. Rinse a clean 50-mL plastic graduated cylinder three times with the sample.



6. Fill the rinsed cylinder to the 50-mL mark with sample.

7. Prepared Sample: Pour the contents of the 50-mL cylinder into one of the flasks.



8. Add 1.0 mL of FerroZine Iron Reagent Solution to one of the flasks using the Repipet Dispenser. Swirl to mix.

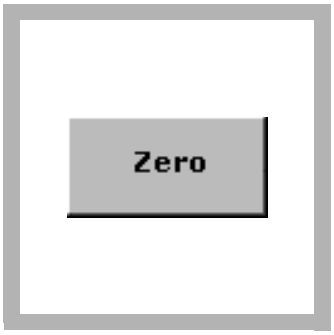
9. Blank Preparation: Measure a second 50 mL portion of sample into the graduated cylinder and pour the contents into the second flask.

10. Press **TIMER>OK**.

A five-minute reaction period will begin.

11. When the timer expires, the display will show: mg/L Fe

Pour the contents of the flask containing the blank into the Pour-Thru Cell.



12. When the flow stops, press **ZERO**.

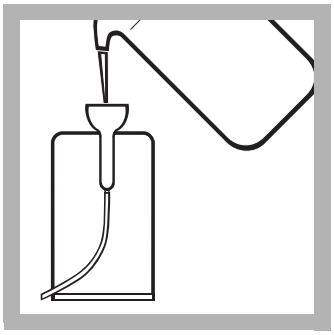
The display will show:

0.000 mg/L Fe

13. Pour the contents of the flask containing the prepared sample into the Pour-Thru Cell.

14. When the flow stops, press **READ**.

Results are in mg/L Fe.



15. Flush the Pour-Thru Cell with at least 50 mL of deionized water immediately after use.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels
Strong Chelants (EDTA)	Interfere at all levels. Use the FerroVer® or TPTZ methods for these samples. Use the TPTZ method for low iron concentrations.
Cobalt	May give slightly high results
Copper	May give slightly high results
Hydroxides	Boil the sample, with the FerroZine® Iron Reagent added to it from step 8, for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with step 8. Return the sample volume to 50 mL with deionized water.
Magnetite (black iron oxide) or Ferrites	**1.** Fill a 50-mL graduated cylinder with 50 mL of sample. **2.** Transfer the sample into a clean glass 125-mL Erlenmeyer flask. **3.** Add 1.0-mL of FerroZine Iron Reagent Solution[1] and swirl to mix. **4.** Place the flask on a hot plate or over a flame and bring to a boil. **5.** Continue boiling gently for 20 to 30 minutes. ***Note:*** *Do not allow to boil dry.* **6.** Return the boiled sample to the graduated cylinder. Rinse the Erlenmeyer flask with small amounts of deionized water and empty into the graduated cylinder. ***Note:*** *A purple color will develop if iron is present.* **7.** Return the sample volume to the 50-mL mark with deionized water. **8.** Pour the solution into a 125-mL Erlenmeyer flask and swirl to mix. **9.** Proceed with steps 8–13.
Rust	Boil the sample, with the FerroZine® Iron Reagent added to it from step 8, for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with step 8. Return the sample volume to 50 mL with deionized water.

[1] See Optional Reagents and Apparatus on page 6.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with concentrated Nitric Acid, ACS* (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only reporting dissolved iron, filter the sample immediately after collection and before adding nitric acid.

Before testing, adjust the sample pH to 3–5 with Ammonium Hydroxide, ACS*. Do not exceed pH 5, or iron may precipitate. Correct test results for volume additions.

Labware

All containers used in this test must be cleaned thoroughly to remove any traces of iron. Rinse labware and the Pour-Thru Cell with a 1:1 HCl solution* or with a 1:50 dilution of FerroZine® Reagent. Rinse several times with deionized water.

Keep flasks tightly closed when not in use. Dedicate these containers for iron analysis only. If containers are rinsed and capped after each use, only occasional treatment with HCl or FerroZine® is necessary.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of Ammonium Hydroxide*, followed by several rinses with deionized water. Cover the Pour-Thru Cell when it is not in use.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading the test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off an Iron Voluette® Ampule Standard, 25-mg/L Fe.
5. Use the TenSette® Pipet to add 0.2, 0.4, and 0.6 mL of standard to three 50-mL samples, respectively.
6. Analyze each sample as described above. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 6.

Standard Solutions Method
To check the accuracy, use a 1.0 mg/L Iron Standard Solution or prepare a 1.0 mg/L iron working solution as follows:

1. Pipet 5.00 mL of iron standard solution, 100-mg/L Fe, into a 500-mL volumetric flask.

2. Dilute to volume with deionized water. Prepare this solution daily. Analyze the working solution according to the above procedure.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The FerroZine® Iron Reagent forms a purple colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols, and with digestion can be used to analyze samples containing magnetite (black iron oxide) or ferrites. The test results are measured at 562 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
FerroZine® Iron Reagent Solution	1 mL	500 mL	2301-49
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 50-mL, poly	1	each	1081-41
Dispenser, fixed volume, 1.0-mL, Repipet Jr., with bottle	1	each	21113-02
Flask, Erlenmeyer, PMP w/cap, 125-mL	2	each	20898-43
Pour-Thru Cell Kit	1	each	59404-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Flask, volumetric, Class A, 500 mL	each	14574-49
Iron Standard Solution, 100-mg/L Fe	100 mL	14175-42
Iron Standard Solution, Voluette® ampule, 25-mg/L Fe, 10-mL	16/pkg	14253-10
Iron Standard Solution, 1 mg/L Fe	500 mL	139-49
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	28337-49
Pipet, TenSette 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00

Optional Reagents and Apparatus

Description	Cat. No.
Ammonium Hydroxide, ACS, 58%	106-49
Hydrochloric Acid, 1:1, 6N	884-49
Nitric Acid ACS, concentrated	152-49

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Updated November 2005 Edition 1

Iron, Total

Method 8112

TPTZ Method[1]

Powder Pillows or AccuVac® Ampuls

(0.012 to 1.800 mg/L)

Scope and Application: For water, wastewater, and seawater

[1] Adapted from G. Frederic Smith Chemical Co., *The Iron Reagents*, 3rd ed. (1980)

Test Preparation

Before starting the test:

Digestion is required for determining total iron.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Rinse all glassware with a 1:1 Hydrochloric Acid Solution[1]. Rinse again with deionized water. This process will remove iron deposits that can cause slightly high results.
After adding reagent, a blue color will develop if iron is present.
Adjust the pH of stored samples to 3–4. Do not exceed pH 5 or iron may precipitate.

Collect the following items:	**Quantity**
Powder Pillow Test:	
TPTZ Iron Reagent Powder Pillow	2
Sample Cells, 1-inch square, 10-mL	2
AccuVac Test:	
TPTZ Low Range Iron Reagent AccuVac®	1
Beaker, 50-mL	1
Sample Cell, 10-mL	1

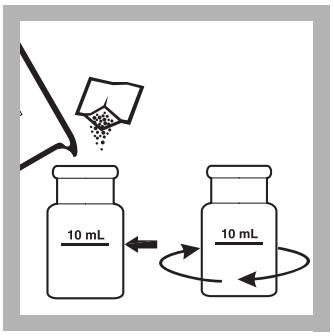
Note: *Reorder information for consumables and replacement items is on page 5.*

[1] See Optional Reagents and Apparatus on page 5.

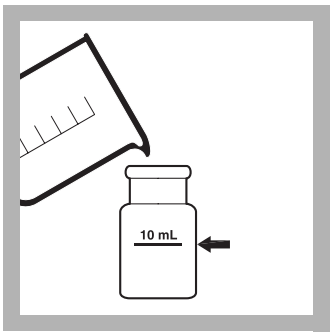
TPTZ Powder Pillows — Method 8112

1. Press **STORED PROGRAMS**.

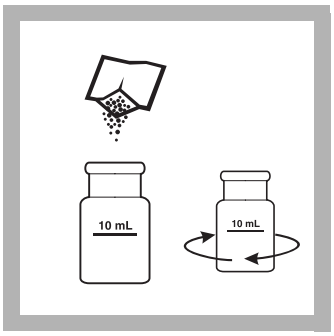
2. Select the test.



3. Prepared Sample: Fill one clean, square sample cell with 10 mL of sample.
Add the contents of one 10-mL TPTZ Iron Reagent Powder Pillow to the prepared sample. Swirl at least 30 seconds to dissolve.



4. Blank Preparation: Fill a second square sample cell with 10 mL of deionized water

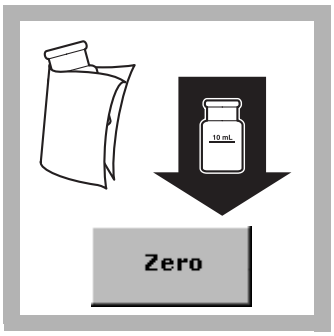


5. Add the contents of one 10-mL TPTZ Iron Reagent Powder Pillow to the reagent blank. Swirl at least 30 seconds to dissolve.

6. Press **TIMER>OK**.

A three-minute reaction period will begin.

Proceed to step 7 while the timer is running.

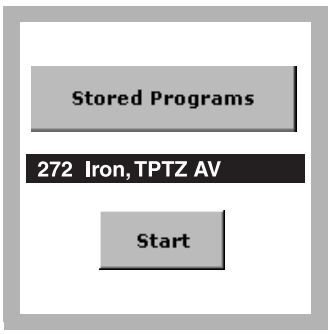


7. When the timer expires, insert the blank into the cell holder with the fill line facing right. Press **ZERO**.
The display will show:
0.000 mg/L Fe

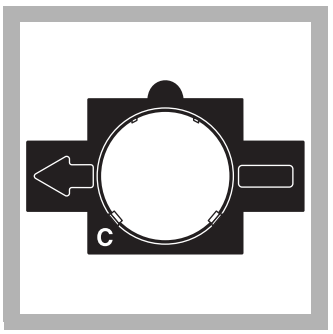
8. Insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Fe.

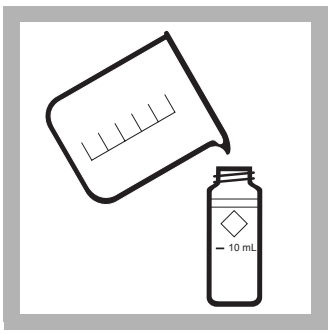
TPTZ AccuVac® Ampul — Method 8112



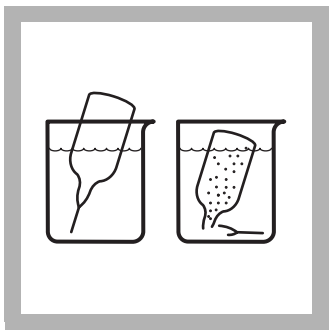
1. Select the test.



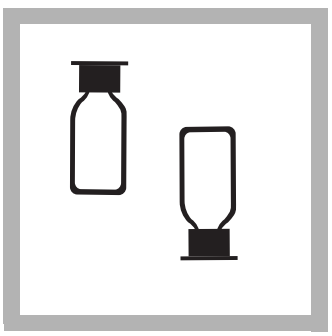
2. Insert Adapter C.



3. Blank Preparation: Fill a round sample cell with 10 mL of sample.



4. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill a TPTZ Iron AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely.

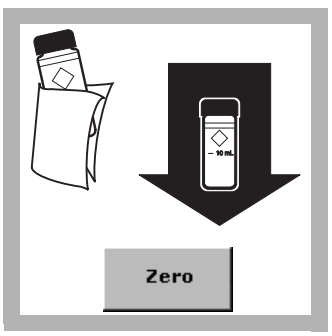


5. Invert the Ampul repeatedly to mix.

6. Press **TIMER>OK**.

A three-minute reaction period will begin.

Complete step 7 during this period.



7. When the timer expires, insert the blank into the adapter. Press **ZERO**.

The display will show:

0.000 mg/L Fe

8. Insert the prepared sample into the adapter.

Press **READ**. Results are in mg/L Fe.

Interferences

Interference tests (Table 1) were performed using an iron concentration of 0.5 mg/L. When interferences occurred, the color formation was inhibited or a precipitate formed. The following do not interfere with the test when present up to the levels given.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Cadmium	4.0 mg/L
Chromium ($^{3+}$)	0.25 mg/L
Chromium ($^{6+}$)	1.2 mg/L
Cobalt	0.05 mg/L
Copper	0.6 mg/L
Cyanide	2.8 mg/L
Manganese	50.0 mg/L
Mercury	0.4 mg/L
Molybdenum	4.0 mg/L
Nickel	1.0 mg/L
Nitrite Ion	0.8 mg/L
Color or turbidity	In the powder pillow procedure, if the sample, without a TPTZ Iron Reagent Powder Pillow, has a color or turbidity greater than the blank (deionized water plus TPTZ Iron Reagent), then use the sample as the blank.
pH	A sample pH of less than 3 or greater than 4 after the addition of reagent may inhibit color formation, cause the developed color to fade quickly, or to result in turbidity. Adjust the sample pH in the sample cell before the addition of reagent to between 3 and 4 by using a pH meter or pH paper and adding dropwise an appropriate amount of iron-free acid or base such as 1.0 N Sulfuric Acid Standard Solution[1] or 1.0 N Sodium Hydroxide Standard Solution[1]. Make a volume correction if significant volumes of acid or base are used.

[1] See Optional Reagents and Apparatus on page 5.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with about 2 mL/L Nitric Acid, ACS*. Store preserved samples up to six months at room temperature. If reporting only dissolved iron, filter sample immediately after collection and before adding nitric acid.

Before testing, adjust the pH of the stored sample to between 3–4 with 5.0 N Sodium Hydroxide Standard Solution*. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume additions.

* See Optional Reagents and Apparatus on page 5.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a fresh bottle of Iron Standard Solution, 10-mg/L Fe.

5. Prepare three sample spikes. Fill three sample cells with 10 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

Note: *For AccuVac® Ampuls, fill three mixing cylinders* each with 50-mL of sample and spike with 0.5 mL, 1.0 mL, and 1.5 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Using Class A glassware, prepare a 1.000-mg/L iron standard solution by pipetting 5.00 mL of Iron Standard Solution, 100-mg/L, into a 500-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the iron procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The TPTZ Iron Reagent forms a deep blue-purple color with ferrous iron. The indicator is combined with a reducing agent which coverts precipitated or suspended iron, such as rust, to the ferrous state. The amount of ferric iron present can be determined as the difference between the results of a ferrous iron test and the concentration of total iron. Test results are measured at 590 nm.

* See Optional Reagents and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
TPTZ Iron Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	26087-99
OR			
TPTZ Low Range Iron Reagent AccuVac® Ampuls	1	25/pkg	25100-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Iron Standard Solution, 100-mg/L Fe	100 mL	14175-42
Iron Standard Solution, 10-mg/L Fe	500 mL	140-49
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	28337-49
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	28336-49
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing, 50 mL	1896-41
Nitric Acid, concentrated	152-49
Sodium Hydroxide, 5.0 N	2450-26
Sodium Hydroxide Standard Solution, 1.0 N	1270-32
Sulfuric Acid, 1.0 N	1045-32

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Updated November 2005 Edition 1

Iron, Total

Method 8365 **FerroMo Method[1]**

Powder Pillows **(0.01 to 1.80 mg/L)**

Scope and Application: For cooling water containing molybdate-based treatment

[1] Adapted from G. Frederick Smith Chemical Co., *The Iron Reagents*, 3rd ed. (1980)

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Rinse glassware with a 1:1 hydrochloric acid solution. Rinse again with deionized water. These two steps will remove iron deposits that can cause slightly high results.
After the addition of the reagent, the sample pH should be between 3–5.
If the sample contains high levels of molybdate (100 mg/L MoO_4^{2-} or greater), read the sample immediately after zeroing the blank.

Collect the following items:	**Quantity**
FerroMo® Reagent 1 Powder Pillow	1
FerroMo® Reagent 2 Powder Pillow	1
Cylinder, graduated mixing, 25-mL, with stopper	1
Cylinder, graduated mixing, 50-mL, with stopper	1
Sample Cells, 1-inch square, 10 mL, matched pair	2

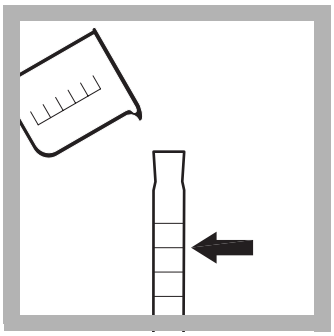
Note: *Reorder information for consumables and replacement items is on page 4.*

Note: *Digestion is required for total iron determination.*

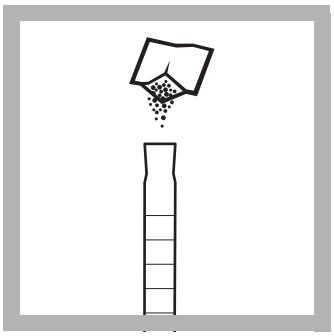
Powder Pillows Method 8365

1. Press **STORED PROGRAMS**.

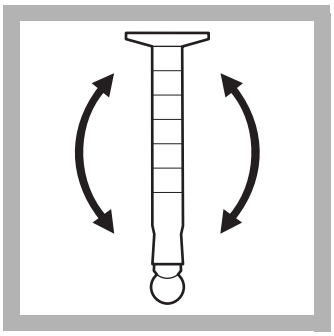
2. Select the test.



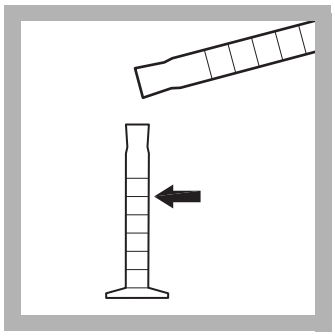
3. Prepared Sample: Fill a 50-mL graduated mixing cylinder with 50 mL of sample.



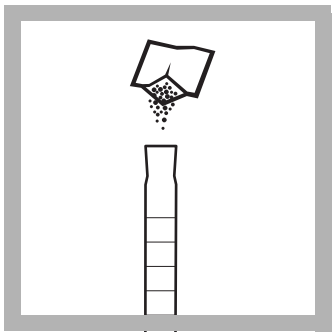
4. Add the contents of one FerroMo Iron Reagent 1 Powder Pillow to the graduated mixing cylinder. Stopper.



5. Invert several times to dissolve the reagents.



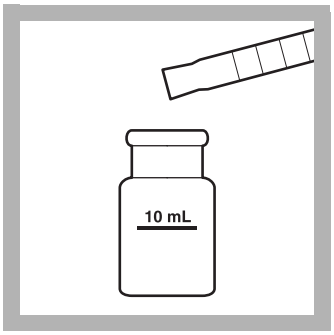
6. Fill a clean, 25-mL graduated cylinder to the 25-mL mark with prepared sample. Save the remaining prepared sample for step 10.



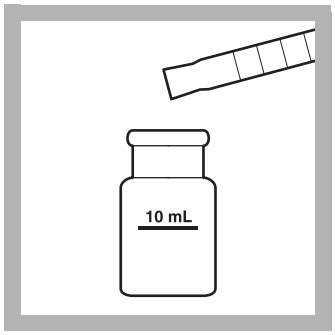
7. Developed Sample: Add the contents of one FerroMo Iron Reagent 2 Powder Pillow to the sample in the 25-mL mixing cylinder. Stopper and invert to dissolve the reagents. A blue color will develop if iron is present.
Note: *A small amount of undissolved reagent will not affect the results.*

8. Press **TIMER>OK**.

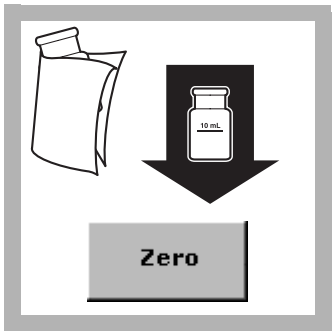
A three-minute reaction period will begin.



9. When the timer expires, pour the developed sample from step 7 into a square sample cell.

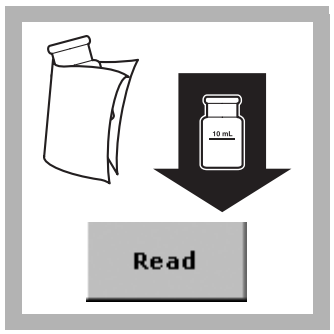


10. Blank Preparation: Fill a second square sample cell with the remaining prepared sample from step 6.



11. When the timer expires, insert the blank into the cell holder with the fill line facing right. Press **ZERO**.

The display will show: 0.00 mg/L Fe



12. Insert the developed sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Fe.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
pH	A sample pH of less than 3 or greater than 4 after the addition of reagent may inhibit color formation, cause the developed color to fade quickly, or result in turbidity. Adjust the sample pH in the graduated cylinder before the addition of reagent to between 3 and 8 by using a pH meter or pH paper and adding, dropwise, an appropriate amount of iron-free acid or base such as 1.0 N Sulfuric Acid Standard Solution[1] or 1.0 N Sodium Hydroxide Standard Solution[1]. Make a volume correction if significant volumes of acid or base are used.

[1] See Optional Reagents and Apparatus on page 4.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with hydrochloric acid (about 2 mL per liter)*. Samples preserved in this manner can be stored up to six months at room temperature. If only dissolved iron is to be reported, filter sample immediately after collection through a 0.45-micron filter or equivalent medium before adding hydrochloric acid.

Before testing, adjust the sample pH to 3–5 with 5.0 N Sodium Hydroxide Standard Solution*. Do not exceed pH 5 as iron may precipitate. Correct test results for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open an Iron Voluette® Ampule Standard, 50-mg/L Fe.

5. Prepare three sample spikes. Fill three mixing cylinders* with 50 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. The iron concentration should increase by 0.10 mg/L. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Standard Solution Method

1. Using Class A glassware, prepare a 1.00-mg/L iron standard solution by pipetting 10.0 mL of Iron Standard Solution, 100-mg/L, into a 1-liter volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the iron procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

* See Optional Reagents and Apparatus on page 4.

FerroMo Iron Reagent 1 contains a reducing agent combined with a masking agent. The masking agent eliminates interference from high levels of molybdate. The reducing agent converts precipitated or suspended iron, such as rust, to the ferrous state. FerroMo Iron Reagent 2 contains the indicator combined with a buffering agent. The indicator reacts with ferrous iron in the sample, buffered between pH 3 and 5, resulting in a deep blue-purple color. Test results are measured at 590 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
FerroMo® Iron Reagent Set (100 tests), includes:	—	—	25448-00
(4) FerroMo® Reagent 1 Powder Pillows	1	25/pkg	25437-68
(2) FerroMo® Reagent 2 Powder Pillows	1	50/pkg	25436-66

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated mixing, 25-mL, with stopper	1	each	20886-40
Cylinder, graduated mixing, 50-mL, with stopper	1	each	20886-41
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Iron Standard Solution, 100-mg/L	100 mL	14175-42
Iron Standard Solution, 10-mL Voluette® ampule, 50-mg/L Fe.	16/pkg	14254-10
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing	20886-41
Flask, volumetric, Class A	14574-53
Pipet, volumetric, Class A, 10.0 mL	14515-38
Pipet Filler, safety bulb	14651-00
Sodium Hydroxide Standard Solution, 1.0 N	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	2450-32
Sulfuric Acid Standard Solution,1.0 N	1270-32
Sodium Hydroxide Standard Solution, 5.0 N	2450-32

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Updated November 2005 Edition 1

Iron, Total

★Method 8008 — FerroVer® Method[1]

Powder Pillows or AccuVac® Ampuls — (0.02 to 3.00 mg/L)

Scope and Application: For water, wastewater, and seawater; digestion is required for determining total iron; USEPA approved for reporting wastewater analysis[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*
[2] *Federal Register*, June 27, 1980; 45 (126:43459)

Test Preparation

Before starting the test:

Digestion is required for determining total iron for EPA reporting purposes.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the user manual for more information.

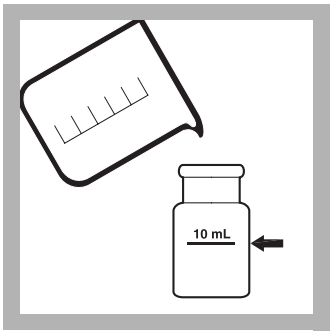
Collect the following items:	**Quantity**
Powder Pillow Test:	
FerroVer® Iron Reagent Powder Pillow	1
Sample Cells,1-inch square, 10 mL	2
Beaker, 50-mL	1
FerroVer® Iron Reagent AccuVac® Ampul	
Beaker, 50-mL	1
Sample Cells, 10-mL, with cap	1

Note: *Reorder information for consumables and replacement items is on page 6.*

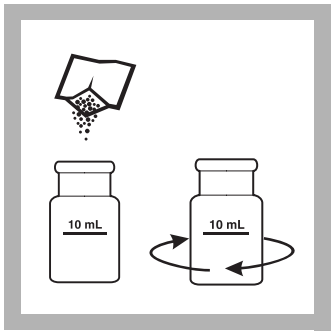
Powder Pillows — Method 8008

1. Press **STORED PROGRAMS**.

2. Select the test.



3. Prepared Sample: Fill a clean square sample cell with 10 mL of sample.



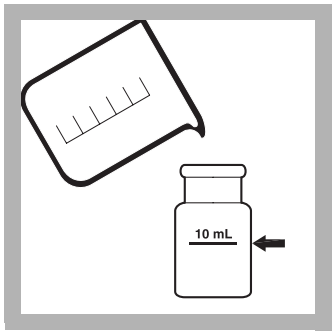
4. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell. Swirl to mix.

An orange color will form, if iron is present

5. Press **TIMER**>**OK**.

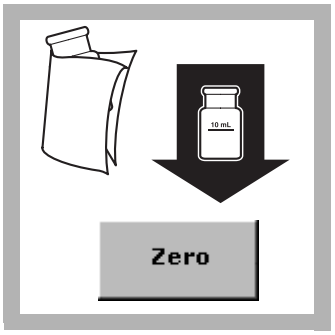
A three-minute reaction period will begin.

(Allow samples that contain rust to react for at least 5 minutes.)



6. Blank Preparation:

Fill a second square sample cell with 10 mL of sample.

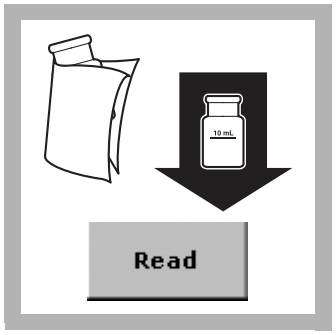


7. When the timer expires, insert the blank into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

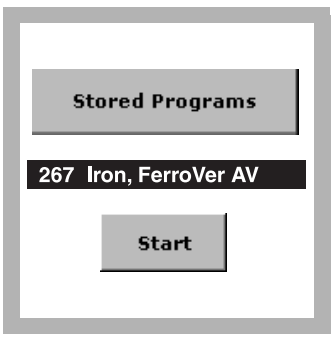
0.00 mg/L Fe



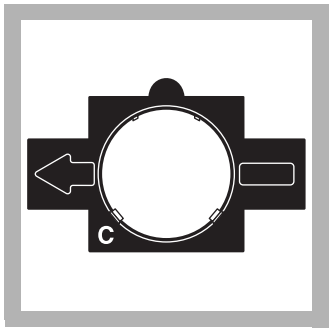
8. Place the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Fe.

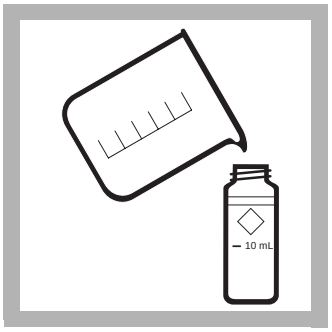
AccuVac Ampul — Method 8008



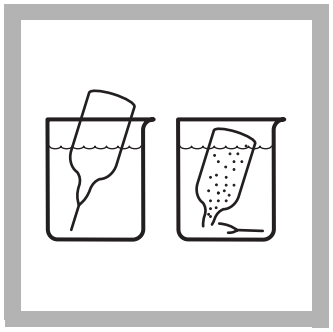
1. Select the test.



2. Insert Adapter C.



3. Blank Preparation: Fill a round sample cell with 10 mL of sample.



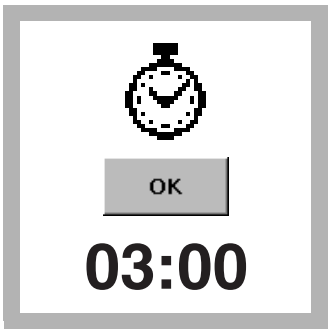
4. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a FerroVer Iron AccuVac® Ampul with sample.

Keep the tip immersed while the Ampul fills completely.

5. Quickly invert the Ampul several times to mix.

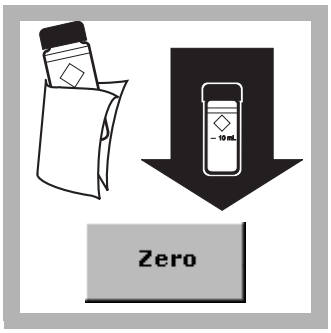
Wipe off all liquid and fingerprints.



6. Press **TIMER>OK**.

A three-minute reaction period will begin.

(Allow samples that contain rust to react for at least 5 minutes.)



7. When the timer expires, insert the blank into the cell holder.

Press **ZERO**.

The display will show:

0.00 mg/L Fe

8. Insert the AccuVac Ampul into the cell holder.

Press **READ**. Results are in mg/L Fe.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium, Ca^{2+}	No effect at less than 10,000 mg/L as $CaCO_3$.
Chloride, Cl^-	No effect at less than 185,000 mg/L.
Copper, Cu^{2+}	No effect. Masking agent is contained in FerroVer Reagent.
High Iron Levels	Inhibit color development. Dilute sample and re-test to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion. After digestion, adjust sample to pH 3–5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as calcium carbonate.
Molybdate Molybdenum	No effect at 50 mg/L as Mo.
High Sulfide Levels, S^{2-}	**1.** Treat in fume hood or well-ventilated area. Add 5 mL hydrochloric acid[1], ACS to 100 mL sample in a 250-mL Erlenmeyer flask. Boil 20 minutes. **2.** Cool. Adjust pH to 3–5 with Sodium Hydroxide[1]. Readjust volume to 100 mL with deionized water. **3.** Analyze.
Turbidity	**1.** Add 0.1 g scoop of RoVer® Rust Remover to the blank. Swirl to mix. **2.** Zero the instrument with this blank. **3.** If sample remains turbid, add three 0.2 g scoops of RoVer to a 75-mL sample. Let stand 5 minutes. **4.** Filter through a Glass Membrane Filter and Filter Holder[1]. **5.** Use filtered sample in steps 6 and 3.
Extreme Sample pH	Adjust pH to 3–5.
Highly Buffered Samples	Adjust pH to 3–5.

[1] See Optional Reagents and Apparatus on page 6.

Sample Collection, Storage, and Preservation

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with concentrated nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Before analysis, adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions.

If only dissolved iron is to be determined, filter the sample before acid addition.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off an Iron Voluette Ampule Standard, 25-mg/L.
5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to the unspiked sample. Press the timer icon. After the timer expires, read the result.
6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Press the timer icon. After the timer expires, read the result.
7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Press the timer icon. After the timer expires, read the result. Each addition should reflect approximately 100% recovery.

Note: *For AccuVac® Ampuls, fill three mixing cylinders* with 50-mL of sample and spike with 0.2 mL, 0.4 mL, and 0.6 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers*. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 6.

Standard Solution Method

1. Prepare a 2.00-mg/L Fe standard solution by pipetting 2.00 mL of Iron Standard Solution, 100-mg/L, into a 100-mL volumetric flask. Dilute to the mark with deionized water. Stopper and invert to mix. Prepare this solution daily. Perform the iron procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

FerroVer Iron Reagent converts all soluble iron and most insoluble forms of iron in the sample to soluble ferrous iron. The ferrous iron reacts with the 1,10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration. Test results are measured at 510 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
FerroVer® Iron Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	21057-69
OR			
FerroVer® Iron Reagent AccuVac® Ampuls	1	25/pkg	25070-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVacs)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cells, 10-mL, with cap	1	each	21228-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Iron Standard Solution, 100-mg/L	100 mL	14175-42
Iron Standard Solution, 10-mL Voluette® Ampule, 25-mg/L as Fe	16/pkg	14253-10
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	28337-49
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	28336-49
Water, deionized	4 L	272-56
Pipet, TenSette, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00

Optional Reagents and Apparatus

Description	Cat. No.
Beaker, 50-mL	500-41H
Cylinder, mixing	1896-41
Hydrochloric Acid, concentrated	134-49
Nitric Acid, concentrated	152-49
Sodium Hydroxide Standard Solution, 5.0 N	2450-32
Glass Membrane Filter	2530-00
Glass Membrane Filter Holder	2340-00

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Updated November 2005 Edition 1

Method 8317

LeadTrak®[1] Fast Column Extraction Method (5 to 150 µg/L)

Scope and Application: For drinking water

[1] Patent Number 5,019,516

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
The sampling requirements for “first-draw” analysis are detailed in Sample Collection, Storage, and Preservation on page 5.
Reagents will stain the sample cells, rinse the cells with 1:1 HNO3, followed by deionized water.

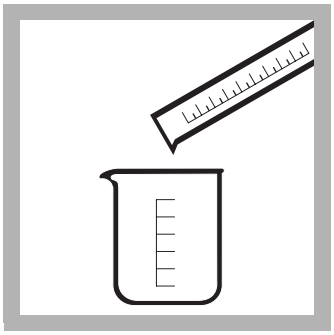
Collect the following items:	Quantity
LeadTrak® Reagent Set	1
Beaker, polypropylene, 150-mL	2
Beaker, polypropylene, 250-mL	1
Clamp, 2-prong extension, with clamp holder	1
Cylinder, graduated polypropylene, 25-mL	1
Cylinder, graduated polypropylene, 100-mL	1
Dropper, 0.5 and 1.0 mL marks	1
Sample Cells, 1-inch square	1
Support for Ring Stand	1

Note: *Reorder information for consumables and replacement items is on page 8.*

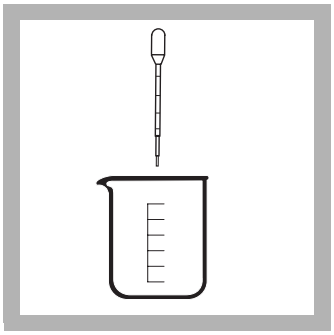
Fast Column Extraction — Method 8317

1. Press **STORED PROGRAMS**.

2. Select the test.



3. Fill a 100-mL plastic graduated cylinder with 100 mL of the sample. Pour the measured sample into a 250-mL plastic beaker.

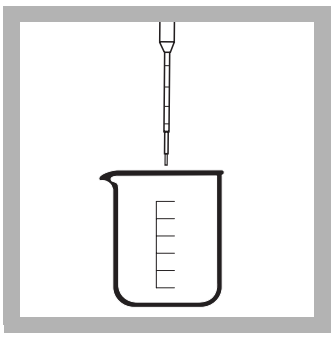


4. Using a plastic 1-mL dropper, add 1.0 mL of pPb-1 Acid Preservative Solution to the sample and swirl to mix.

If the sample has been preserved previously with pPb-1 Acid Preservative at a ratio of 1.0 mL per 100 mL sample, omit steps 4 and 5.

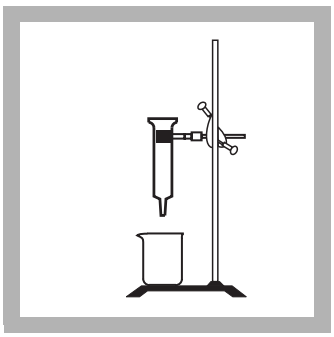
5. Press **TIMER>OK**.

A two-minute reaction period will begin.



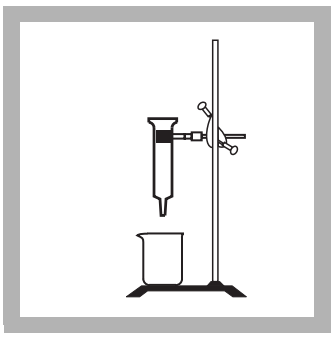
6. When the timer expires, use a second 1-mL plastic dropper to add 2.0 mL of pPb-2 Fixer Solution. Swirl to mix.

Field samples that have been preserved with nitric acid or samples that have been digested may exceed the buffer capacity of the Fixer Solution. After step 6, check the pH of these samples and adjust with 5 N Sodium Hydroxide to a pH of 6.7–7.1 before proceeding with step 7.



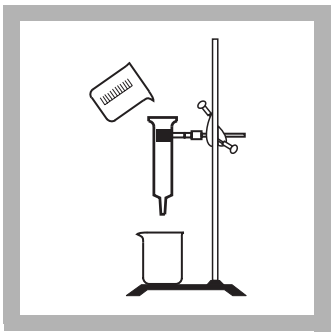
7. Mount a new Fast Column Extractor in a ring stand with a clamp. Place a 150-mL plastic beaker under the Extractor.

A Fast Column Extractor is included in the LeadTrak® Reagent Set. A new extractor is required for each test.



8. Soak the cotton plug with deionized water and compress it with the plunger. Remove the plunger. If the cotton plug moves up the column, push it back to the bottom with a clean, blunt rod.

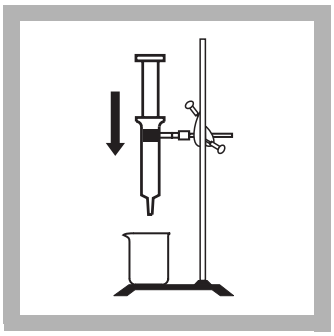
The cotton plug should fit snugly against the inner wall of the column.



9. Pour the prepared sample slowly into the center of the Column Extractor. Wait for the sample to flow through.

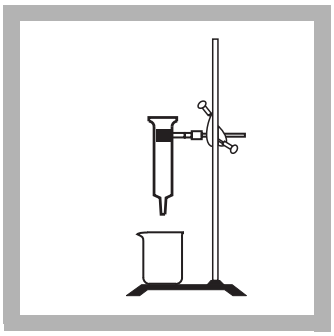
The sample solution should flow relatively slowly (2 drops per second) through the column.

Keep the level of the sample solution just above the cotton plug.



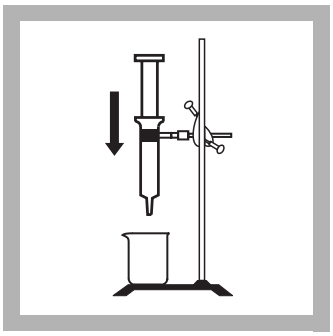
10. After the flow has stopped, fully compress the absorbent pad in the Extractor with the plunger. Discard the contents of the beaker. Slowly withdraw the plunger from the Extractor.

The absorbent pad should remain at the bottom of the Extractor when the plunger is removed. If the cotton plug moves up the column, push it back to the bottom with a clean, blunt rod.



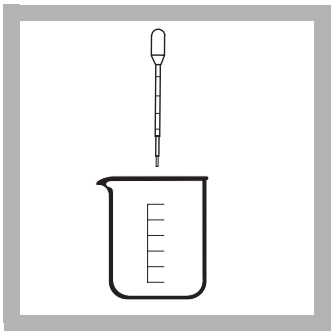
11. Place a clean, dry 150-mL beaker under the Extractor. Using a 25-mL plastic graduated cylinder, add 25 mL of pPb-3 Eluant Solution to the Extractor.

Keep the level of the eluent solution just above the absorbent pad.

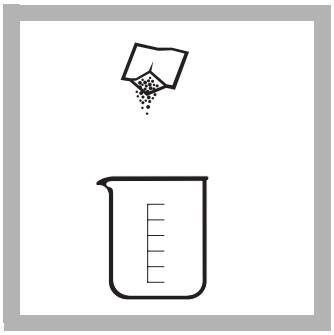


12. Allow the Eluant Solution to drip slowly from the Extractor.

After the flow has stopped, fully compress the absorbent pad.

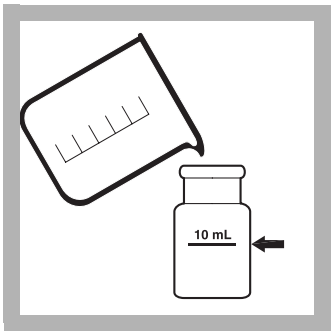


13. Using a 1-mL plastic dropper, add 1.0 mL of pPb-4 Neutralizer Solution to the beaker. Swirl thoroughly to mix and proceed immediately to step 14.



14. Add the contents of one pPb-5 Indicator Powder Pillow to the beaker and swirl thoroughly to mix.

The solution will turn brown.

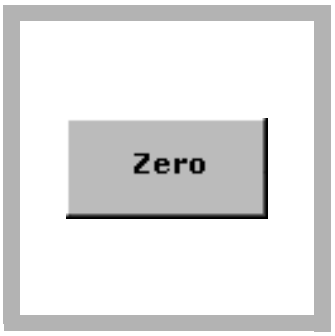


15. Pour 10 mL of solution into a square sample cell.

16. Press **TIMER>OK**.

A second two-minute reaction period will begin.

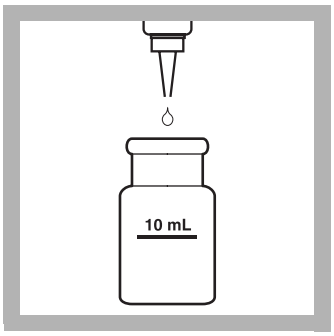
17. When the timer expires, insert the sample cell into the cell holder with the fill line facing right.



18. Press **ZERO**.

The display will show:

0 µg/L Pb



19. Remove the sample cell and add 3 drops of pPb-6 Decolorizer Solution to the cell. Swirl to mix thoroughly.

20. Insert the sample cell into the cell holder with the fill line facing right.

Press **READ**. Results are in µg/L Pb.

Interferences

Interference studies were conducted by preparing a known lead solution of approximately 25 µg/L as well as the potential interfering ion. The ion was said to interfere when the resulting lead concentration changed by ±10%. Samples containing levels exceeding these concentration values may be diluted 1:1 and re-analyzed. Multiply the value obtained by a factor of 2 to determine the lead present in the original sample.

Every effort has been made to prevent contamination in packaging the reagents. Use of black rubber stoppers, black dropper bulbs and droppers with inked graduations may contaminate the sample and should be avoided. Use the plastic droppers provided in the reagent set.

Acid-wash all glassware and plasticware to prevent sample contamination, especially if the previous sample had a high lead level (see Apparatus and Sample Preparation on page 5).

The Extractor plunger may be reused for more than one test but should be rinsed with lead-free water between uses.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum, Al^{3+}	0.5 mg/L
Ammonium, NH_4^+	500 mg/L
Barium, Ba^{2+}	6 mg/L
Calcium, Ca^{2+}	500 mg/L
Chloride, Cl^-	1000 mg/L
Copper, Cu^{2+}	2 mg/L
Fluoride, F^-	10 mg/L
Iron, Fe^{2+}	2 mg/L
Magnesium, Mg^{2+}	500 mg/L
Manganese, Mn^{2+}	0.5 mg/L
Nitrate, NO_3^-	1000 mg/L
Sulfate, SO_4^{2-}	1000 mg/L
Zinc, Zn^{2+}	1 mg/L

Apparatus and Sample Preparation

Because lead is very common to our environment, care must be taken to prevent sample contamination. Follow these steps for greatest test accuracy:

- Lead-free water is necessary to minimize sample contamination when rinsing apparatus or diluting sample. The water may be either distilled or deionized. If the water is obtained from a grocery store, verify the lead concentration is zero from the label. If the lead concentration is uncertain, determine the lead concentration with the LeadTrak test.
- Plastic or glass sample containers and lids may be checked for contamination by rinsing with 1 mL of pPb-1 Acid Preservative Reagent*. Add 100 mL of lead-free water. After 24 hours, analyze this solution using the LeadTrak® test to confirm the absence of lead.
- Rinse glassware used in this test with a small amount of dilute lead-free 0.1 N nitric acid or pPb-1 Acid Preservative Reagent followed by rinsing with lead-free water.
- pPb-5 Indicator may be rinsed from the glass sample cells with a few drops of pPb-1 Acid Preservative Reagent or a small amount of dilute lead-free nitric acid.
- Acidify solutions containing lead with Nitric Acid or pPb-1 to below pH 2 to prevent adsorption of lead onto the container walls. See Sample Collection, Storage, and Preservation.

Sample Collection, Storage, and Preservation

Samples may be collected either from household pipes (point-of-use) or from water sources. Preserved samples may be stored up to six months. Each sample type typically requires different sampling procedures. Consult with the appropriate regulatory agency in your area for more information about your specific sampling requirements.

Sampling for Lead Contamination in Household Pipes for Point-of-Use Drinking Water

- The sample should be collected after sitting in pipes with no flow for a minimum of six hours.
- Add 10 mL of pPb-1 Acid Preservative* to a one-liter bottle.
- Turn on tap and collect exactly the first liter of water in the bottle containing acid preservative.
- Cap and invert several times to mix.
- After two minutes the sample is ready for analysis. Steps 4 and 5 are skipped in the analysis procedure. Use 100 mL of this preserved sample directly in step 6.

Sampling for Lead Contamination from Drinking Water Sources Such as Well Water or Water from Main Supply Lines

- Add 10 mL of pPb-1 Acid Preservative* to a one-liter bottle.
- Turn on the tap for 3–5 minutes or until the water temperature has been stable for 3 minutes.
- Collect exactly one liter of water into the bottle containing the acid preservative.

* See Optional Reagents and Apparatus on page 8.

- Cap and invert several times to mix.
- After two minutes the sample is ready for analysis. Steps 4 and 5 are skipped in the analysis procedure. Use 100 mL of this preserved sample directly in step 6.
- At least one liter should be collected to obtain a representative sample. If less than one liter is collected, use 1 mL of pPb-1 Acid Preservative per 100 mL of sample.
- If nitric acid is to be substituted for pPb-1 as a preservative or the sample is digested, the buffering capacity of the pPb-2 Fixer Solution* may be exceeded. Adjust the sample pH to 6.7–7.1 pH with 5 N Sodium Hydroxide* after step 7.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open a container of 10-mg/L (10,000 µg/L) Lead Standard Solution.
5. Prepare three sample spikes. Fill three beakers with 100 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

*See Optional Reagents and Apparatus on page 8.

Standard Solution Method
Using Class A glassware, prepare a 10-mg/L lead working standard solution by pipetting 1.0 mL of Lead Standard Solution, 1000-mg/L, into a 100-mL volumetric flask. Use a TenSette® Pipet to add 0.2 mL of concentrated nitric acid to the flask. Dilute to the mark with lead-free deionized water. This makes a 10-mg/L working standard.

Pipet 10.00 mL of this working solution into a 1-liter plastic volumetric flask. Dilute to the mark with lead-free water. This 100-µg/L standard solution should be prepared immediately before use. Perform the LeadTrak® procedure as described above.

Alternatively, prepare a 100-µg/L lead standard solution by using a TenSette® Pipet to pipet 0.2 mL from a Lead Voluette® Ampule Standard Solution, 50-mg/L as Pb, into a 100-mL plastic volumetric flask. Dilute to volume with deionized water. Prepare this solution immediately before use.

1. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

2. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Acid soluble lead, as Pb^{2+}, in a potable water sample is first concentrated on a Fast Column Extractor. The lead is then eluted from the Extractor and determined colorimetrically with an indicator. Test results are measured at 477 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
LeadTrak® Reagent Set	1	20/pkg	23750-00

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Beaker, polypropylene, 150-mL	2	each	1080-44
Beaker, polypropylene, 250-mL	1	each	1080-46
Clamp, 2-prong extension	1	each	21145-00
Clamp Holder	1	each	326-00
Cylinder, graduated polypropylene, 25-mL	1	each	1081-40
Cylinder, graduated polypropylene, 100-mL	1	each	1081-42
Dropper, 0.5 and 1.0 mL marks	1	2/pkg	21247-20
Sample Cells, 1-inch square, 10-mL	1	2/pkg	24954-02
Support for Ring Stand	1	each	563-00

Digestion Reagents and Recommended Standards and Apparatus

Description	Unit	Cat. No.
Flask, volumetric, polypropylene, 1000 mL	each	20995-53
Flask, volumetric, polypropylene, 100 mL	each	20995-42
Lead Standard Solution, 1000-mg/L as Pb	100 mL	12796-42
Lead Standard Solution, 50-mg/L 10-mL Voluette® Ampules	16/pkg	14262-10
Lead Standard Solution, 10-mg/L	25 mL	23748-20
Nitric Acid, ACS	500 mL	152-49
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, 10.00 mL	each	14515-38
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
pPb-1 Acid Preservative Reagent	23685-31
pPb-2 Fixer Solution	23686-55
Sodium Hydroxide, 5.0 N	2450-53

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Updated November 2005 Edition 1

Lead

★Method 8033 — Dithizone Method[1]

Powder Pillows — (3 to 300 µg/L)

Scope and Application: For water and wastewater; USEPA accepted for reporting for wastewater analysis (digestion is required).[2]

[1] Adapted from Snyder, L. J., Analytical Chemistry, 19 684 (1947).

[2] Procedure is equivalent to Standard Method 3500-Pb D for wastewater analysis.

Test Preparation

Before starting the test:

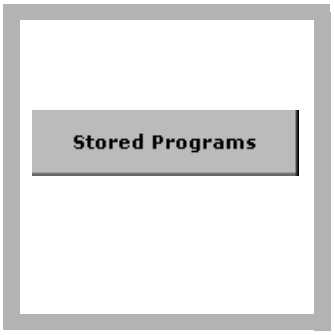
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.
Clean all glassware with a 1:1 Nitric Acid Solution. Rinse with deionized water.
Cloudy and turbid samples may require filtering before running the test. Report results as µg/L soluble lead. Use glass membrane type filter to avoid loss of lead by adsorption onto the filter paper.
If samples cannot be analyzed immediately, see Sample Collection, Preservation, and Storage on page 5. Adjust the pH of preserved samples before analysis.
For more accurate results, adjust the sample to pH 11.0–11.5 using a pH meter in step 10. Omit the five additional drops of Sodium Hydroxide Standard Solution in step 11
The DithiVer powder will not completely dissolve in the chloroform. For further notes see DithiVer Solution Preparation, Storage, and Reagent Blank on page 5.
Read the MSDS before testing. Spilled reagent will affect test accuracy and is hazardous to skin and other materials.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

Collect the following items:	**Quantity**
Citrate Buffer Powder Pillows	1
Chloroform	500 mL
DithiVer Metals Reagent Powder Pillows	1
Lead Reagent Set	1
Potassium Cyanide	2 g
Sodium Hydroxide solution, 5.0 N	5 mL
Sodium Hydroxide Standard Solution, 5.0 N	varies
Cotton Balls	1
Clippers	1
Cylinder, 50-mL graduated mixing	1
Cylinder, 5-mL graduated	1
Cylinder, 50-mL graduated	1
Cylinder, 250-mL graduated	1
Funnel, 500-mL separatory	1
Sample Cells, 1-inch square, 25-mL	2
Spoon, measuring, 1.0-g	1
Support Ring (4-inch) and Stand (5 x 8-inch base)	1

Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows — Method 8033

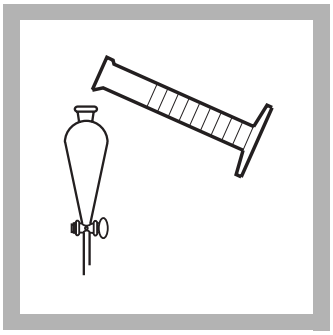
DANGER
Cyanide is a deadly poison. Use a fume hood. Maintain cyanide solutions at pH 11 or greater to prevent formation of cyanide gas.



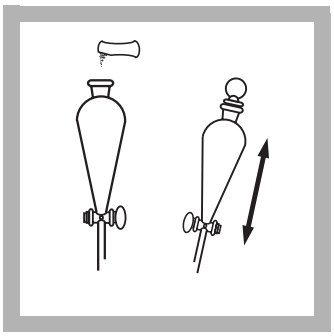
1. Press **STORED PROGRAMS**.

2. Select the test.

3. Fill a 250-mL graduated cylinder to the 250-mL mark with sample.

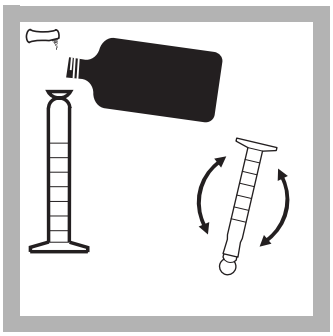


4. Transfer the sample into 500-mL separatory funnel.



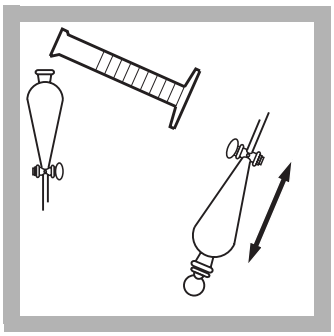
5. Add the contents of one Buffer Powder Pillow for heavy metals, citrate type.

Stopper the funnel and shake to dissolve.



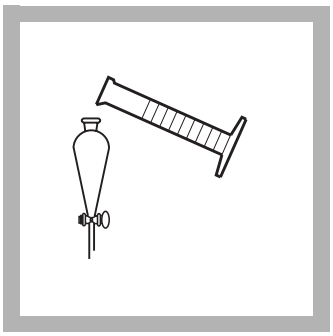
6. DithiVer Solution Preparation:
Add 50 mL of chloroform to a 50-mL mixing graduated cylinder. Add the contents of one DithiVer Metals Reagent Powder Pillow.

Stopper the cylinder. Invert several times to mix.

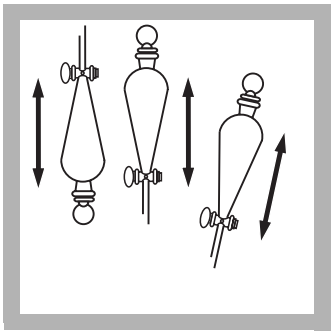


7. Measure 30 mL of the prepared dithizone solution with a second graduated cylinder and add to the separatory funnel.

Stopper and invert to mix. Open stopcock to vent. Close the stopcock.

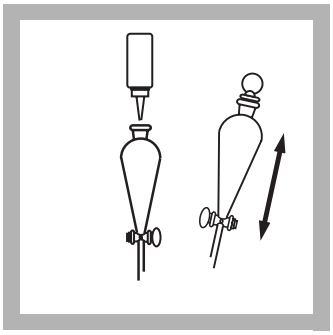


8. Add 5 mL of 5.0 N Sodium Hydroxide Standard Solution.



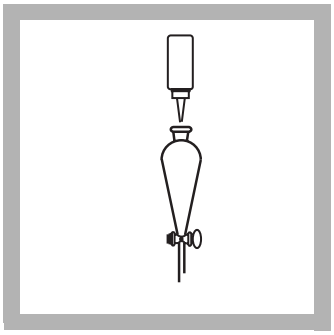
9. Stopper. Invert. Open stopcock to vent. Close the stopcock and shake the funnel once or twice and vent again.

Note: *Add a few drops of 5.25 N Sulfuric Acid Standard Solution if the solution turns orange on shaking. The blue-green color will reappear. To avoid higher blanks, repeat procedure on new sample and use less sodium hydroxide in step 8.*



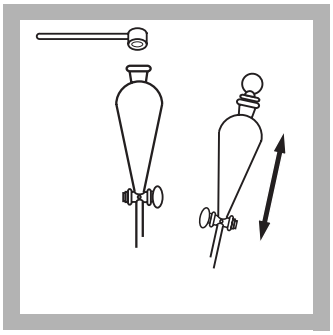
10. Continue adding 5.0 N Sodium Hydroxide Standard Solution dropwise and shaking the funnel after every few drops until the color of the solution being shaken changes from blue-green to orange.

Large amounts of zinc cause the color transition at the end point to be indistinct.



11. Add 5 more drops of 5.0 N Sodium Hydroxide Standard Solution.

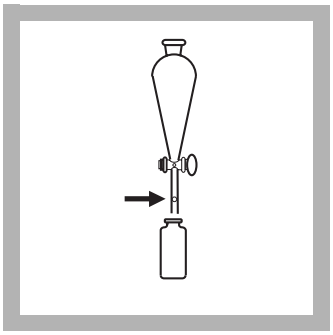
A pink color in the bottom (chloroform) layer at this point does not necessarily indicate lead is present. Only after adding the potassium cyanide in the next step will the presence of lead be confirmed by a pink color.



12. Add 2 heaping 1.0-g scoops of potassium cyanide to the funnel. Stopper.

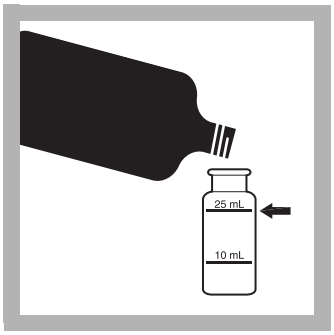
Shake vigorously until the potassium cyanide is all dissolved (about 15 seconds).

13. Wait one minute for the layers to separate. The bottom (chloroform) layer will be pink if lead is present.



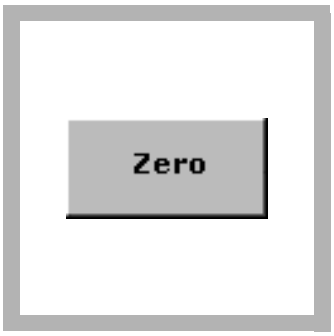
14. Prepared Sample: Insert a cotton plug the size of a pea into the delivery tube of the funnel and slowly drain the bottom (chloroform) layer into a dry 25-mL square sample cell. Stopper.

The lead-dithizone complex is stable for at least thirty minutes if the sample cell is kept tightly capped and out of direct sunlight.



15. Blank Preparation: Fill another 25-mL square sample cell with chloroform. Stopper.

16. Insert the blank into the cell holder with the fill line facing right.



17. Press **ZERO**.

The display will show:

0 µg/L Pb^{2+}

18. Insert the prepared sample into the cell holder with the fill line facing right.

19. Press **READ**.

Results are in µg/L Pb^{2+}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Highly buffered samples or extreme sample pH	All levels. See procedure below.
Bismuth	All levels. See procedure below.
Copper	All levels. See procedure below.
Mercury	All levels. See procedure below.
Silver	All levels. See procedure below.
Tin	All levels. See procedure below.

Table 2 Substances That Do Not Interfere

Aluminum	Lead
Antimony	Magnesium
Arsenic	Manganese
Calcium	Nickel
Chromium	Tin
Cobalt	Zinc
Iron	

Eliminate interference from the metals in Table 1 by the following treatment, beginning after step 6.

1. Measure about 5-mL of the DithiVer solution into the separatory funnel. Stopper the funnel, invert and open the stopcock to vent. Close the stopcock and shake the solution vigorously for 15 seconds. Allow the funnel to stand undisturbed until the layers separate (about 30 seconds). A yellow, red, or bronze color in the bottom (chloroform) layer confirms the presence of interfering metals. Draw off and collect the bottom (chloroform) layer for proper disposal.

2. Repeat extraction with fresh 5-mL portions of prepared dithizone solution (collecting the bottom layer each time in appropriate waste collection vessel) until the bottom layer shows a pure dark green color for three successive extracts. Extractions can be repeated a number of times without appreciably affecting the amount of lead in the sample.

3. Extract the solution with several 2 or 3 mL portions of pure chloroform to remove any remaining dithizone, again collecting the bottom layer each time for proper disposal.

4. Continue the procedure, substituting 28.5 mL of prepared dithizone solution for the 30 mL in step 7.

DithiVer Solution Preparation, Storage, and Reagent Blank

Store DithiVer Powder Pillows away from light and heat. A convenient way to prepare this solution is to add the contents of 10 DithiVer Metals Reagent Powder Pillows to a 500-mL bottle of chloroform and invert several times until well mixed (carrier powder may not dissolve). Store dithizone solution in an amber glass bottle. This solution is stable for 24 hours.

A reagent blank using deionized water should be carried out through the entire method to obtain the most accurate results.

Sample Collection, Preservation, and Storage

Collect samples in an acid-washed glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Adjust the pH to 2.5 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

1. Leave the unspiked sample in the sample cell compartment. Verify that the units displayed are in µg/L.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in top row. See the user manual for more information.

4. Snap the neck off a Lead Voluette Ampule Standard, 50-mg/L Pb.

5. Use the TenSette® Pipet (do not use a glass pipet) to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to three 250-mL samples and mix each thoroughly.

6. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing the soft key under **READ** each time. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 10-mg/L lead standard solution by pipetting 10.00 mL of Lead Standard Solution, 100-mg/L, into a 100-mL volumetric flask.

2. Add 0.2 mL of concentrated nitric acid using a TenSette Pipet to prevent the adsorption of lead onto the container walls. Dilute to the mark with deionized water and mix thoroughly.

3. To make a 200-µg/L standard, pipet 5.00 mL of the 10.0-mg/L standard into 245 mL of deionized water in the 500-mL separatory funnel in step 4 of the Dithizone procedure. Prepare these solutions daily. Perform the lead procedure as described above.

4. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

5. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The dithizone method is designed for the determination of lead in water and wastewater. The DithiVer Metals Reagent is a stable powder form of dithizone. Lead ions in basic solution react with dithizone to form a pink to red lead-dithizonate complex, which is extracted with chloroform. Test results are measured at 515 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Lead Reagent Set (100 Tests)	—	—	22431-00
Includes: (1) 14202-99, (2) 14458-17, (1) 12616-99, (2) 767-14, (1) 2450-53, (2) 2450-26			
Buffer Powder Pillows, citrate	1	100/pkg	14202-99
Chloroform, ACS	30 mL	4 L	14458-17
DithiVer Metals Reagent Powder Pillows	1	100/pkg	12616-99
Potassium Cyanide	0.1 g	125 g	767-14
Sodium Hydroxide Solution, 5.0 N	5 mL	1000 mL	2450-53
Sodium Hydroxide Standard Solution, 5.0 N	varies	59 mL DB	2450-26

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers, for opening powder pillows	1	each	968-00
Cotton Balls, absorbent	1	100/pkg	2572-01
Cylinder, graduated, 5-mL	1	each	508-37
Cylinder, graduated, 50-mL	1	each	508-41
Cylinder, graduated, 250-mL	1	each	508-46
Cylinder, graduated, mixing, 50-mL	1	each	1896-41
Funnel, separatory, 500-mL	1	each	520-49
pH Meter, sens***ion***™1, portable, with electrode	1	each	51700-10
Sample Cell, 1-inch square, 25 mL with cap	2	2/pkg	26126-02

Required Apparatus (continued)

Description	Quantity/Test	Unit	Cat. No.
Spoon, measuring,1-g	1	each	510-00
Support Ring, 4"	1	each	580-01
Support Ring Stand, 5" x 8" base	1	each	563-00

Recommended Standards

Description	Unit	Cat. No.
Lead Standard Solution, 100 mg/L Pb	100 mL	12617-42
Lead Standard Solution, 10-mL Voluette Ampules, 50-mg/L Pb	16/pkg	14262-10

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Chloroform, ACS	500 mL	14458-49
Filter Discs, glass, 47 mm	100/pkg	2530-00
Filter Holder, glass, for 47-mm filter	each	2340-00
Flask, Erlenmeyer, 500-mL	each	505-49
Flask, filtering, 500-mL	each	546-49
Flask, volumetric, Class A, 100-mL	each	14574-42
Nitric Acid Solution, 1:1	500 mL	2540-49
Nitric Acid, ACS	500 mL	152-49
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
Pipet, serological, 2-mL	each	532-36
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet, volumetric, 5.00-mL, Class A	each	14515-37
Pipet, volumetric, 10.00-mL, Class A	each	14515-38
Pipet Filler, safety bulb	each	14651-00
Sulfuric Acid, 5.25 N	100 mL MDB	2449-32
Water, deionized	4 liters	272-56

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Updated November 2005 Edition 1

Lead

Method 10216 **PAR Method**

TNTplus™ 850 **(0.1 to 2.0 mg/L Pb)**

Scope and Application: For wastewater and process control

Test Preparation

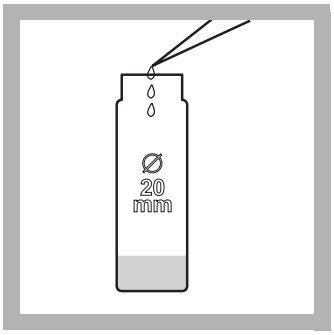
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample pH is 3–9.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F).
Recommended reagent storage is 15–25 °C (59–77 °F).
Samples which are free from complexing agents and have a pH between 3 and 6 can be analyzed directly.
Samples with a pH between 6 and 9 must be additionally digested with Metals Prep Set TNT 890 in order to bring undissolved lead hydroxide or complex lead compounds into solution.
TNTplus methods are activated directly from the Main Menu when the sample vial is inserted into the sample cell holder.

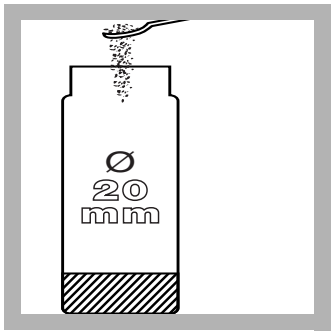
Collect the following items:	**Quantity**
Lead TNT850 Reagent Set	1
Light Shield	1
Pipettor, variable 1–5 mL	1
Pipettor tips for 1–5 mL pipettor	1
Pipet, volumetric 10 mL	1
Safety pipet bulb	1
Pipettor, variable 100–1000µL	1
Pipettor Tips for 100–1000 µL pipettor	1

Note: *Reorder information for consumables and replacement items is on page 4.*

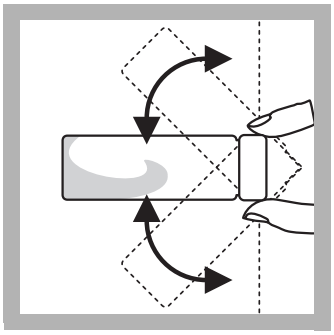
TNTplus Method 10216



1. Pipet 10 mL of sample into the 20-mm reaction tube.

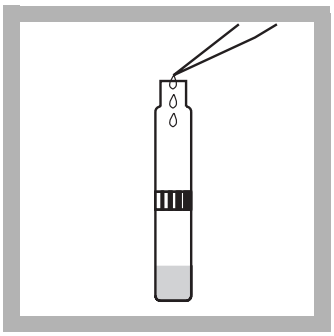


2. Add 1 level spoonful of Reagent A to the reaction tube.

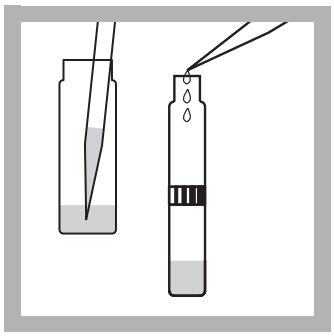


3. Cap the reaction tube and invert 2–3 times.

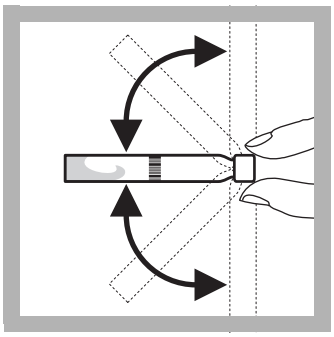
4. Wait two minutes.



5. When the timer expires, add 1.5 mL of Solution B into a sample vial.



6. Pipet 4.0 mL of the pretreated sample from the 20 mm reaction tube prepared in step 3 into the vial.

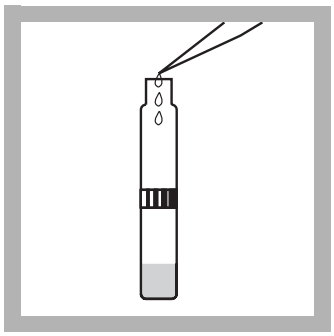


7. Cap and invert the vial 2-3 times.

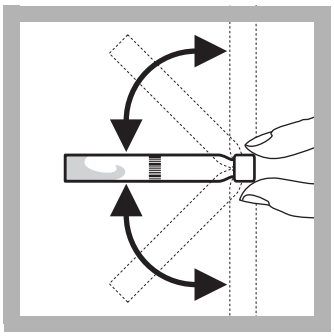
8. Wait two minutes.

Install the Light Shield in Cell Compartment #2.

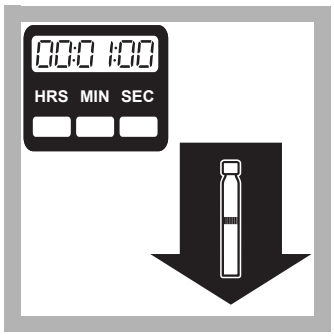
9. Thoroughly clean the outside of the vial and insert it into the sample cell holder. The instrument reads the barcode, then selects the method and sets the blank.



10. Remove the vial and add 0.3 mL (300 µL) of Solution C into the vial.



11. Cap the vial and invert the vial 2–3 times



12. Wait one minute, then insert the prepared vial into the cell holder. The instrument reads the barcode, then reads the sample. Results are in mg/L lead.

Reagent Blank

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 12. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substances	Interference Levels
K+, Na+, Ca^{2+}, Mg^{2+}, NO_3^-, Cl^-, PO_4^{3-}, CO_3^{2-}, SO_4^{2-}	500 mg/L
F^-, NH_4^+, Sr^{2+}	50 mg/L
Ag^+, Cd^{2+}, Cr^{6+}, Zn^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}	25 mg/L
Cr^{3+}, Al^{3+}, Fe^{2+}, Fe^{3+}	10 mg/L
Mn^{2+}, Hg^{2+}	5 mg/L
Sn^{2+}	0.5 mg/L

Sample Collection, Preservation, and Storage

Collect samples in an acid-washed glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Adjust the pH to between 3 and 6 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Prepare a 1.0 mg/L lead standard solution by pipetting 1.0 mL of a 100 mg/L lead standard solution into a 100 mL volumetric flask.

2. Dilute to volume with deionized water. Use 10 mL of this standard in place of the sample in the procedure.

Summary of Method

Lead (II) ions react at pH 9 with 4-(2-pyridylazo)-resorcinol (PAR) to form a red complex. Test results are measured at 520 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Lead TNT 850 Reagent Set	1	25/pkg	TNT850

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipet, variable volume, 1-5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	1	100/pkg	27952-00
Pipet, Volumetric 10 mL	1	each	14515-38
Pipet Filler, safety bulb	1	each	14651-00
Pipet, variable volume, 100–1000 µL	1	each	27949-00
Pipet Tips, for 27949-00 pipet	1	400/pkg	27950-00

Recommended Reagents and Standards

Description	Unit	Cat. No.
Lead Standard Solution, 100 mg/L	100 mL	12617-42
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	2450-26
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pk	20870-79
Flask, volumetric 100 mL	each	14574-42
Metals Prep Set TNT 890	each	TNT890
Pipet, volumetric 1.0 mL	each	14515-35
Test Tube Rack for 13-mm vials	each	24979-00

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Updated November 2005 Edition 1

Manganese

Method 8149 **1-(2-Pyridylazo)-2-Naphthol PAN Method[1]**

Powder Pillows **LR (0.006 to 0.700 mg/L)**

Scope and Application: For water and wastewater; digestion is required for determining total manganese

[1] Adapted from Goto, K., et al., *Talanta*, 24, 652-3 (1977)

Test Preparation

Before starting the test:

Rinse all glassware with 1:1 Nitric Acid Solution. Rinse again with deionized water.
The alkaline cyanide solution contains cyanide. Cyanide solutions should be collected for disposal as a reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. Refer to the current MSDS for safe handling and disposal instructions.

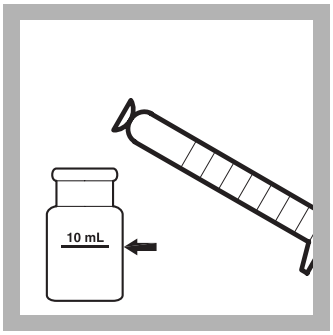
Collect the following items:	**Quantity**
Alkaline Cyanide Reagent	12 drops
Ascorbic Acid Powder Pillows	2
PAN Indicator Solution, 0.1%	12 drops
Deionized Water	10 mL
Sample Cells, 1-inch square, 10-mL	2
Stoppers for 18 mm tube	2

Note: *Reorder information for consumables and replacement items is on page 4.*

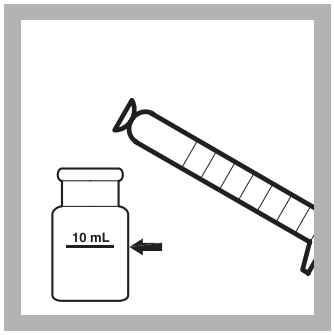
Powder Pillows Method 8149

1. Press **STORED PROGRAMS**.

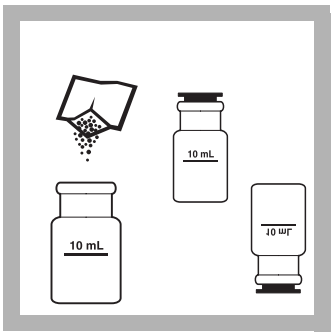
2. Select the test.



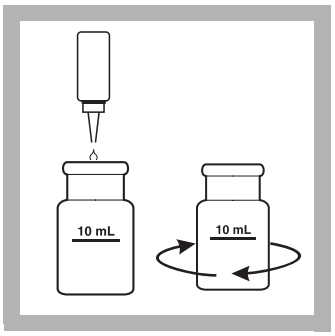
3. Blank Preparation: Pour 10.0 mL of deionized water into a square sample cell.



4. Prepared Sample: Pour 10.0 mL of sample into another square sample cell.

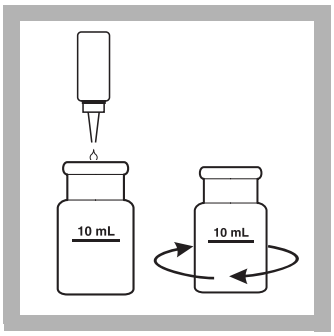


5. Add the contents of one Ascorbic Acid Powder Pillow to each cell. Stopper and invert to dissolve the powder.



6. Add 12 drops of Alkaline-Cyanide Reagent Solution to each cell. Swirl gently to mix.

A cloudy solution may form. The turbidity should dissipate after step 7.



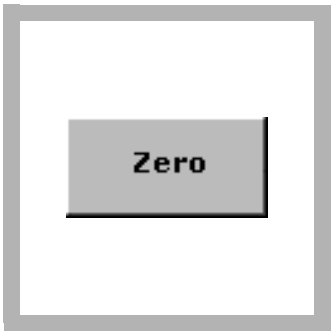
7. Add 12 drops of PAN Indicator Solution, 0.1%, to each sample cell. Swirl gently to mix.

An orange color will develop in the sample if manganese is present.

8. Press **TIMER>OK**.

A two-minute reaction period will begin.

9. When the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.

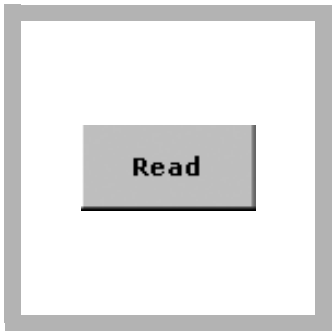


10. Press **ZERO**.

The display will show:

0.000 mg/L Mn

11. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.



12. Press **READ**.

Results are in mg/L Mn.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	20 mg/L
Cadmium	10 mg/L
Calcium	1000 mg/L as $CaCO_3$
Cobalt	20 mg/L
Copper	50 mg/L
Iron	25 mg/L (If sample contains more than 5 mg/L iron, allow a 10-minute reaction period in step 8.)
Lead	0.5 mg/L
Magnesium	300 mg/L as $CaCO_3$
Nickel	40 mg/L
Zinc	15 mg/L

For samples that contain hardness greater than 300 mg/L $CaCO_3$, add 10 drops of Rochelle Salt Solution* to the sample **after** adding the Ascorbic Acid Powder Pillow in step 5.

Sample Collection, Storage, and Preservation

Collect samples in a clean plastic container. Adjust the pH to 2 or less with Concentrated Nitric Acid* (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the pH to between 4–5 with 5.0 N Sodium Hydroxide* before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Manganese Voluette® Ampule Standard, 10-mg/L Mn.

5. Prepare three sample spikes. Fill three Mixing Cylinders* with 10 mL of sample. Use the TenSette® Pipet* to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 0.5-mg/L manganese standard solution by pipetting 2.0 mL of Manganese Voluette Standard Solution, 250-mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. This solution should be prepared daily. Perform the manganese procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 4.

Summary of Method

The PAN method is a highly sensitive and rapid procedure for detecting low levels of manganese. An ascorbic acid reagent is used initially to reduce all oxidized forms of manganese to Mn^{2+}. An alkaline-cyanide reagent is added to mask any potential interferences. PAN Indicator is then added to combine with the Mn^{2+} to form an orange-colored complex. Test results are measured at 560 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Manganese Reagent Set, 10-mL (50 tests), includes:	—	—	26517-00
Alkaline Cyanide Reagent	12 drops	50mL SCDB	21223-26
Ascorbic Acid Powder Pillows	2 pillows	100/pkg	14577-99
PAN Indicator Solution, 0.1%	12 drops	50 mL SCDB	21224-26
Water, deionized	10 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stoppers for 18 mm Tube	2	6/pkg	1731-06

Recommended Standards

Description	Unit	Cat. No.
Manganese Standard Solution, 10-mg/L Mn, 2-mL ampule	20/pkg	26058-20
Manganese Standard Solution, 250-mg/L Mn, 10-mL Voluette® ampule	16/pkg	14258-10

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Cylinder, mixing, 25 mL	each	20886-40
Nitric Acid, concentrated, 500 mL	—	152-49
Pipet, TenSette® 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Rochelle Salt Solution, 29 mL	—	1725-33
Sodium Hydroxide, 5.0 N, 100 mL	—	2450-32
Stopper for 18 mm tube	25/pkg	1731-25

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Updated November 2005 Edition 1

Manganese

★Method 8034 — Periodate Oxidation Method[1]

Powder Pillows — HR (0.1 to 20.0 mg/L)

Scope and Application: For soluble manganese in water and wastewater; USEPA approved for reporting wastewater analyses (digestion required)[2].

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

[2] *Federal Register*, 44(116) 34193 (June 14, 1979)

Test Preparation

Before starting the test:

Digestion is required for reporting wastewater analyses.
If only dissolved manganese is to be determined, filter the sample before acid addition.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

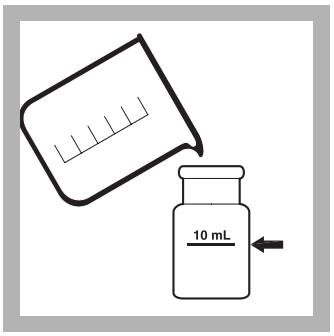
Collect the following items:	**Quantity**
High Range Manganese Reagent Set	1
Sample Cells, 1-inch square, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 4.*

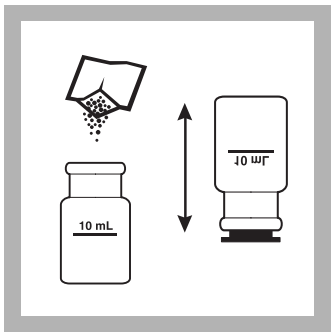
Powder Pillows — Method 8034

1. Press **STORED PROGRAMS**.

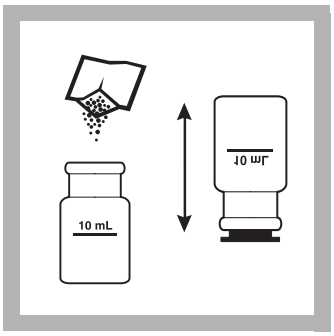
2. Select the test.



3. Prepared Sample: Fill a square sample cell with 10 mL of sample.



4. Add the contents of one Buffer Powder Pillow, Citrate Type for Manganese. Stopper and invert to mix.

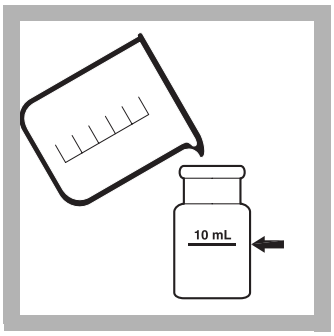


5. Add the contents of one Sodium Periodate Powder Pillow to the sample cell. Stopper and invert to mix.

A violet color will develop if manganese is present.

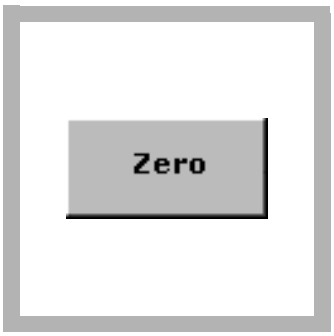
6. Press **TIMER>OK**.

A two-minute reaction period will begin.



7. Blank Preparation: Fill a second square sample cell with 10 mL of sample.

8. When the timer expires, insert the blank into the cell holder with the fill line facing right.



9. Press **ZERO**.

The display will show:

0.0 mg/L Mn

10. Within eight minutes after the timer expires, insert the sample into the cell holder with the fill line facing right.

11. Press **READ**.

Results are in mg/L Mn.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed plastic bottles. Do not use glass containers due to possible adsorption of Mn to glass. If samples are acidified, adjust the pH to 4–5 with 5.0 N Sodium Hydroxide* before analysis. Do not exceed pH 5, as manganese may precipitate. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Manganese Voluette® Ampule Standard, 250-mg/L Mn*.

5. Prepare three sample spikes. Fill three sample cells with 10 mL of sample. Use the TenSette® Pipet* to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 10.0-mg/L manganese standard solution by pipetting 10.0 mL of Manganese Standard Solution, 1000-mg/L, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the manganese periodate oxidation procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 4.

Summary of Method

Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration. Test results are measured at 525 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Manganese Reagent Set, High Range (100 tests), includes:	—	—	24300-00
Buffer Powder Pillows, citrate type for Manganese	1	100/pkg	21076-69
Sodium Periodate Powder Pillows for Manganese	1	100/pkg	21077-69

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper, rubber	1	6/pkg	1731-06

Recommended Standards

Description	Unit	Cat. No.
Manganese Standard Solution, 1000-mg/L Mn,	100 mL	12791-42
Manganese Standard Solution, 250-mg/L Mn, 10-mL Voluette® ampule	16/pkg	14258-10
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Manganese Voluette® Ampule Standard, 250-mg/L	14258-10
Pipet, TenSette®, 0.1–1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01	21856-96
Sodium Hydroxide, 5.0 N, 100 mL	2450-32

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Updated November 2005 Edition 1

Mercury

Method 10065 — Cold Vapor Mercury Concentration Method[1] (0.1 to 2.5 µg/L)

Scope and Application: For water, wastewater, and seawater

[1] Patent no. 5,733,786

Test Preparation

Before starting the test:

Perform phase 1 of the procedure in a fume hood. Toxic chlorine or other gases may be produced.
Use dedicated digestion glassware and sample cells for this procedure.
Determine a reagent blank for each new lot of reagent by running the entire procedure, including the digestion, using one liter of deionized water instead of sample. Add the same amount of potassium permanganate as required by the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

Collect the following items:	**Quantity**
Cold Vapor Mercury Reagent Set (see Required Reagents on page 10 for contents of reagent set)	1
Digestion Reagents and Apparatus (see Required Digestion Reagents and Apparatus on page 11)	varies
Cold Vapor Mercury Apparatus Set	1
Sample Cells, 1-inch square, 10-mL, matched pair	2
See Required Apparatus on page 10 for a complete list of required apparatus.	

Note: *Reorder information for consumables and replacement items is on page 10.*

Phase 1: Sample Digestion

DANGER

Test must be performed under a fume hood – toxic gases may be produced!

1. Transfer one liter of the sample to a 2000-mL Erlenmeyer flask. Add a 50-mm magnetic stir bar to the sample. Set the flask on a magnetic stirring hot plate and begin stirring.

2. Add 50 mL of concentrated sulfuric acid to the sample.

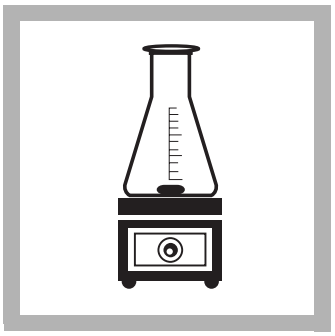
3. Add 25 mL of concentrated nitric acid to the sample.

4. Add 4.0 g of potassium persulfate to the sample. Stir until dissolved.

Alternatively, add one 5-gram measuring scoop of potassium persulfate to the sample.

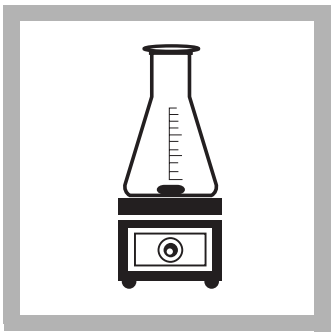
5. Add 7.5 g of potassium permanganate to the sample. Stir until dissolved.

Alternatively, add a 10-gram measuring scoop of potassium permanganate to the sample.



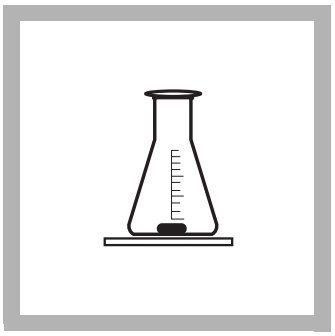
6. Cover the flask with a watch glass. Begin heating the sample to a temperature of 90 °C after the reagents have dissolved. **Do not boil.**

For a mercury standard or reagent blank in distilled water, the heat step is not necessary.



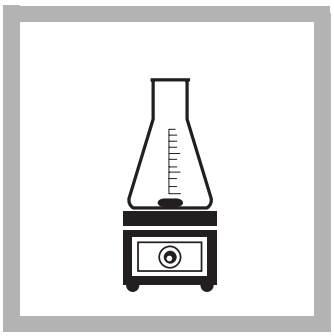
7. Continue to stir and heat the sample at 90 °C for two hours.

The solution must remain dark purple throughout the entire digestion. Some samples, such as sea waters, industrial effluents or other samples high in organic matter or chloride concentration, require additional permanganate. It may be difficult to see a dark purple color if the sample contains black/brown manganese dioxide precipitate. Add more potassium permanganate if the solution is not dark purple.

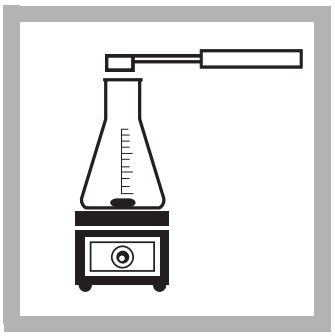


8. Cool the digested sample to room temperature.

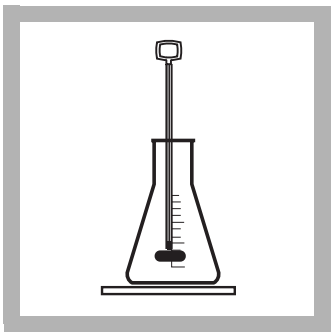
A brown/black precipitate of manganese dioxide may settle during cooling. If the digested sample does not have a purple color, the digestion may be incomplete. Add more potassium permanganate. Return the sample to the magnetic stirring hot plate and continue the digestion until the purple color persists.



9. Return the cool, digested sample to the cool, magnetic stirring hot plate. Turn on the stirrer.



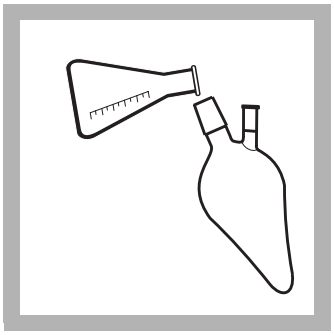
10. Using a 0.5-g measuring spoon, add 0.5 g additions of hydroxylamine-hydrochloride until the purple color disappears. Wait 30 seconds after each addition to see if the purple disappears. Add hydroxylamine-hydrochloride until all manganese dioxide is dissolved.



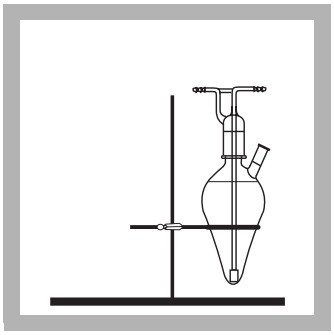
11. Remove the stir bar.

12. The digested sample is now ready for processing by cold vapor separation and preconcentration. Proceed to Phase 2.

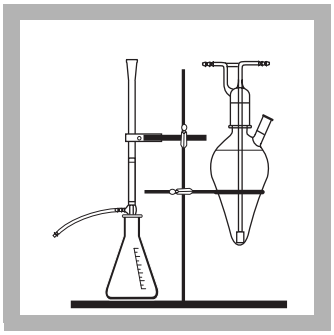
Phase 2: Cold Vapor Separation and Preconcentration of Mercury



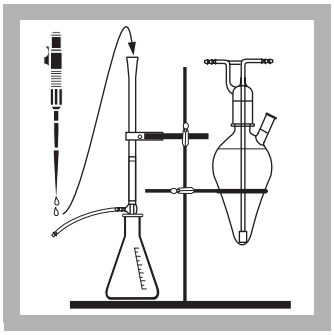
1. Transfer the digested sample to the Cold Vapor Gas Washing Bottle. (The volume of the digested sample should contain 0.1 to 2.5 µg Hg.)



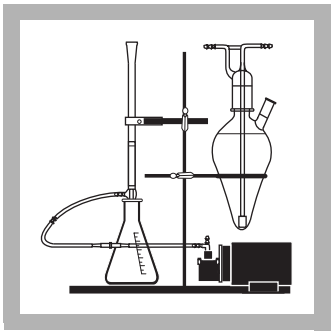
2. Set the Gas Washing Bottle in the support ring. Place the top on the Gas Washing Bottle. Wait until step 9 to connect the mercury absorber column to the Gas Washing Bottle.



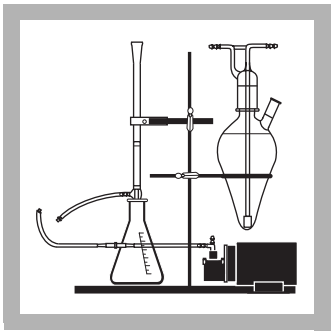
3. Connect the 100-mL Erlenmeyer flask to the mercury absorber column.



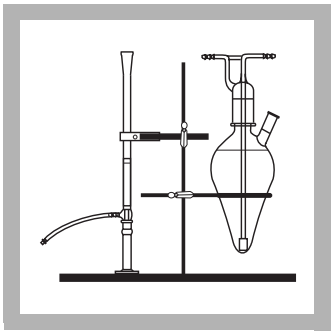
4. Pipet 8 mL of HgEx Reagent B into the Mercury Absorber column.



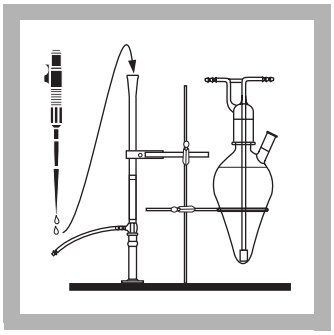
5. Connect the power to the vacuum pump and apply vacuum to the Mercury Absorber Column. Draw most of the HgEx Reagent B into the Erlenmeyer flask.



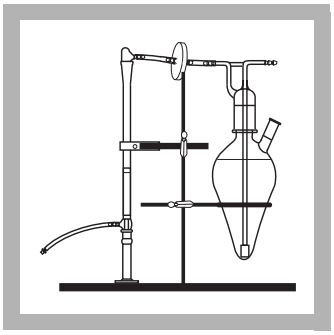
6. Disconnect the vacuum using the quick disconnect when HgEx Reagent B begins to drip from the inner delivery tube on the Mercury Absorber Column (about 10 seconds after starting the vacuum). Do not draw enough air through the column to begin drying the packing.



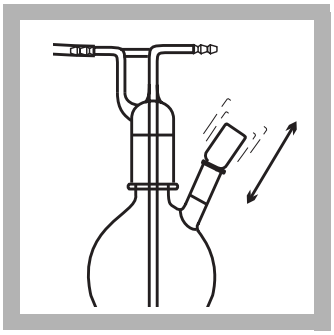
7. Remove the 100-mL Erlenmeyer flask from the Mercury Absorber Column. Replace it with the 10-mL Distilling Receiver.



8. Pipet 2 mL of HgEx Reagent C into the Mercury Absorber Column.

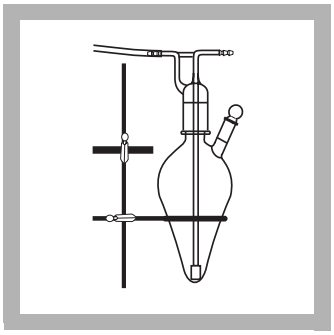


9. Connect the Mercury Absorber column to the Gas Washing Bottle using the glass elbow.

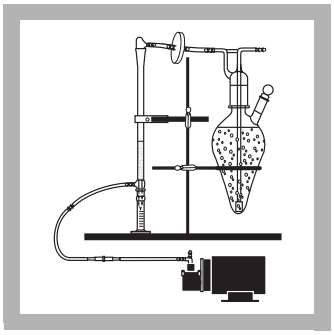


10. Shake an ampule of HgEx Reagent A to suspend undissolved reagent.

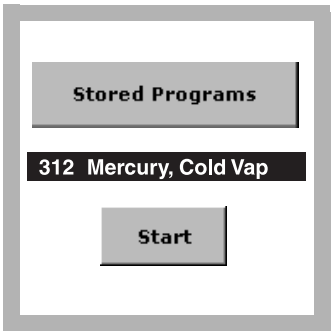
Open the ampule and gently shake the contents into the Gas Washing Bottle through the side neck.



11. Stopper the side neck on the Glass Washing Bottle.



12. Reconnect the vacuum to the Mercury Absorber Column using the quick disconnect. The vacuum will pull HgEx Reagent C through the Mercury Absorber Column packing and into the 10-mL receiver. Air bubbles should be produced at the gas dispersion tube in the Gas Washing Bottle. Perform steps 13–14 immediately.

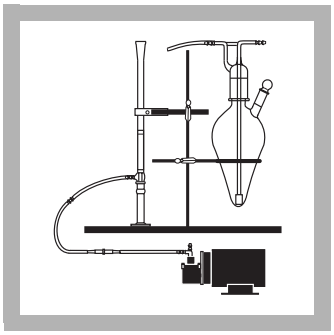


13. Select the test.

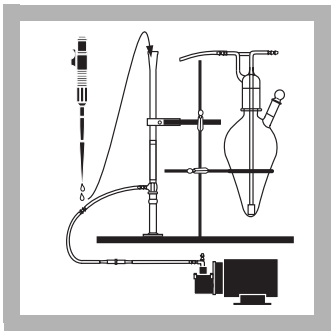
14. Press **TIMER>OK**.

A five-minute reaction period will begin. Let the solution bubble for this period.

Air flow rate through the Gas Washing Bottle should be between 1–5 L/min. Allow more bubbling time for lower air flow rates. For example, if the air flow rate is 1 L/min., let the solution bubble for 10 minutes.

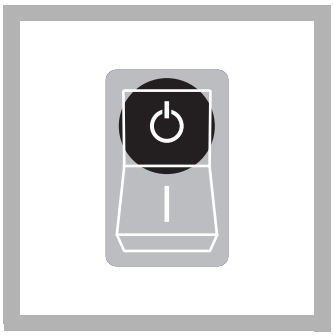


15. After the timer expires, remove the glass elbow from the top of the Mercury Absorber Column. Keep the vacuum pump on.



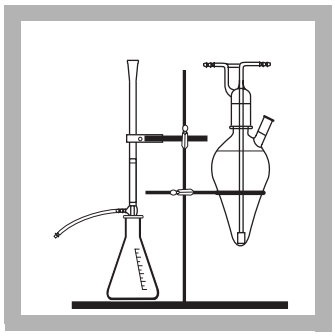
16. Pipet 8 mL of HgEx Reagent B into the Mercury Absorber Column to elute the captured mercury.

Continue to apply vacuum to pull the HgEx Reagent B into the Distilling Receiver.

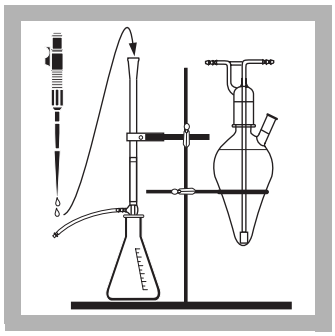


17. Turn off or disconnect power to the vacuum pump when the volume in the Distilling Receiver reaches the 10 mL mark.

If necessary, the volume in the Distilling Receiver may be brought up to 10 mL with HgEx Reagent B. To avoid low volumes in the future, disconnect the vacuum a little sooner in step 6. This leaves more HgEx Reagent B in the packing of the Mercury Absorber Column.



18. Remove the distilling Receiver from the Mercury Absorber Column. Reconnect the 100-mL Erlenmeyer flask to the column.

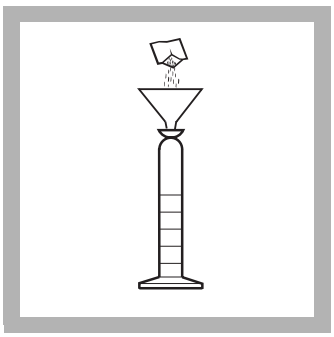


19. Pipet 3 mL of HgEx Reagent B into the Mercury Absorber Column without applying vacuum. This keeps the absorber packing wet between tests.

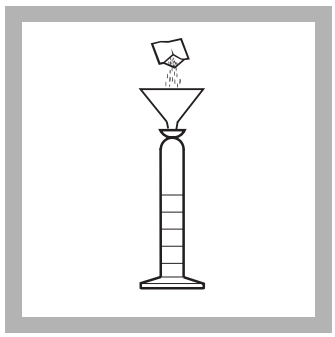
The Mercury Absorber Column eluate in the Distilling Receiver is ready for analysis.

Proceed to Phase 3.

Phase 3: Colorimetric Analysis



1. Using the funnel provided, add the contents of one HgEx Reagent 3 foil pillow to the eluate in the Distilling Receiver. Stopper the receiver. Invert to dissolve the reagent.

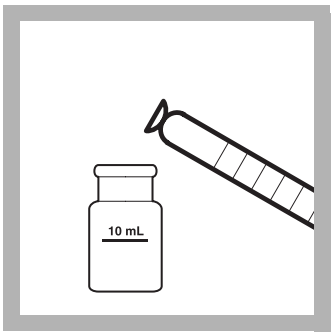


2. Add the contents of one HgEx Reagent 4 foil pillow to the Distilling Receiver using the funnel provided. Stopper the receiver. Invert to dissolve the reagent.

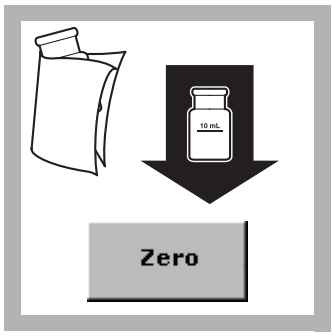
3. Add 8 drops of HgEx Reagent 5 to the Distilling Receiver. Stopper the Receiver. Invert to mix.

4. Press **TIMER>OK**.

A two-minute reaction period will begin.



5. During the reaction period, transfer the solution to a sample cell.



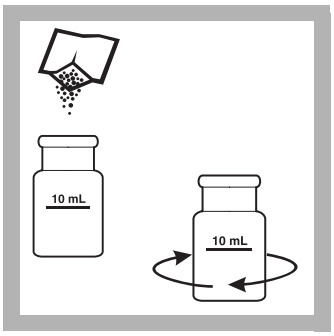
6. Wipe the sample cell. After the timer expires, insert the sample into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

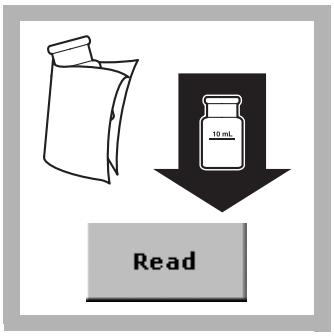
0.1 µg/L Hg

(This program uses a non-zero intercept.)



7. Remove the cell from the cell holder. Add the contents of one HgEx Reagent 6 foil pillow to the solution. Swirl the cell until the reagent is completely dissolved. Immediately go to step 8.

Do not use the funnel to add HgEx Reagent 6 to the sample cell. Any HgEx Reagent 6 in the funnel will make mercury undetectable in subsequent tests.



8. Return the sample cell to the cell holder with the fill line facing right.

Press **READ**. Results are in µg/L Hg.

Interferences

Standards were used to prepare a single test solution with the following matrix. A second test solution containing only mercury at the same concentration was prepared as the control. The two solutions were digested then analyzed concurrently. There was no interference from the matrix of the test solution at the concentrations listed.

In addition, no interference occurred with a test solution containing 1000 mg/L Na^{+}, 1000 mg/L K^{+}, 1000 mg/L Mg^{2+}, and 400 mg/L Ca^{2+}.

Table 1 Interfering Substances and Levels

Ion or Substance	Concentration
Ag^{+}	7 mg/L Ag^{+}
Al^{+3}	10 mg/L Al^{+3}
Au^{+3}	500 µg/L Au^{+3}
Cd^{+2}	10 mg/ L Cd^{+2}
Co^{+2}	10 mg/L Co^{+2}
Cr^{+6}	10 mg/L Cr^{+6}
Cu^{+2}	10 mg/L Cu^{+2}
F^{-}	1.0 mg/L F^{-}
Fe^{+2}	100 mg/L Fe^{+2}
Hg^{+2}	1 µg/L Hg^{+2}
Mo^{+6}	10 mg/L Mo^{+6}
Ni^{+2}	10 mg/L Ni^{+2}

Table 1 Interfering Substances and Levels (continued)

Ion or Substance	Concentration
NO_3^-–N	50 mg/L NO_3^-–N
Pb^{2+}	10 mg/L Pb^{2+}
SiO_2	100 mg/L SiO_2
Zn^{+2}	10 mg/L Zn^{+2}

Sample Collection and Preservation

Collect 1000 mL of sample in an analytically clean, glass or polyethylene terephthalate (PET) container. Add 10 mL of concentrated hydrochloric acid to preserve the sample before sample collection. Fill the container completely full to minimize air space when closed. Close a glass container with a ground glass stopper. Close a PET container with a PET cap or a polypropylene cap (no liner).

Store aqueous samples at 2–6 °C. Acid-preserved samples are stable for at least 6 months.

Accuracy Check

Standard Additions Method

1. Prepare a 10.0-mg/L Mercury Standard Solution as described under Standard Solution Method, step 3a.
2. Use a TenSette® Pipet to add 0.10 mL of the 10.0-mg/L Mercury Standard Solution to the purged solution in the Gas Washing Bottle after an analysis has been performed. Immediately stopper the Gas Washing Bottle.
3. Begin at step 3 of Phase 2. Follow the procedure steps.
4. Test the eluate as described in Phase 3. The displayed concentration should be 0.9–1.1 µg/L Hg.

Standard Solution Method

1. Transfer 800 mL of deionized water into the Gas Washing Bottle.
2. Add 50 mL of concentrated sulfuric acid and 25 mL of concentrated nitric acid to the water. Swirl to mix.
3. Prepare a 0.1-mg/L mercury standard solution by serially diluting a 1000-mg/L Mercury Standard Solution:
   a. To make a 10.0 mg/L standard, add 1.0 mL of concentrated nitric acid to a 500-mL volumetric flask. Dilute 5.00 mL of a 1000-mg/L standard to 500 mL with deionized water. Mix well.
   b. To make a 1.0-mg/L standard solution, add 0.2 mL of concentrated nitric acid to a 100-mL volumetric flask. Dilute 10.0 mL of the 10.0-mg/L standard to 100 mL with deionized water. Mix well.
   c. To make a 0.1-mg/L standard solution, add 0.2 mL of concentrated nitric acid to a 100-mL volumetric flask. Dilute 10.00 mL of the 1.0-mg/L solution to 100 mL with deionized water. Mix well.

4. Pipet 10.0 mL of the 0.1-mg/L mercury standard solution into the Gas Washing Bottle. Swirl to mix.

5. Begin at step 2 of Phase 2. Follow the procedure steps.

6. Test the eluate as described in Phase 3. The displayed concentration should be 0.9–1.1 µg/L Hg.

System Start Up

For more accurate results, perform a few analyses on mercury standards and blanks for system equilibration before beginning sample testing. This allows the system to stabilize before processing samples.

Startup Standard
Test a mercury standard solution by following the procedure under Accuracy Check using the Standard Solution Method. Continue with step 1 of the Startup Standard procedure if the value is not within specified limits.

1. Pipet 10.0 mL of the 0.1-mg/L mercury standard solution into the purged solution in the Gas Washing Bottle. Immediately stopper the Gas Washing Bottle.

2. Begin at step 3 of Phase 2. Follow the procedure steps.

3. Test the eluate as described in Phase 3. The displayed concentration should be 0.9–1.1 µg/L Hg. Repeat steps 1–3 if the value is not within these limits.

Startup Blank
Run a system blank by using the purged solution in the Gas Washing Bottle after a satisfactory test of the Startup Standard has been completed.

1. Leave the purged solution in the Gas Washing Bottle. Do not add an aliquot of mercury standard.

2. Begin at step 3 of Phase 2. Follow the procedure steps.

3. Test the eluate as described in Phase 3. The displayed concentration should be ≤ 0.2 µg/L Hg. Repeat the Startup Blank procedure until a reproducible value is obtained.

Storage and Maintenance of the Cold Vapor Mercury Apparatus

Storage
Store the apparatus as follows for fastest system stabilization and greatest sensitivity:

- Store the Gas Washing Bottle filled with deionized water containing 15 mL of concentrated sulfuric acid. Seal the bottle with the Gas Washing Bottle stopper and top.

- Store the Mercury Absorber Column with the packing wetted with HgEx Reagent B. The erlenmeyer flask should be kept attached underneath the column. The top of the Mercury Absorber column should be attached to the Gas Washing Bottle with the glass elbow as in the procedure.

Glassware Care
Use of dedicated glassware and sample cells is recommended because of the sensitivity of this procedure. Thoroughly clean the glassware and sample cells between tests. After washing, rinse with 1:1 hydrochloric acid solution, then rinse several times with deionized water.

Maintaining the System

- With proper care and storage, the Mercury Absorber Column may be used an unlimited number of times.
- Replace the Mercury Scrubber in the air trap housing at least once for every reagent set used.
- Moisture build up on the Gas Washing Bottle side of the Acro 50 Vent Filter will reduce the purging air flow rate. If this occurs replace the filter or dry it in an oven at 110 °C.

Summary of Method

The sample is digested to convert all forms of mercury in the sample to mercuric (Hg^{2+}) ions. The mercuric ions in the digested sample are converted to mercury vapor in a semi-closed system. The vapor is carried into a chemically activated absorber column by ambient air where the mercury vapor is converted to mercuric chloride.

The mercuric chloride is eluted off the column and a sensitive indicator is added. The instrument is zeroed using the absorbance peak of the unreacted indicator. A complexing agent is added to break the mercury:indicator complex. The increase in unreacted indicator causes an increase in absorbance proportional to the amount of mercury in the original sample. Test results are measured at 412 nm.

Safety

Wear personal protective equipment such as safety glasses with side shields, or a face shield to protect your eyes. Use other protective equipment as necessary (such as a fume hood) to avoid chemical exposure. Perform all steps exactly as prescribed in the procedure.

Pollution Prevention and Waste Management

Proper management and disposal of waste is the responsibility of the waste generator. It is up to the generator to arrange for proper disposal and comply with applicable local, state, and federal regulations governing waste disposal. The manufacturer makes no guarantees or warranties, express or implied, for the waste disposal information represented in this procedure.

1. Dispose of the solution in the Gas Washing Bottle by neutralizing the solution to a pH of 6–9 and flushing to the sanitary sewer with water for several minutes.

2. The mercury contained in one liter of sample is concentrated by a factor of 100 by the Mercury Absorber Column. Mercury analysis within the range of the test may produce a solution in the sample cell that is above the RCRA Toxicity Characteristic limit of 0.20 mg/L Hg. The sample cell will contain 0.25 mg/L mercury if the original sample was at 2.5 µg/L mercury (the upper limit of the test range). Dispose of the solution in the sample cell as a hazardous waste if the test result was over 2 µg/L mercury in the original sample. Otherwise, pour the solution into the sanitary sewer and flush with water for several minutes.

3. The mercury scrubber will capture mercury vapor if the Mercury Absorber Column is not properly activated using HgEx Reagent B and HgEx Reagent C. In addition, mercury is also captured if the capacity of the Absorber Column is exceeded. If the Mercury Scrubber has captured mercury vapor, it must be disposed of according to applicable regulations.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Cold Vapor Mercury Reagent Set (25 tests), Includes:			26583-00
HgEx™ Reagent A, Stannous Sulfate Solution, 20-mL ampules	1	25/pkg	26588-25
HgEx™ Reagent B, Sulfuric Acid Solution	19 mL	500 mL	26589-49
HgEx™ Reagent C, Sodium Hypochlorite Solution	2 mL	55 mL	26590-59
HgEx™ Reagent 3, Alkaline Reagent Powder Pillows	1 pillow	25/pkg	26584-48
HgEx™ Reagent 4, Indicator Powder Pillows	1 pillow	25/pkg	26585-48
HgEx™ Reagent 5, Sodium Hydroxide Solution	8 drops	10 mL SCDB	26586-36
HgEx™ Reagent 6, Complexing Reagent Powder Pillows	1 pillow	25/pkg	26587-48
Mercury Scrubber	2/reagent set	2/pkg	26558-00

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cold Vapor Mercury Apparatus Set	1	each	26744-00
Acro 50 Vent Filter	1	18/pkg	26833-18
Air Trap Holder Assembly	1	each	26639-00
Ampule Breaker	1	each	25640-00
Breaker/Capper Tool for Mercury Scrubber	1	each	26640-00
C-flex Tubing, 0.25-inch ID, white	4 ft	25 ft	23273-67
Clamp for Mercury Absorber Column	1	each	26562-00
Clamp Holder	2	each	326-00
Cylinder, graduated, 50-mL	1	each	508-41

Required Apparatus (continued)

Description	Quantity/Test	Unit	Cat. No.
Distilling Receiver, 10-mL	1	each	26554-38
Flask, Erlenmeyer, 100-mL	1	each	26553-42
Funnel, micro	1	each	25843-35
Gas Washing Bottle, 1200-mL	1	each	26622-00
Glass Elbow, 90-degree, with hose adapter	1	each	26552-00
Mercury Absorber Column	1	each	26555-10
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-01	varies	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette Pipet	varies	50/pkg	21997-96
Sample Cells, 1-inch square, 10-mL, matched pair	1	2/pkg	24954-02
Support Ring for Gas Washing Bottle	1	each	26563-00
Stopper, for Distilling Receiver	1	each	26559-00
Stopper, for Gas Washing Bottle	1	each	26623-00
Support, Base and Rod	1	each	329-00
Tubing Quick Disconnect, HDPE	1	12/pkg	14810-00
Vacuum Pump, 115 VAC w/ North American Plug	1	each	28248-00
Vacuum Pump, 220 VAC w/ North American Plug	1	each	28248-01
Vacuum Pump, 220 V w/ European Plug	1	each	28248-02

Required Digestion Reagents and Apparatus

Description	Quantity/Test	Unit	Cat. No.
Flask, Erlenmeyer, 2000-mL	1	each	24894-54
Hot Plate/Stirrer, 120 VAC	1	each	23442-00
Hot Plate/Stirrer, 240 VAC	1	each	23440-02
Hydroxylamine Hydrochloride, ACS	varies	113 g	246-14
Nitric Acid, ACS	25 mL	500 mL	152-49
Potassium Permanganate, ACS	varies	454 g	168-01H
Potassium Persulfate, ACS	4.0 g	454 g	26175-01
Sulfuric Acid, ACS, concentrated	50 mL	4 kg	979-09
Spoon, measuring, 0.5-g	1	each	907-00
Stir Bar	1	each	20953-55
Thermometer, –20 to 110 °C	1	each	566-01
Watch Glass, Pyrex, 65-mm	1	each	578-67

Recommended Standards

Description	Unit	Cat. No.
Mercury Standard Solution, 1000-mg/L Hg (NIST)	100 mL	14195-42
Water, deionized	4 L	272-56

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Updated November 2005 Edition 1

Molybdenum, Molybdate

Method 8036

Mercaptoacetic Acid Method[1]

Powder Pillows or AccuVac® Ampuls

HR (0.2 to 40.0 mg/L)

Scope and Application: For water and wastewater.

[1] Adapted from *Analytical Chemistry, 25(9) 1363 (1953)*

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.
Filter turbid samples using filter paper[1] and a funnel[1].
After all reagents have been added, the presence of molybdenum will cause a yellow color to form.

Collect the following items:	**Quantity**
Powder Pillow Test:	
MolyVer® 1 Molybdenum Reagent Powder Pillows	1
MolyVer® 2 Molybdenum Reagent Powder Pillows	1
MolyVer® 3 Molybdenum Reagent Powder Pillows	1
Sample Cells, 1-inch square, 10-mL	2
AccuVac Test:	
CDTA Solution, 0.4 M	4 drops
MolyVer® 6 Reagent AccuVac® Ampuls	1
Beaker, 50 mL	1
Sample Cells, 10-mL, with cap	1

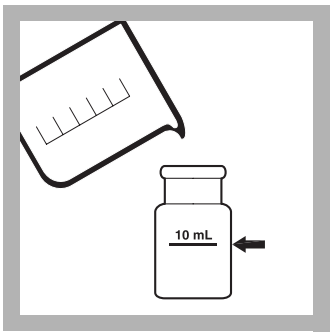
Note: *Reorder information for consumables and replacement items is on page 6.*

[1] See Optional Reagents and Apparatus on page 6.

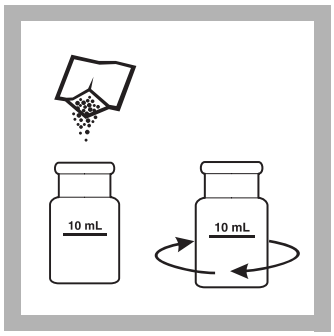
Powder Pillows — Method 8036

1. Press **STORED PROGRAMS**.

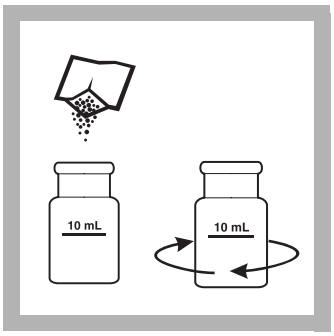
2. Select the test.



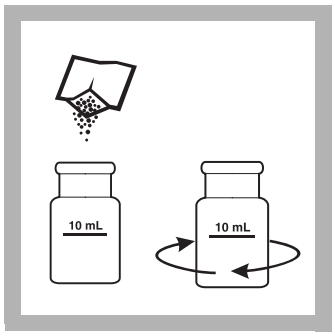
3. Fill a square sample cell with 10-mL of sample.



4. Prepared Sample: Add the contents of one MolyVer® 1 Reagent Powder Pillow. Swirl to mix.



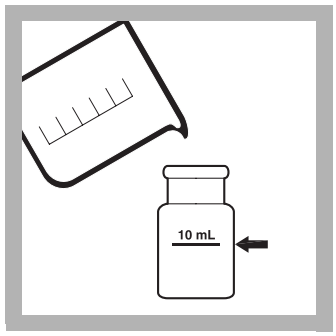
5. Add the contents of one MolyVer 2 Reagent Powder Pillow. Swirl to mix.



6. Add the contents of one MolyVer 3 Reagent Powder Pillow. Swirl to mix.

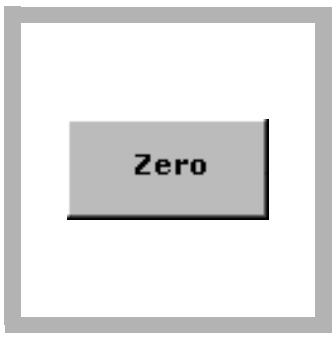
7. Press **TIMER>OK**.

A five-minute reaction period will begin.



8. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of the original sample.

9. Insert the blank into the cell holder with the fill line facing right.



10. Press **ZERO**.

The display will show:

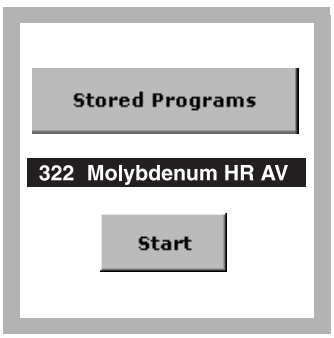
0.0 mg/L Mo^{6+}

11. Insert the prepared sample into the cell holder with the fill line facing right.

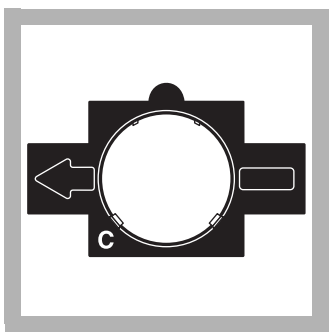
12. Press **READ**.

Results are in mg/L Mo^{6+}.

AccuVac Ampul — Method 8036

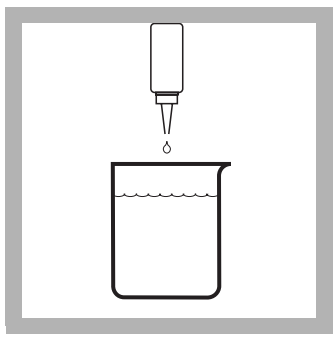


1. Select the test.

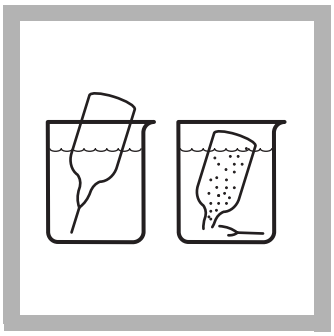


2. Insert Adapter C.

3. Blank Preparation: Fill a round sample cell with 10 mL of sample.



4. Prepared Sample: Collect 40 mL of sample in a 50-mL beaker. Add four drops of 0.4 M CDTA Standard Solution to the sample in the beaker. Swirl to mix.



5. Fill a MolyVer 6 AccuVac® Ampul with the treated sample.

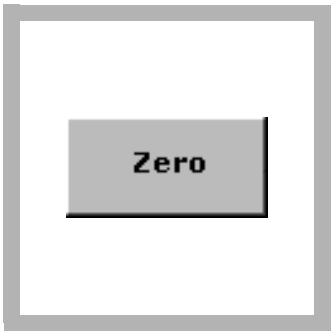
6. Invert the Ampul several times to mix.

Undissolved reagent will not affect test results.

7. Press **TIMER>OK**.

A five-minute reaction period will begin.

8. When the timer expires, insert the blank into the cell holder.



9. Press **ZERO**.

The display will show:

0.0 mg/L Mo^{6+}

10. Insert the prepared sample into the cell holder.

Press **READ**. Results are in mg/L Mo^{6+}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 50 mg/L
Chromium	Greater than 1000 mg/L
Copper	Samples containing 10 mg/L copper or more will exhibit an increasing positive interference upon standing. Read these samples as soon as possible after the five minute reaction period is complete.
Iron	Greater than 50 mg/L
Nickel	Greater than 50 mg/L
Nitrite	Interference from up to 2000 mg/L as NO_2^- can be eliminated by adding one Sulfamic Acid Powder Pillow[1] to the sample.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

[1] See Optional Reagents and Apparatus on page 6.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL/L). Preserved samples can be stored up to 6 months at room temperature. Adjust the pH to 7 with 5.0 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a bottle of Molybdenum Standard Solution, 1000-mg/L Mo^{6+}.

5. Prepare three sample spikes. Fill three Mixing Cylinders* with 30 mL of sample. Use the TenSette® Pipet to add 0.2 mL, 0.4 mL, and 0.6 mL of standard, respectively, to each sample and mix thoroughly.

Note: *For AccuVac® Ampuls, fill three Mixing Cylinders* with 60-mL of sample and spike with 0.4 mL, 0.8 mL, and 1.2 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL Beakers*. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

6. Analyze each sample spike as described in the procedure above, starting with the 0.2 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 6.

Standard Solution Method
To assure the accuracy of the test, use a Molybdenum Standard Solution, 10.0-mg/L Mo^{6+}. Follow the procedure for powder pillows or AccuVac Ampuls.

Standard Adjust

1. Use a Molybdenum Standard Solution, 10.0 mg/L Mo^{6+} to perform the molybdenum procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 3 provides the mercaptoacetic acid which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration. Test results are measured at 420 nm.

Consumables and Replacement Items

Required Reagents (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Molybdenum Reagent Set, for 10-mL samples (100 tests):	—	—	26041-00
MolyVer® 1 Molybdenum Reagent Powder Pillows	1	100/pkg	26042-99
MolyVer® 2 Molybdenum Reagent Powder Pillows	1	100/pkg	26043-99
MolyVer® 3 Molybdenum Reagent Powder Pillows	1	100/pkg	26044-99
OR			
CDTA Solution, 0.4 M	4 drops	15 mL SCDB	26154-36
MolyVer® 6 Reagent AccuVac® Ampuls	1	25/pkg	25220-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-00

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Molybdenum Standard Solution, 10-mg-L as Mo	100 mL	14187-42
Molybdenum Standard Solution, 1000-mg-L as Mo	100 mL	14186-42
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Beakers, 50-mL	500-41H
Cylinder, mixing, 50 mL	1896-41
Filter Paper	1894-57
Funnel	1083-67
Pipet, TenSette®, 0.1 to 1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01	21856-96
Sulfamic Acid Powder Pillow	1055-99

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Updated November 2005 Edition 1

Molybdenum, Molybdate

Method 8169

Powder Pillows

Ternary Complex Method

(0.02 to 3.00 mg/L)

Scope and Application: For boiler and cooling tower waters

Test Preparation

Before starting the test:

Analyze samples immediately after collection.
Filter turbid samples using filter paper[1] and a funnel[1].

[1] See Optional Reagents and Apparatus on page 5.

Collect the following items:	**Quantity**
Molybdenum Reagent Set for 20-mL sample	
Molybdenum 1 Reagent (LR) Molybdate Powder Pillow	1
Molybdenum 2 Reagent Solution	0.5 mL
Cylinder, graduated mixing, 25-mL	1
Sample Cells, 1-inch square, 10 mL, matched pair	2

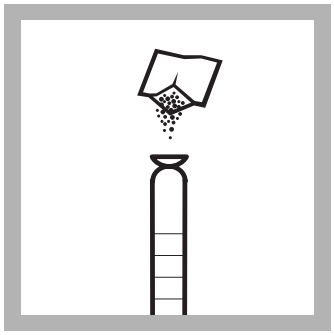
Note: *Reorder information for consumables and replacement items is on page 5.*

Powder Pillows — Method 8169

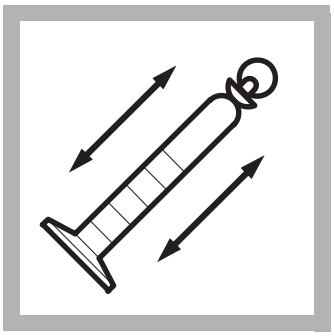
1. Press **STORED PROGRAMS**.

2. Select the test.

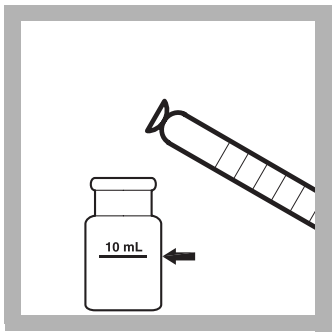
3. Fill a 25-mL graduated mixing cylinder with 20 mL of sample.



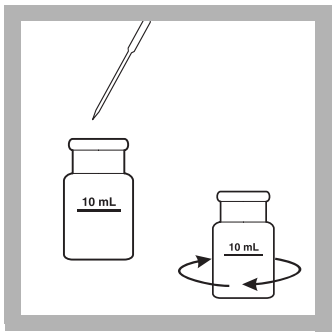
4. Add the contents of one Molybdenum 1 Reagent Powder Pillow to the graduated cylinder.



5. Prepared Sample: Stopper the cylinder and shake to dissolve the reagent.



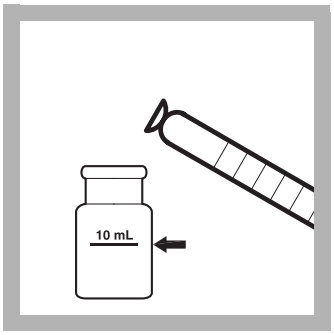
6. Pour 10-mL of the prepared sample into a square sample cell.



7. Developed Sample: Add 0.5 mL of Molybdenum 2 Reagent to the sample cell. Swirl to mix.

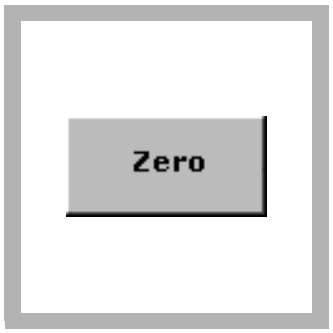
8. Press **TIMER>OK**.

A two-minute reaction period will begin.



9. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of the remaining prepared sample.

10. Wipe the blank and insert it into the cell holder with the fill line facing right.



11. Press **ZERO**.

The display will show: 0.00 mg/L Mo^{6+}

12. Wipe the developed sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Mo^{6+}.

Interferences

Interferences studies were conducted by preparing a molybdenum standard solution (2-mg/L Mo^{6+}) as well as a solution of the potential interfering ion. When the standard solution concentration changed by ± 5% with a given ion concentration, the ion was considered an interference. Tables 1–3 list the details of these studies.

Table 1 Substances That Cause a Negative Interference

Interfering Substance	Interference Levels and Treatments
Alum	Greater than 7 mg/L
Aluminum	Greater than 2 mg/L
AMP (Phosphonate)	Greater than 15 mg/L
Bicarbonate	Greater than 5650 mg/L
Bisulfate	Greater than 3300 mg/L
Borate	Greater than 5250 mg/L
Chloride	Greater than 1400 mg/L
Chromium	Greater than 4.5 mg/L[1]

Table 1 Substances That Cause a Negative Interference (continued)

Interfering Substance	Interference Levels and Treatments
Copper	Greater than 98 mg/L
Diethanoldithiocarbamate	Greater than 32 mg/L
EDTA	Greater than 1500 mg/L
Ethylene Glycol	Greater than 2% (by volume)
Iron	Greater than 200 mg/L
Lignin Sulfonate	Greater than 105 mg/L
Nitrite	Greater than 350 mg/L
Orthophosphate	Greater than 4500 mg/L
Phosphonohydroxyacetic Acid	Greater than 32 mg/L
HEDP (Phosphonate)	The presence of the phosphonate HEDP at concentrations up to 30 mg/L will increase the apparent molybdenum concentration reading by approximately 10% (positive interference). Multiply the value obtained in step12 by 0.9 to obtain the actual Mo^{6+} concentration.
Sulfite	Greater than 6500 mg/L

[1] Read the molybdenum concentration immediately after the beep of the 2-minute reaction period.

Table 2 Substances That Cause a Positive Interference

Interfering Substance	Interference Levels and Treatments
Benzotriazole	Greater than 210 mg/L
Carbonate	Greater than 1325 mg/L
Morpholine	Greater than 6 mg/L
Phosphonate HEDP	Positive interference of about 10% up to 30 mg/L. As the concentration increases above 30 mg/L, a decrease in the molybdenum concentration reading occurs (negative interference).
Silica	Greater than 600 mg/L

Table 3 Non-interfering Substances

Interfering Substance	Interference Levels and Treatments
Bisulfite	9600 mg/L
Calcium	720 mg/L
Chlorine	7.5 mg/L
Magnesium	8000 mg/L
Manganese	1600 mg/L
Nickel	250 mg/L
PBTC (phosphonate)	500 mg/L
Sulfate	12,800 mg/L
Zinc	400 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagent and require sample pretreatment. Adjust the sample pH to between 3–5 by adding, dropwise, an appropriate amount of acid or base such as 1.0 N Sulfuric Acid Standard Solution*, or 1.0 N Sodium Hydroxide Standard Solution*. If significant volumes of acid or base are used, a volume correction should be made by dividing the total volume (sample + acid + base) by the original volume and multiplying the test result by this factor.

After a number of samples have been analyzed, the sample cells may exhibit a slight blueish discoloration. Rinse the cells with 1:1 Hydrochloric Acid Solution† to eliminate this build-up.

* See Optional Reagents and Apparatus on page 5.

Sample Collection, Storage, and Preservation

Collect samples in glass or plastic bottles. Analyze samples immediately.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Molybdenum Voluette® Ampule Standard, 500-mg/L Mo^{6+}.

5. Prepare three sample spikes. Fill three mixing cylinders* with 100 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze 20 mL of each standard addition sample as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Standard Solution Method

1. Using Class A glassware, prepare a 2.00-mg/L molybdenum standard solution by pipetting 10.00 mL of Molybdenum Standard Solution, 10.00-mg/L, into a 50-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the molybdenum procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and sensitizing agent to give a stable blue complex. Test results are measured at 610 nm.

† See Optional Reagents and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Molybdenum Reagent Set for 20-mL sample (100 tests), includes:	—	—	24494-00
(1) Molybdenum 1 Reagent (LR) Molybdate Powder Pillows	1	100/pkg	23524-49
(1) Molybdenum 2 Reagent Solution	0.5 mL	50 mL MDB	23525-12

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Cylinder, graduated mixing, 25-mL	1	each	1896-40

Recommended Standards

Description	Unit	Cat. No.
Molybdenum Standard Solution, 10-mg/L Mo^{6+}	100 mL	14187-42
Molybdenum Standard Solution, 10-mL Voluette® ampule, 500-mg/L Mo^{6+}	16/pkg	14265-10
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing, 100 mL	1896-42
Filter Paper	1894-57
Funnel	1083-67
Hydrochloride Acid Solution 1:1	884-49
Pipet, TenSette®, 0.1–1.0 mL	19700-01
Pipet Tips, for TenSette Pipet 19700-01	21856-96
Sodium Hydroxide Standard Solution, 1.0 N	1045-32
Sulfuric Acid Standard Solution, 1.0 N	1270-32

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Updated November 2005 Edition 1

Nickel

Method 8150
Powder Pillows

1-(2 Pyridylazo)-2-Napthol (PAN) Method[1]
(0.006 to 1.000 mg/L)

Scope and Application: For water and wastewater; digestion is required for determining total nickel

[1] Adapted from Watanabe, H., *Talanta*, 21 295 (1974)

Test Preparation

Before starting the test:

Cobalt concentration can be determined with the same sample by using Program Number 110.

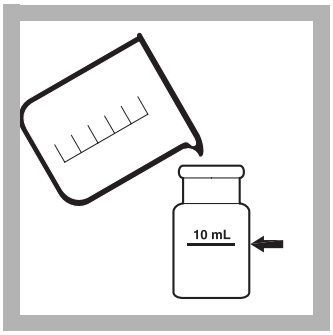
Collect the following items:	**Quantity**
EDTA Powder Pillow	2
Phthalate-Phosphate Reagent Powder Pillows	2
PAN Indicator Solution, 0.3%	1 mL
Deionized Water	25 mL
Sample Cells, 1-inch square, 10-mL	2
Stoppers	2

Note: *Reorder information for consumables and replacement items is on page 5.*

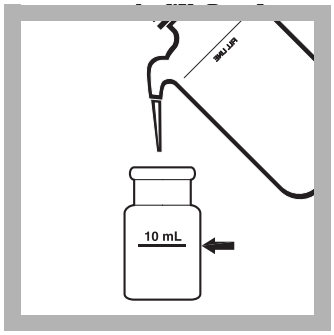
Powder Pillows Method 8150

1. Press **STORED PROGRAMS**.

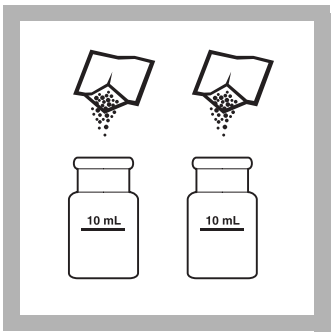
2. Select the test.



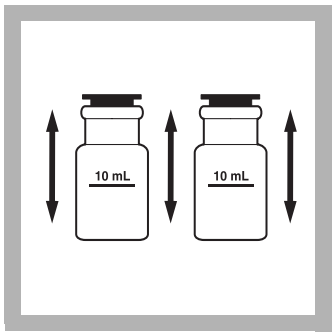
3. Prepared Sample: Fill a square sample cell to the 10-mL mark with sample.



4. Blank Preparation: Fill a second square sample cell to the 10-mL mark with deionized water.

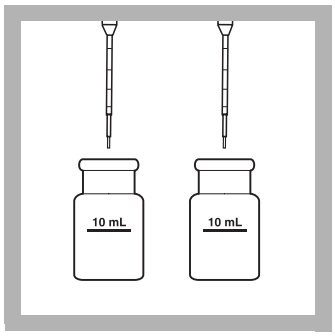


5. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell.

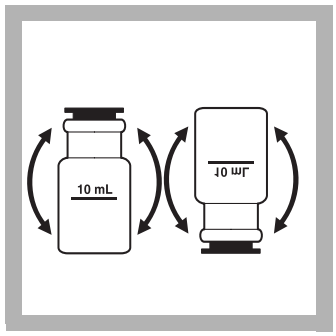


6. Stopper the cells. Immediately shake to dissolve.

If the sample contains iron, make sure that all the powder is dissolved before proceeding to step 7.



7. Using the provided plastic dropper, add 0.5 mL of 0.3% PAN Indicator Solution to each cell.

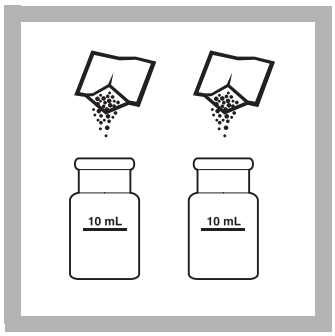


8. Stopper the cells. Invert several times to mix.

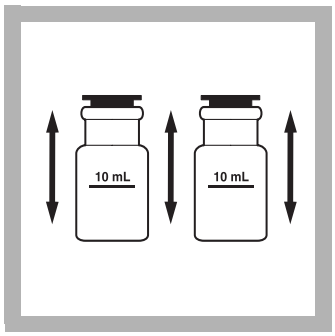
9. Press **TIMER>OK**.

A 15-minute reaction period will begin.

During color development, the sample solution color may vary from yellowish-orange to dark red, depending on the chemical makeup of the sample. The blank should be yellow.

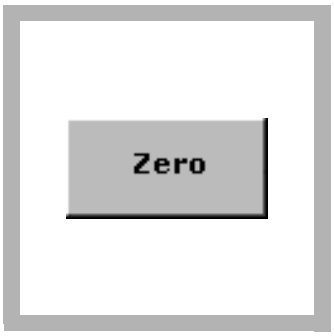


10. When the timer expires, add the contents of one EDTA Reagent Powder Pillow to each cell.



11. Stopper the cells and shake to dissolve.

12. Wipe the blank and insert it into the cell holder with the fill line facing right.



13. Press **ZERO**.

The display will show:

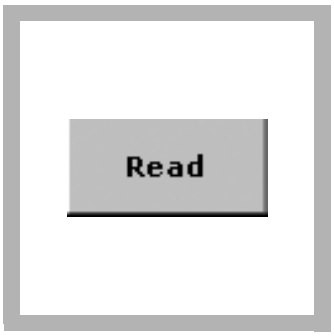
0.000 mg/L Ni

The instrument will zero at 560 and 620 nm.

14. Wipe the sample cell and insert it into the cell holder with the fill line facing right.

The instrument will read the sample at 560 and 620 nm.

Results are in mg/L Ni.



15. Press **READ**. The instrument will read the sample at 560 and 620 nm.

Results are in mg/L Ni and mg/L Co.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Al^{3+}	32 mg/L
Ca^{2+}	1000 mg/L as ($CaCO_3$)
Cd^{2+}	20 mg/L
Cl^{-}	8000 mg/L
Chelating agents	Interfere at all levels. Use either the Digesdahl or vigorous digestion to eliminate this interference.
Cr^{3+}	20 mg/L
Cr^{6+}	40 mg/L
Cu^{2+}	15 mg/L
F^{-}	20 mg/L
Fe^{3+}	10 mg/L
Fe^{2+}	Interferes directly and must not be present.
K^{+}	500 mg/L
Mg^{2+}	400 mg/L
Mn^{2+}	25 mg/L
Mo^{6+}	60 mg/L
Na^{+}	5000 mg/L
Pb^{2+}	20 mg/L
Zn^{2+}	30 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with Nitric Acid*, about 5 mL per liter. Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution*. If the sample is less than 10 °C, warm it to room temperature.

Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct test results for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off a Nickel Voluette® Ampule Standard, 50-mg/L Ni.
5. Prepare three sample spikes. Fill three Mixing Cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Transfer 10 mL of each solution into a 10-mL sample cell and analyze as described in the procedure. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 5.00-mg/L Nickel stock solution by pipetting 5.00 mL of Nickel Standard Solution, 1000-mg/L as Ni, into a 1-L volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily.
2. Prepare a 0.5-mg/L Ni working solution by pipetting 10.0 mL of the 5.00-mg/L nickel stock solution into a 100-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the nickel procedure as described above.
3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 5.

Summary of Method

After buffering the sample and masking any Fe^{3+} with pyrophosphate, the nickel is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator. The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt. The instrument automatically adjusts for cobalt interference by measuring the absorbance of the sample at both 560 nm and 620 nm. This method is unique because both nickel and cobalt can be determined on the same sample.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Nickel Reagent Set (100 Tests), includes:	—	—	26516-00
(2) EDTA Reagent Powder Pillows	2	100/pkg	7005-99
(2) Phthalate-Phosphate Reagent Powder Pillows	2	100/pkg	26151-99
(1) PAN Indicator Solution, 0.3%	1 mL	100 mL MDB	21502-32
Water, deionized	25 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square glass, 10 mL, matched pair	2	2/pkg	24954-02
Stoppers	2	6/pkg	1731-06

Recommended Standards

Description	Unit	Cat. No.
Nickel Standard Solution, 1000-mg/L Ni (NIST)	100 mL	14176-42
Nickel Standard Solution, 50-mg/L Ni (NIST), 10-mL ampules	16/pkg	25576-10

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing	1896-40
Nitric Acid 1:1	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	2450-32

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Updated November 2005 Edition 1

Nickel

★Method 8037 — Heptoxime Method[1]

Powder Pillows — (0.02 to 1.80 mg/L Ni)

Scope and Application: For water, wastewater, and seawater; USEPA accepted for reporting wastewater analyses (digestion required)[2]

[1] Adapted from *Chemie Analytique*, 36 43 (1954)

[2] Procedure is equivalent to Standard Method 3500-Ni D for wastewater

Test Preparation

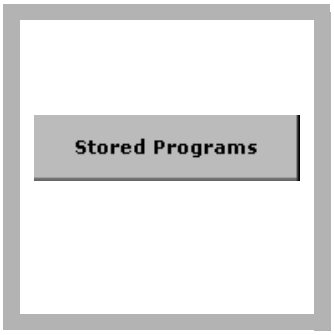
Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Make the cotton plug pea-size. A larger plug will restrict the flow; a smaller plug may become dislodged from the delivery tube of the funnel.
Chloroform (D022) solutions are regulated as hazardous waste by the Federal RCRA. Do not pour these materials down the drain. Water saturated with chloroform, chloroform solutions, and the cotton plug used in the delivery tube of the separatory funnel should be collected for disposal with laboratory solvent wastes. Refer to a current MSDS for safe handling and disposal instructions.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

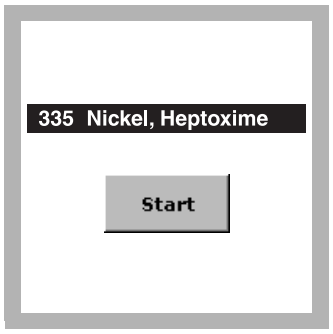
Collect the following items:	**Quantity**
Chloroform, ACS	30 mL
Nickel 1 Reagent Powder Pillow	1
Nickel 2 Reagent Powder Pillow	1
Clippers for Opening Pillows	1
Cotton Balls	varies
Cylinder, graduated. 10-mL	1
Cylinder, graduated, 500-mL	1
Funnel, separatory with stand and stopper	1
Sample Cells, 1-inch square, 25-mL	2

Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows Method 8037



1. Press **STORED PROGRAMS**.



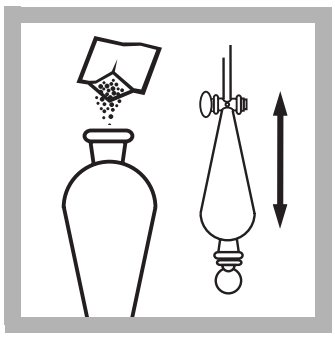
2. Select the test.

3. Measure 300 mL of sample in a 500-mL graduated cylinder. Pour into a 500-mL separatory funnel.

4. Add the contents of one Nickel 1 Reagent Powder Pillow to the funnel. Stopper and invert to mix.

5. Press **TIMER>OK**.

A five-minute reaction period will begin.

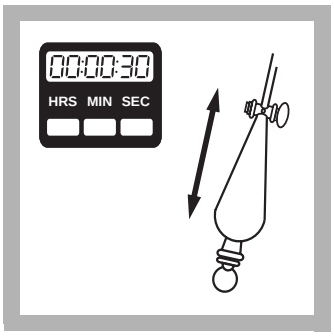


6. When the timer expires, add the contents of one Nickel 2 Reagent Powder Pillow to the funnel. Stopper and invert to mix.

7. Press **TIMER>OK**.

A second five-minute reaction period will begin.

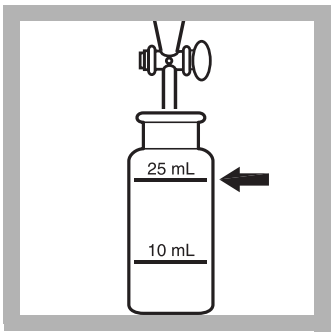
8. When the timer expires, add 10 mL of chloroform. Stopper and invert gently. With the funnel inverted and the tip pointed away from people, open the stopcock to vent.



9. Close the stopcock and invert for 30 seconds.

10. Press **TIMER>OK**.

A third five-minute reaction period will begin. Invert the funnel several times over the five minute period.

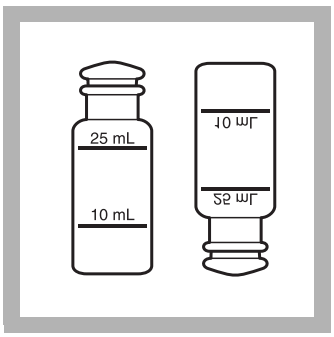


11. Prepared Sample: When the timer expires, wait for the layers to separate. Insert a pea-sized cotton plug into the delivery tube of the funnel. Remove the stopper and drain the chloroform layer (bottom layer) into a 25-mL square sample cell.

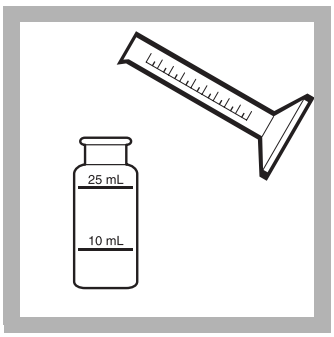
Stopper the funnel.

12. Repeat steps 8 through 11 two additional times with 10-mL volumes of chloroform. The five-minute reaction period is not necessary.

Stopper the funnel and invert to mix. Wait for the layers to separate, and continue.

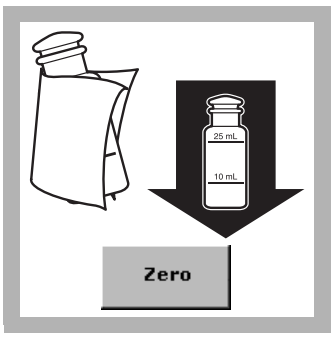


13. Cap the sample cell and invert to mix the extracts. The final volume will be about 25 mL due to the slight solubility of chloroform in water.



14. Blank Preparation: Fill a second square sample cell with 25 mL of chloroform.

Cap the cell.



15. Wipe the blank and insert it into the cell holder with the fill line facing right. Press **ZERO**.

The display will show:

0.00 mg/L Ni

16. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Results are in mg/L Ni.

Interferences

Cobalt, copper, and iron interferences can be overcome by adding additional Nickel 1 Reagent Powder Pillows in step 4. The tolerance limits of these interferences are shown in Table 1.

Table 1 Interfering Substances

Pillows of Nickel 1 Reagent	Tolerance Limit (mg/L):		
	Cobalt	Copper	Iron
1	1	10	20
2	7	16	65
3	13	22	110
4	18	28	155
5	25	35	200

A preliminary acid digestion is required to determine any suspended or precipitated nickel and to eliminate interference by organic matter. To eliminate this interference or to determine total recoverable nickel perform the USEPA approved digestion.

Sample Collection, Storage, and Preservation

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with Nitric Acid*, about 5 mL per liter. Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the sample pH to between 3–8 with 5.0 N Sodium Hydroxide Standard Solution*. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct the test results for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off a Nickel Voluette® Ampule Standard, 300-mg/L Ni.
5. Prepare three sample spikes. Use the TenSette® Pipet to add 0.2 mL, 0.4 mL, and 0.6 mL of standard, respectively, to three 300-mL samples and mix thoroughly.
6. Analyze as described in the procedure. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 6.

Standard Solutions Method

1. Prepare a 10.0-mg/L nickel working standard solution by pipetting 10.0 mL of a Nickel Standard Solution, 1000-mg/L, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Prepare a 1.0-mg/L nickel standard solution by diluting 50.0 mL of the 10-mg/L working standard solution to 500 mL in a volumetric flask. Perform the heptoxime procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Nickel ion reacts with heptoxime to form a yellow-colored complex which is then extracted into chloroform to concentrate the color and enable a more sensitive determination. Chelating agents are added to the sample to overcome the interferences caused by cobalt, copper, and iron. Readings are taken at 430 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Nickel Reagent Set (50 Tests), includes:	—	—	22435-00
(3) Chloroform, ACS	30 mL	500 mL	14458-49
(2) Nickel 1 Reagent Powder Pillows	1	25/pkg	2123-68
(2) Nickel 2 Reagent Powder Pillows	1	25/pkg	2124-68

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers	1	each	968-00
Cotton Balls, absorbent	1	100/pkg	2572-01
Cylinder, graduated, 10-mL	1	each	508-38
Cylinder, graduated, 500-mL	1	each	508-49
Funnel, separatory, 500-mL	1	each	520-49
Ring, support, 4-inch	1	each	580-01
Sample Cells, 1-inch square glass, 25 mL, with cap	2	2/pkg	26126-02
Stand, support, 5 x 8-inch base	1	each	563-00

Recommended Standards

Description	Unit	Cat. No.
Nickel Standard Solution, 1000-mg/L Ni (NIST)	100 mL	14176-42
Nickel Standard Solution, 300-mg/L Ni (NIST), 10-mL Voluette® Ampules	16/pkg	14266-10
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing	1896-40
Nitric Acid 1:1	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	2450-53

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Updated November 2005 Edition 1

Nickel

Method 10220
TNTplus™ 856

Dimethylglyoxime Method
(0.1 to 6.0 mg/L Ni)

Scope and Application: For wastewater, drinking water and process analysis.

Test Preparation

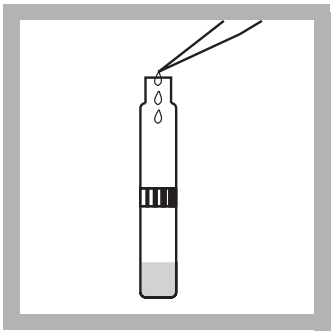
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample pH is 3–10.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F).
Nickel concentration greater that the measuring range cause precipitation in the vial. In such cases, the water sample must first be diluted with deionized water.
Undissolved nickel or nickel contained in complexes can only be determined after digestion with Metals Prep Set TNT 890.
TNTplus methods are activated directly from the Main Menu when the sample vial is inserted into the sample cell holder.

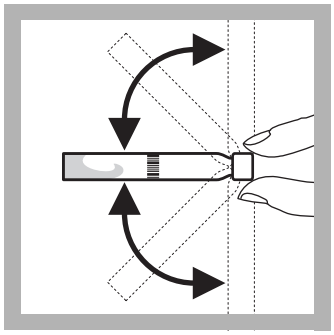
Collect the following items:	**Quantity**
Light Shield	1
Nickel TNT856 Reagent Set	1
Pipettor, variable, 100–1000 µL	1
Pipettor tips, for 100–1000 µL pipettor	1
Pipettor, variable, 1–5 mL	1
Pipettor, tips for 1–5 mL pipettor	1

Note: *Reorder information for consumables and replacement items is on page 4.*

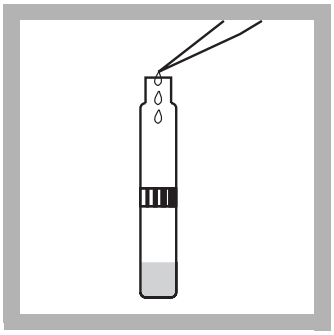
TNTplus Method 10220



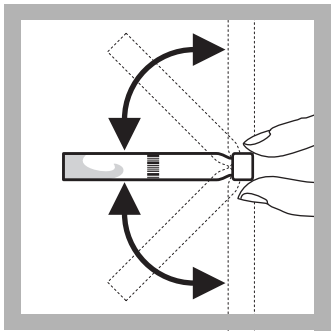
1. Pipet 2.0 mL of sample into the vial.



2. Cap the vial and invert 2–3 times until the freeze-dried contents in the vial are completely dissolved.



3. Pipet 0.2 mL (200 µL) of Solution A into the vial.



4. Cap and invert the vial 2–3 times.

5. Wait three minutes.

Install the Light Shield in Cell Compartment #2.

6. Thoroughly clean the outside of the vial. Insert the prepared vial into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L Ni

No instrument zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 6. Press **OPTIONS**>**MORE**>**REAGENT BLANK**. Select **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity the procedure is repeated and the color forming reagent that is present in Solution A is not added.

To determine the sample blank run the procedure as given, but substitute 0.2 mL of deionized water in place of the 0.2 mL of Solution A in step 3. Use the red stopper to cap the sample vial. The value obtained in step 6 is then subtracted from the value obtained on the original sample to give the corrected sample concentration.

Alternatively, samples that contain only turbidity may be first filtered through a membrane filter and then analyzed. Report the results as soluble nickel.

Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. The cumulative effects and influence of other ions has not been determined. Undissolved nickel or nickel contained in complexes can only be determined after digestion with Metals Prep Set TNT 890.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels
K^+, Na^+, Cl^-, SO_4^{2-}	1000 mg/L
NH_4^+, NO_3^-, Ca^{2+}, PO_4^{3-}, CO_3^{2-}	500 mg/L
Cr^{6+}, Zn^{2+}, F^-, NO_2^-	50 mg/L
Al^{3+}, Cr^{3+}, Cd^{2+}, Co^{2+}, Sn^{2+}, Pb^{2+}	10 mg/L
Fe^{2+}, Fe^{3+}, Mn^{2+}, Cu^{2+}, Mg^{2+}, Hg^{2+}	5 mg/L
Ag^+	1 mg/L

Sample Collection, Preservation, and Storage

Collect samples in an acid-washed glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Adjust the pH between 3–10 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

To check the accuracy of the method prepare a 4.0 mg/L nickel standard solution by pipetting 1.0 mL of a 1000 mg/L nickel standard solution into a 250 mL volumetric flask. Dilute to volume with deionized water.Prepare this solution daily. Use 2.0 mL of this standard in place of the sample in the procedure.

Summary of Method

In the presence of an oxidizing agent, nickel ions react with dimethylglyoxime in an alkaline solution to form an orange-brown-colored complex. Test results are measured at 463 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Nickel TNT 856 Reagent Set	1	25/pkg	TNT856

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 1–5 mL	1	each	27951-00
Pipettor Tips, for 27951-00 pipettor	1	100/pkg	27952-00
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27950-00

Recommended Reagents and Standards

Description	Unit	Cat. No.
Nickel Standard Solution, 1000 mg/L	100 mL	14176-42
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	2450-26
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
Filter Holder, glass for vacuum filtration (SUVA)	each	2340-00
Filter, membrane, 47-00; 0.45 -micron, hydrophilic, polyethersulfone for SUVA	each	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	each	546-53
Flask, volumetric 250 mL	each	14574-46
Pipet, volumetric 1.0 mL	each	14515-35
Metals Prep Set TNT 890	each	TNT890
Test Tube Rack for 13-mm vials	each	24979-00
Tubing, rubber	12 ft	560-19

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Updated November 2005 Edition 1

Nitrate

Method 8039

Powder Pillows or AccuVac® Ampuls

Cadmium Reduction Method

HR (0.3 to 30.0 mg/L NO_3^-–N)

Scope and Application: For water, wastewater, and seawater.

Test Preparation

Before starting the test:

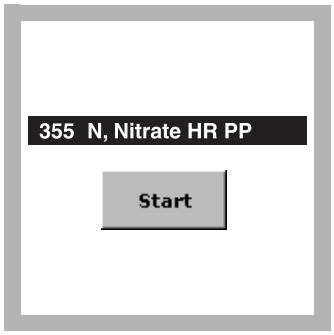
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
A deposit of unoxidized metal will remain after the NitraVer® 5 dissolves. The deposit will not affect results.
This method is technique-sensitive. Shaking time and technique influence color development. For most accurate results, make successive tests on a 10-mg/L Nitrate Nitrogen Standard solution. Adjust shaking time and technique to obtain the correct result.
Rinse the sample cell immediately after use to remove all cadmium particles. Prepared samples will contain cadmium and must be disposed of according to Federal, State, and local hazardous waste regulations. Refer to the current MSDS for safe handling and disposal instructions.

Collect the following items:	**Quantity**
Powder Pillow Test:	
NitraVer® 5 Nitrate Reagent Powder Pillow	1
Sample Cells, 1-inch square, 10-mL, with stopper	2
AccuVac Test:	
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cell, 10-mL round, with cap	1

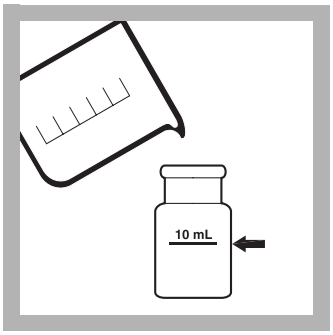
Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows — Method 8039

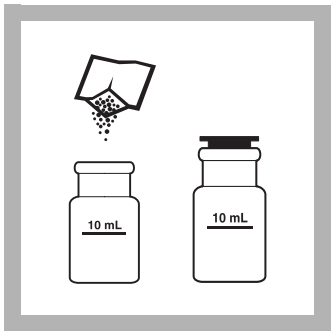
1. Press **STORED PROGRAMS**.



2. Select the test.



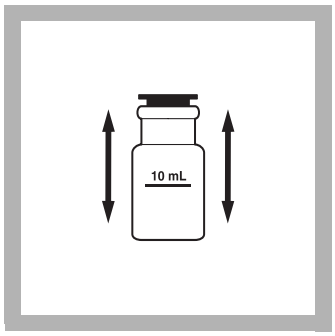
3. Fill a square sample cell with 10 mL of sample.



4. Prepared Sample: Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow. Stopper.

5. Press **TIMER>OK**.

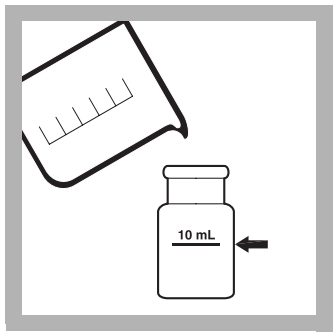
A one-minute reaction period will begin.



6. Shake the cell vigorously until the timer expires.

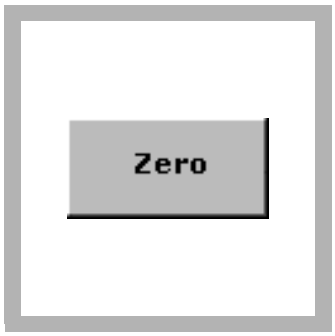
7. When the timer expires, press **TIMER>OK** again. A five-minute reaction period will begin.

An amber color will develop if nitrate is present.



8. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of sample.

9. Wipe the blank and insert it into the cell holder with the fill line facing right.



10. Press **ZERO**.

The display will show:

0.0 mg/L NO_3^-–N

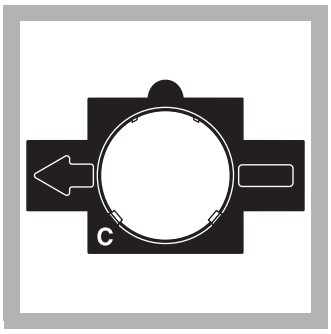
11. Within one minute after the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing right.

12. Press **READ**.

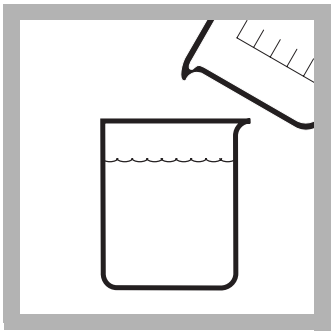
Results are in mg/L NO_3^-–N.

AccuVac® Ampul — Method 8039

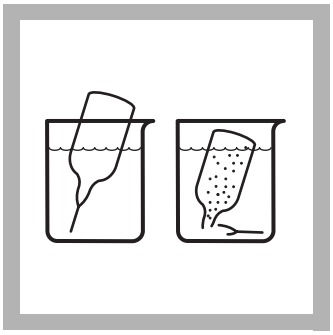
1. Select the test



2. Insert Adapter C.



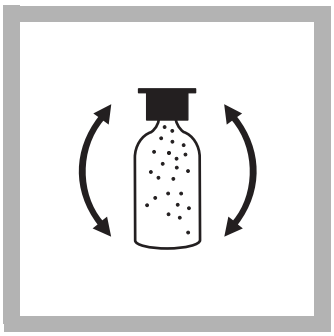
3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.



4. Tap the bottom of a NitraVer 5 Nitrate AccuVac® Ampul on a hard surface to dislodge powder. Fill the Ampul with sample. Keep the tip immersed while the Ampul fills completely. Insert a stopper over the Ampul tip.

5. Press **TIMER>OK**.

A one-minute reaction period will begin.



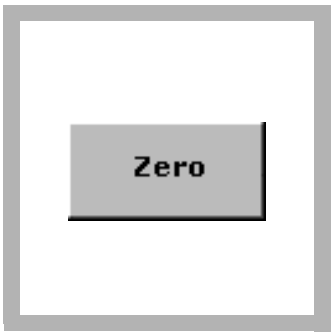
6. Invert the Ampul a full rotation once per second until the timer expires.

7. When the timer expires, press **TIMER>OK** again. A five-minute reaction period will begin. Do not agitate or disturb the sample during this time.

An amber color will develop if nitrate is present.

8. Blank Preparation: When the timer expires, fill a round sample cell with 10 mL of sample.

9. Wipe the blank and insert it into the cell holder.

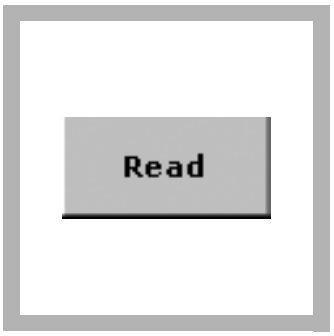


10. Press **ZERO**.

The display will show:

0.0 mg/L NO_3^-–N

11. Within one minute after the timer expires, wipe the Ampul and insert into the cell holder.



12. Press **READ**.

Results are in mg/L NO_3^-–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	Interferes at all levels
Nitrite	Interferes at all levels Compensate for nitrite interference as follows: Before performing *step 3*, add 30-g/L Bromine Water[1] dropwise to the sample until a yellow color remains. Add one drop of 30-g/L Phenol Solution[1] to destroy the color. Proceed with step 3. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels

[1] See Optional Reagents and Apparatus on page 6.

Sample Collection, Storage, and Preservation

More reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 24 hours at 4 °C. To preserve samples for longer periods, add 2 mL of Concentrated Sulfuric Acid (H_2SO_4)* per liter and store at 4 °C.

Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives. Correct the test result for volume additions by dividing the total volume (acid + base + sample) by the original sample volume and multiplying the test result by this factor.

* See Optional Reagents and Apparatus on page 6.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Nitrate Nitrogen Voluette® Ampule Standard, 250-mg/L NO_3^-–N.

5. Prepare three sample spikes. Fill three sample cells* with 10 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

Note: *For AccuVac® Ampuls, fill three mixing cylinders* with 50 mL of sample and spike with 0.4 mL, 0.8 mL, and 1.2 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers*. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. To test accuracy, use a 10.0-mg/L Nitrate Nitrogen Standard Solution in place of the sample and perform the procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical forms). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Cadmium metal reduces nitrates in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt. The salt couples with gentisic acid to form an amber colored solution. Test results are measured at 500 nm.

* See Optional Reagents and Apparatus on page 6.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
NitraVer® 5 Nitrate Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	21061-69
OR			
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1	25/pkg	25110-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper, Neoprene, solid, size #2	2	12/pkg	14808-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3^-–N	500 mL	307-49
Nitrate Nitrogen Standard Solution Ampule, 250-mg/L NO_3^-–N	16/pkg	25577-10
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Wastewater Influent Standard, Mixed Parameter, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Bromine Water	2211-20
Cylinder, mixing, 50 mL	20886-41
Phenol Solution	2112-20
Sodium Hydroxide Standard Solution, 5.0 N	2450-26
Sulfuric Acid, concentrated	979-49

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Updated November 2005 Edition 1

Nitrate

Method 8192
Powder Pillows

Cadmium Reduction Method
LR (0.01 to 0.50 mg/L NO_3^-–N)

Scope and Application: For water, wastewater, and seawater

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
A deposit of unoxidized metal will remain after the NitraVer® 6 dissolves. The deposit will not affect results.
Shaking time and technique influence color development. Analyze a standard solution several times and adjust the shaking time to obtain the correct result. Use this time for analyzing samples.
Rinse the sample cell and mixing cylinder immediately after use to remove all cadmium particles.
Properly dispose of the used sample. Prepared samples contain cadmium and must be disposed of according to Federal, State, and local hazardous waste regulations. Refer to the current MSDS for safe handling and disposal information.

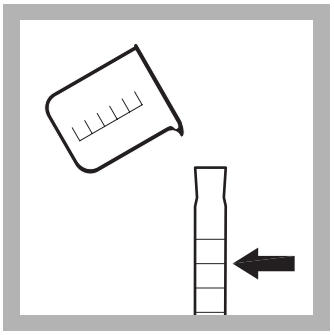
Collect the following items:	**Quantity**
NitraVer® 6 Nitrate Reagent powder pillow	1
NitraVer® 3 Nitrite Reagent powder pillow	1
Cylinder, graduated, mixing, 25-mL	1
Sample Cells, 1-inch square, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 5.*

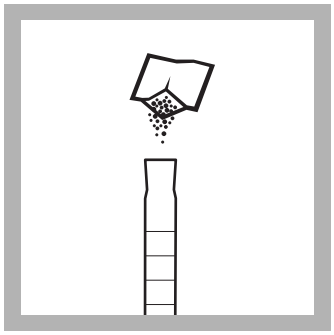
Powder Pillows — Method 8192

1. Press **STORED PROGRAMS**.

2. Select the test.



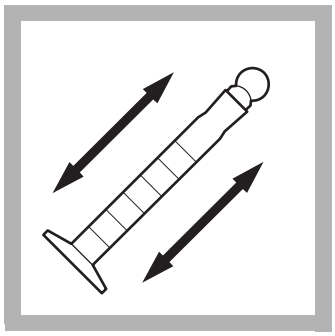
3. Fill a 25-mL graduated mixing cylinder with 15 mL of sample.



4. Add the contents of one NitraVer 6 Reagent Powder Pillow to the cylinder. Stopper.

5. Press **TIMER>OK**.

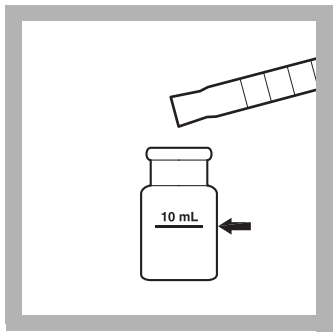
A 3-minute reaction time will begin.



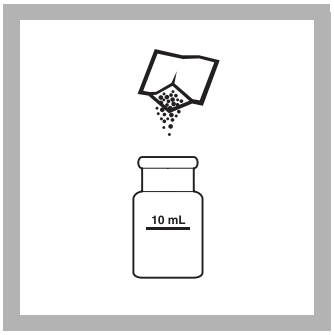
6. Shake the cylinder vigorously during the three-minute timer.

7. When the timer expires, press **TIMER>OK** again.

A 2-minute reaction period will begin.



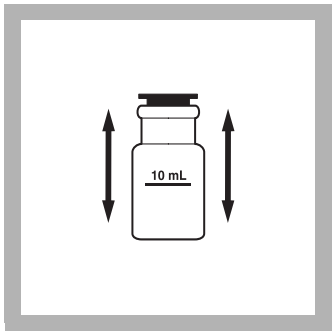
8. When the timer expires, carefully pour 10 mL of the sample into a clean square sample cell. Do not transfer any cadmium particles to the sample cell.



9. Prepared Sample: Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell.

10. Press **TIMER>OK**.

A 30-second reaction time will begin.

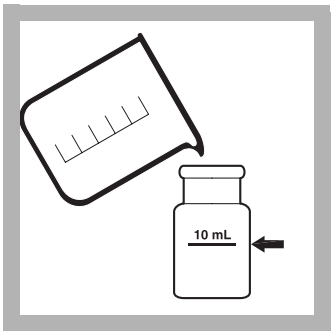


11. Cap and shake the sample cell gently during the 30-second timer.

A pink color will develop if nitrate is present.

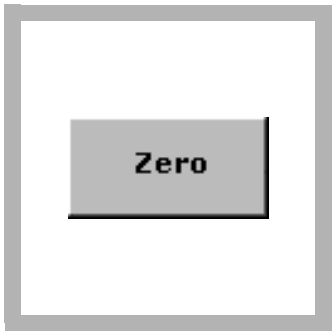
12. Press **TIMER>OK**.

A 15-minute reaction period will begin.



13. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of original sample.

14. Insert the blank into the cell holder with the fill line facing right.



15. Press **ZERO**.

The display will show:

0.00 mg/L NO_3^-–N

16. Insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L NO_3^-–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium	100 mg/L
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels: This method measures both the nitrate and nitrite in the sample. If nitrite is present, the nitrite nitrogen test (Program #371) should be done on the sample. Pretreat the nitrate nitrogen sample with the following pretreatment. Then subtract the amount of nitrite found from the results of the LR nitrate nitrogen test. Add 30-g/L Bromine Water[1] dropwise to the sample in step 4 until a yellow color remains. Mix after each drop. Add one drop of 30-g/L Phenol Solution[1] to destroy the color. Proceed with the LR Nitrate procedure.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interferes at all levels

[1] See Optional Reagents and Apparatus on page 5.

Sample Collection, Storage, and Preservation

More reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 48 hours at 4 °C. To preserve samples for longer periods, add 2 mL of Concentrated Sulfuric Acid* per liter and store at 4 °C.

Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives. Correct the test result for volume additions by dividing the total volume (acid + base + sample) by the original sample volume and multiplying the test result by this factor.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Nitrate Nitrogen Voluette® Ampule Standard, 12.0-mg/L NO_3^-–N*.

* See Optional Reagents and Apparatus on page 5.

5. Prepare three sample spikes. Fill three mixing cylinders* with 15 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. To test accuracy, use a 0.40-mg/L NO_3^-–N standard in place of the sample and perform the procedure as described. Prepare this standard by diluting 4.00 mL of a 10-mg/L Nitrate Nitrogen Standard Solution to 100.0 mL with deionized water.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Cadmium metal reduces nitrates in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt. The salt couples with chromotropic acid to form a pink-colored product. Test results are measured at 507 nm.

* See Optional Reagents and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Low Range Nitrate Reagent Set (100 tests), includes:	—	—	24298-00
NitraVer® 6 Nitrate Reagent Powder Pillows	1	100/pkg	21072-49
NitriVer® 3 Nitrite Reagent Powder Pillows	1	100/pkg	21071-69

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated mixing, 25-mL	1	each	20886-40
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Flask, volumetric, Class A, 100-mL	each	14574-42
Nitrate Nitrogen Standard Solution, 10.0-mg/L NO_3^-–N	500 mL	307-49
Nitrate Nitrogen Standard Solution, Voluette® Ampule, 12-mg/L NO_3^-–N	16/pkg	14333-10
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 4.00 mL	each	14515-04
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Bromine Water, 30 g/L	2211-20
Phenol Solution, 30-g/L	2112-20
Sodium Hydroxide Standard Solution, 5.0 N	2450-53
Sulfuric Acid, concentrated	979-49

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Updated November 2005 Edition 1

Nitrate

Method 10020

Test ‘N Tube™ Vials

Chromotropic Acid Method

HR (0.2 to 30.0 mg/L NO_3^-–N)

Scope and Application: For water and wastewater

Test Preparation

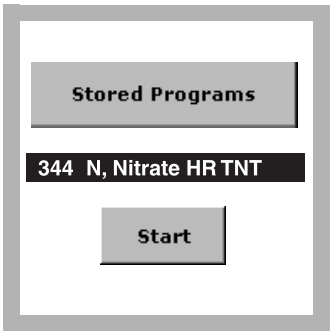
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water (nitrate-free) in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
This test is technique-sensitive. Invert the vials as described here to avoid low results: Hold the vial in a vertical position with the cap pointing up. Turn the vial upside-down. Wait for all of the solution to flow down to the cap. Pause. Return the vial to an upright position. Wait for all the solution to flow to the bottom of the vial. This process equals one inversion.

Collect the following items:	**Quantity**
Test ‘N Tube™ NitraVer® X Reagent Set	1
Funnel, micro, poly	1
Light Shield	1
Pipet, TenSette®, 0.01 to 1.0 mL, plus tips	1
Test Tube Rack	1–3

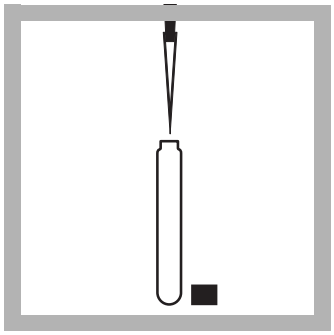
Note: *Reorder information for consumables and replacement items is on page 4.*

Test ‘N Tube — Method 10020

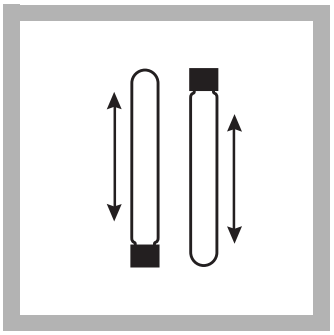


1. Select the test.

Install the Light Shield in Cell Compartment #2.

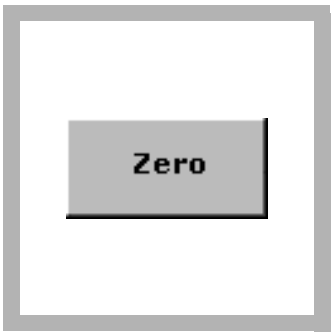


2. Blank Preparation: Remove the cap from a NitraVer X Reagent A Test ‘N Tube vial and add 1.00 mL of sample.



3. Cap the tube and invert ten times to mix.

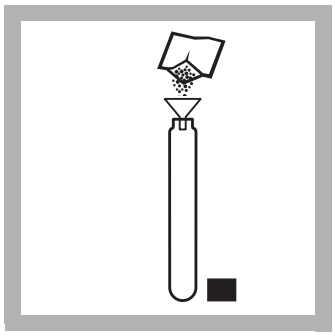
4. Wipe the blank and insert it into the 16 mm round cell holder.



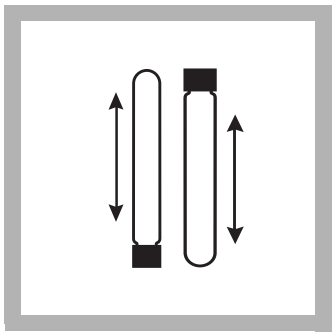
5. Press **ZERO**.

The display will show:

0.0 mg/L NO_3^-–N



6. Prepared Sample: Remove the vial from the instrument. Using a funnel, add the contents of one NitraVer X Reagent B Powder Pillow to the vial.



7. Cap and invert ten times to mix.

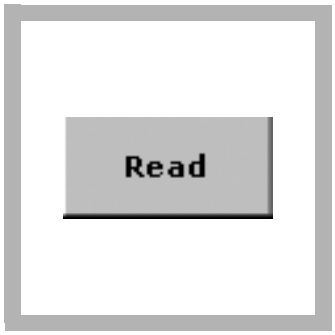
Some solid matter will not dissolve.

8. Press **TIMER>OK**.

A five-minute reaction period will begin. Do not invert the vial again.

A yellow color will develop if nitrate is present.

9. Within five minutes after the timer expires, wipe the prepared sample and insert it into the cell holder.



10. Press **READ**.

Results are in mg/L NO_3^-–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Barium	A negative interference at concentrations greater than 1 mg/L.
Chloride	Does not interfere below 1000 mg/L.
Nitrite	A positive interference at concentrations greater than 12 mg/L. Remove nitrite interference up to 100 mg/L by adding 400 mg (one full 0.5 g measuring spoon) of Urea[1] to 10 mL of sample. Swirl to dissolve. Proceed with the nitrate test as usual.
Copper	Positive at all levels.

[1] See Optional Reagents and Apparatus on page 4.

Sample Collection, Preservation, and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with Concentrated Sulfuric Acid, ACS* (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a High Range Nitrate Nitrogen Voluette® Ampule Standard, 500 mg/L NO_3^-–N.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. To test accuracy, use a 10.0-mg/L Nitrate Nitrogen Standard Solution in place of the sample and perform the procedure as described above.

2. To adjust the calibration curve using the reading obtained with the 10.0-mg/L Nitrate Nitrogen Standard Solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 4.

Summary of Method

Nitrate in the sample reacts with chromotropic acid under strongly acidic conditions to yield a yellow product with a maximum absorbance at 410 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Test 'N Tube™ NitraVer® X Nitrate Reagent Set	1	50/pkg	26053-45

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Funnel, micro, poly	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	varies	50/pkg	21856-96
Test Tube Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrate Nitrogen Standard Solution, 10-mg/L: N	500 mL	307-49
Nitrate Nitrogen Standard Solution, Voluette® Ampule, 500-mg/L N	16/pkg	14260-10
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing, 25 mL	20886-40
Sodium Hydroxide, 5.0 N	2450-26
Sulfuric Acid ACS, Concentrated	979-49
Urea	11237-26

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Updated November 2005 Edition 1

Nitrate

Method 10206 **Dimethylphenol Method**

TNTplus 836 **HR (5–35 mg/L NO_3^-–N or 22–155 mg/L NO_3)**

Scope and Application: For wastewater, drinking water, surface water, and process water

Test Preparation

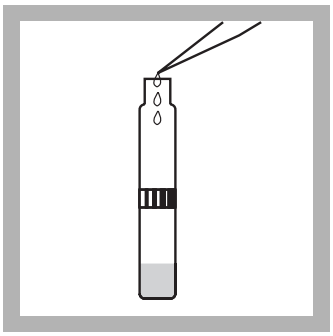
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
If the test is not performed at the recommended temperature an incorrect result may be obtained. Analyze samples as soon as possible.
Recommended sample pH is 3–10.
Recommended sample and reagent temperature is 20–23 °C (68–73.4 °F).
Recommended reagent storage is 15–25 °C (59–77 °F).
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.

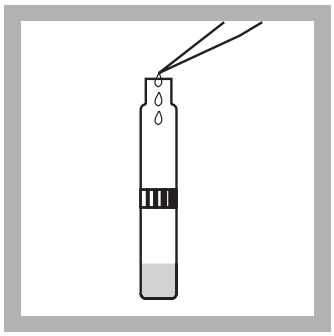
Collect the following items:	**Quantity**
Light Shield	1
Nitrate HR TNT 836 Reagent Set	1
Pipettor for 0.2 mL Sample	1
Pipettor Tip	2

Note: *Reorder information for consumables and replacement items is on page 4.*

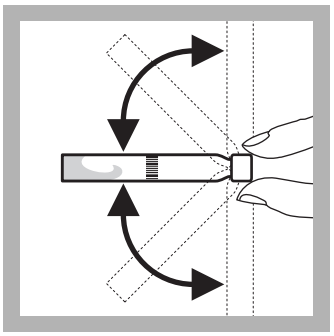
TNTplus Method 10206



1. Pipet 0.2 mL (200 µL) of sample into the reagent vial.



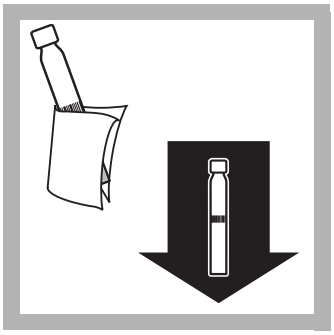
2. Pipet 1.0 mL (1000 µL) of Solution A into the vial.



3. Cap and invert the reaction tube 2–3 times until no more streaks can be seem in the vial solution.

4. Wait 15 minutes.

Install the Light Shield in Cell Compartment #2.



5. After the timer expires wipe the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test. No Zero is required.

Results are in mg/L NO_3–N.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 5. Press **OPTIONS**>**MORE**>**REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity the procedure is repeated and the color forming reagent that is present in Solution A is not added.

To determine the sample blank run the procedure as given, but substitute 1.0 mL of deionized water in place of the 1.0 mL of Solution A in step 2. Use the original cap to cap the sample vial. The value obtained in step 5 is then subtracted from the value obtained on the original sample to give the corrected sample concentration.

Alternatively, samples that contain only turbidity may be first filtered through a membrane filter and then analyzed.

Samples without color or turbidity do not require sample blanks.

Interferences

The items listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects or influence of other ions has not been determined. High loads of oxidizable organic substances (COD) cause the reagent to change color and to give high-bias results. The test can thus only be used for wastewater analyses if the COD is less than 500 mg/L. Measurement results can be verified using sample dilutions or standard additions.

Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results). Add 50 mg of sulfamic acid (amidosulfonic acid) to 5.0 mL of sample, dissolve and wait for 10 minutes. Analyze the prepared sample as described in the procedure above.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
K^+	2000 mg/L
Na^+	1500 mg/L
Cl^-	1000 mg/L
COD	500 mg/L
Ca^{2+}	250 mg/L
Ag^+	100 mg/L
Pb^{2+}, Zn^{2+}, Ni^{2+}, Fe^{3+}, Cd^{2+}, Cu^{2+}	50 mg/L
Fe^{2+}	20 mg/L
Co^{2+}	10 mg/L
Cr^{6+}	5 mg/L
NO_2^-	2 mg/L

Sample Collection, Preservation, and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible to prevent bacteria degradation of the nitrate. If immediate analysis is not possible, store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to 20–23 °C before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with Sulfuric Acid, ACS* (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to 20–23 °C and neutralize with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of method with a 10 mg/L nitrate nitrogen standard. Use 0.2 mL of this 10 mg/L standard in place of the sample in step 2.

2. Alternately, use 0.2 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 10 mg/L nitrate nitrogen combined with ammonia, phosphate, sulfate and organic material.

* See Optional Reagents and Apparatus on page 4.

Summary of Method

Nitrate ions in solutions containing sulfuric and phosphoric acids react with 2,6-dimethylphenol to form 4-nitro-2,6-dimethylphenol. Test results are measured at 345 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Nitrate TNTplus, HR TNT 836	1	25/pkg	TNT836

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27950-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrate Nitrogen Standard Solution, 10-mg/L NO_3–N	500 mL	307-49
Nitrate Nitrogen Standard Solution, 1000 mg/L NO_3–N	500 mL	12792-49
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Balance, AccuLab VI–Series, 120 g capacity	26947-00
Bottle, sampling, low density poly, w/cap, 500 mL, 12/pkg	20870-79
Filter Holder, glass for vacuum filtration (SUVA)	2340-00
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	546-53
Sodium Hydroxide, 5.0 N, 50 mL	2450-26
Sulfamic Acid, 113 g	2344-14
Sulfuric Acid ACS, concentrated, 500 mL	979-49
Test Tube Rack for 13-mm vials	24979-00
Tubing, rubber, 12-ft	560-19

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Updated November 2005 Edition 1

Nitrate

Method 10206 — Dimethylphenol Method

TNTplus 835

LR (0.23 to 13.50 mg/L NO_3^-–N or 1.00 to 60.00 mg/L NO_3)

Scope and Application: For wastewater, drinking water, surface water, and process water

Test Preparation

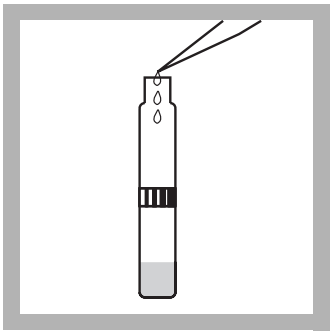
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
If the test is not performed at the recommended temperature an incorrect result may be obtained. Analyze samples as soon as possible.
Recommended sample pH is 3–10.
Recommended sample and reagent temperature is 20–23 °C (68–73.4 °F).
Recommended reagent storage is 15–25 °C (59–77 °F).
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.

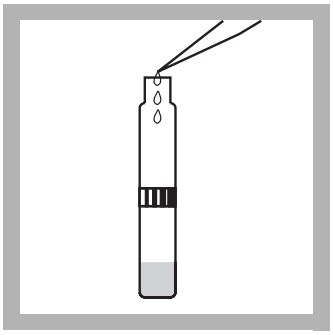
Collect the following items:	**Quantity**
Light Shield	1
Nitrate LR TNT 835 Reagent Set	1
Pipettor for 0.2 mL Sample	1
Pipettor Tip	2

Note: *Reorder information for consumables and replacement items is on page 4.*

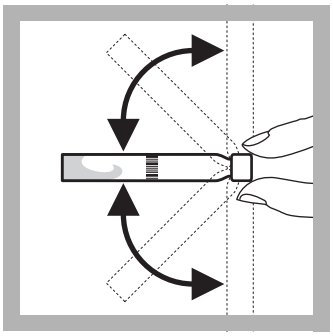
TNTplus — Method 10206



1. Pipet 1.0 mL (1000 µL) of sample into the reagent vial.



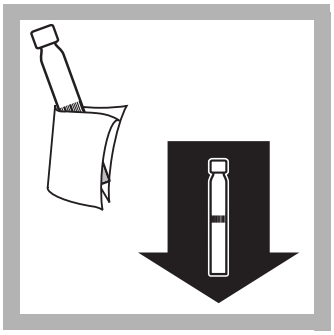
2. Pipet 0.2 mL (200 µL) of Solution A into the vial.



3. Cap and invert the reaction tube 2–3 times until no more streaks can be seem in the reaction tube solution.

4. Wait 15 minutes.

Install the Light Shield in Cell Compartment #2.



5. After the timer expires wipe the vial and insert the prepared vial into the cell holder. The instrument reads the barcode, then selects and performs the correct test. No Zero is required.

Results are in mg/L NO_3–N

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 5. Press **OPTIONS**>**MORE**>**REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity the procedure is repeated and the color forming reagent that is present in Solution A is not added.

To determine the sample blank run the procedure as given, but substitute 0.2 mL of deionized water in place of the 0.2 mL of Solution A in step 2. Use the original cap to cap the sample vial. The value obtained in step 5 is then subtracted from the value obtained on the original sample to give the corrected sample concentration.

Alternatively, samples that contain only turbidity may be first filtered through a membrane filter and then analyzed.

Samples without color or turbidity do not require sample blanks.

Interferences

The items listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. The cumulative effects and influence of other ions has not been determined. High loads of oxidizable organic substances (COD) cause the reagent to change color and to give high-bias results. The test can thus only be used for wastewater analyses if the COD is less than 500 mg/L. Measurement results can be verified using sample dilutions or standard additions.

Nitrite concentrations of more than 2.0 mg/L interfere (high-bias results). Add 50 mg of sulfamic acid (amidosulfonic acid) to 5.0 mL of sample, dissolve and wait for 10 minutes. Analyze the prepared sample as described in the procedure above.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
K^+, Na^+, Cl^-	500 mg/L
Ag^+	100 mg/L
Pb^{2+}, Zn^{2+}, Ni^{2+}, Fe^{3+}, Cd^{2+}, Sn^{2+}, Ca^{2+}, Cu^{2+}	50 mg/L
Fe^{2+}, Co^{2+}	10 mg/L
Cr^{6+}	5 mg/L
NO_2^-	2 mg/L

Sample Collection, Preservation, and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible to prevent bacteria degradation of the nitrate. If immediate analysis is not possible, store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to 20–23 °C before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with Sulfuric Acid, ACS* (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to 20–23 °C and neutralize with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of method with a 10 mg/L nitrate nitrogen standard. Use 1.0 mL of this 10 mg/L standard in place of the sample in step 1.

2. Alternately, use 1.0 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 1. This standard contains 10 mg/L nitrate nitrogen combined with ammonia, phosphate, sulfate, and organic material.

Summary of Method

Nitrate ions in solutions containing sulfuric and phosphoric acids react with 2,6-dimethylphenol to form 4-nitro-2,6-dimethylphenol. Test results are measured at 345 nm.

* See Optional Reagents and Apparatus on page 4.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Nitrate TNTplus, LR TNT 835	1	25/pkg	TNT835

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27950-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrate Nitrogen Standard Solution, 10-mg/L	500 mL	307-49
Nitrate Nitrogen Standard Solution, 1000 mg/L	500 mL	12792-49
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Balance, AccuLab VI–Series, 120 g capacity	26947-00
Bottle, sampling, low density poly, w/cap, 500 mL, 12/pkg	20870-79
Filter Holder, glass, for vacuum filtration (SUVA)	2340-00
Filter, membrane, 47-00; 0.45-micron, hydrophilic, polyethersulfone	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	546-53
Sodium Hydroxide, 5.0 N, 50 mL	2450-26
Sulfamic Acid, 113 g	2344-14
Sulfuric Acid ACS, concentrated, 500 mL	979-49
Test Tube Rack for 13-mm vials	24979-00
Tubing, rubber, 12-ft	560-19

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Updated November 2005 Edition 1

Nitrate

Method 8171

Powder Pillows or AccuVac® Ampuls

Cadmium Reduction Method

MR (0.1 to 10.0 mg/L NO_3^-–N)

Scope and Application: For water, wastewater, and seawater

Test Preparation

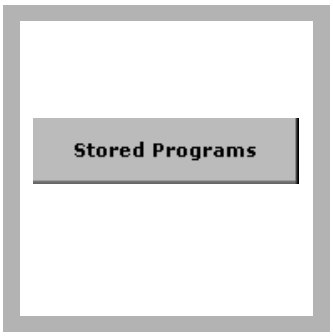
Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.
A deposit of unoxidized metal will remain after the NitraVer® 5 dissolves. The deposit will not affect results.
This method is technique-sensitive. Shaking time and technique influence color development. For most accurate results, make successive tests on a 10.0-mg/L Nitrate Nitrogen Standard solution. Adjust shaking times to obtain the correct result.
Rinse the sample cell immediately after use to remove all cadmium particles. Retain the used sample for proper hazardous waste disposal for cadmium.
Prepared samples will contain cadmium and must be disposed of according to Federal, State, and local hazardous waste regulations. Refer to the current MSDS for safe handling and disposal instructions.

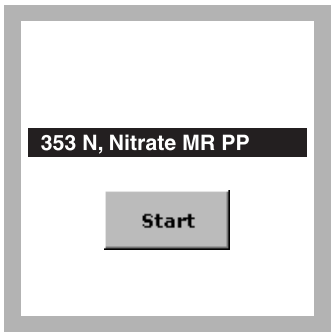
Collect the following items:	**Quantity**
Powder Pillow Test:	
NitraVer® 5 Nitrate Reagent Powder Pillow	1
Sample Cells, 1-inch square, 10-mL	2
Stopper, Neoprene #2, solid	2
AccuVac Test:	
Collect at least 40 mL of sample in a 50-mL beaker	40 mL
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1
Beaker, 50-mL (AccuVac test)	1
Sample Cell, 10-mL	1

Note: *Reorder information for consumables and replacement items is on page 6.*

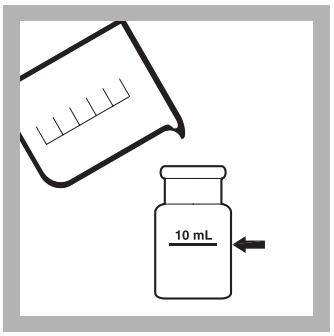
Powder Pillows — Method 8171



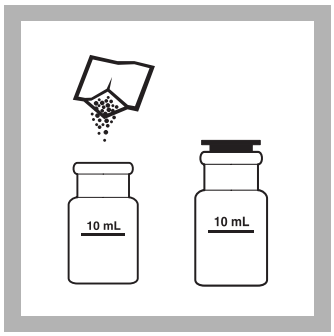
1. Press **STORED PROGRAMS**.



2. Select the test.



3. Fill a square sample cell with 10 mL of sample.



4. Prepared Sample: Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow. Insert a stopper into the cell.

5. Press **TIMER>OK**.

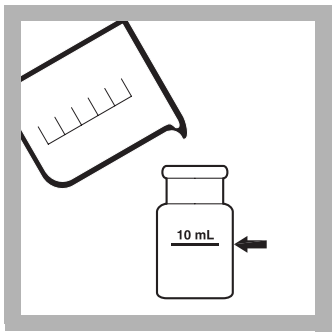
A one-minute reaction period will begin.

Shake the cell vigorously until the timer expires.

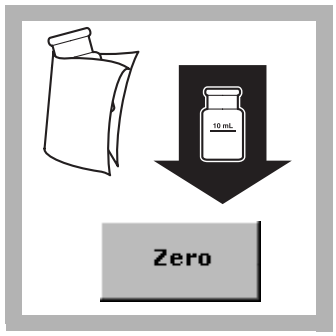
6. When the timer expires, press **TIMER>OK**.

A five-minute reaction period will begin.

An amber color will develop if nitrate is present.



7. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of sample.

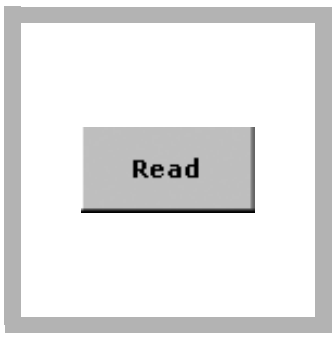


8. Insert the blank into the cell holder with the fill line facing right. Press **ZERO**.

The display will show:

0.0 mg/L NO_3^-–N

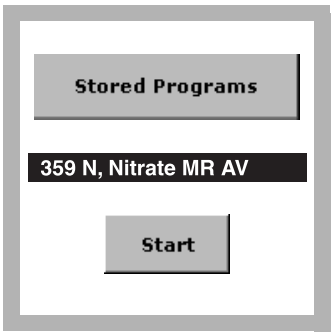
9. Within two minutes after the timer expires, insert the prepared sample into the cell holder with the fill line facing right.



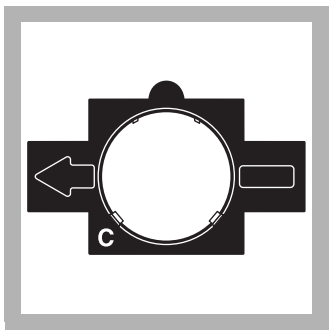
10. Press **READ**.

Results are in mg/L NO_3^-–N.

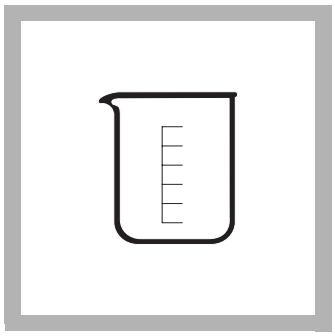
AccuVac® Ampul — Method 8171



1. Select the test.



2. Insert Adapter C.

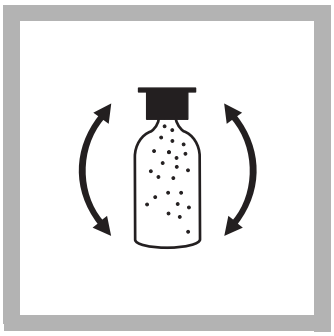


3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

4. Fill a NitraVer 5 Nitrate AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills completely. Place a stopper over the Ampul tip.

5. Press **TIMER>OK**.

A one-minute reaction period will begin.

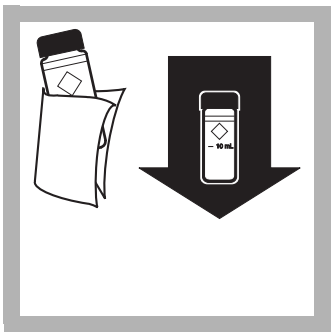


6. Invert the Ampul 48–52 times as the timer counts down.

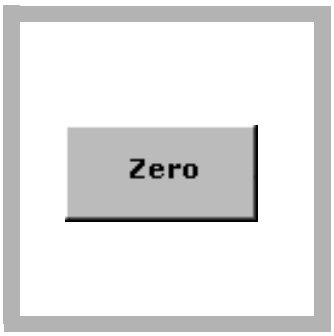
7. When the timer expires, press **TIMER>OK**.

A five-minute reaction period will begin. An amber color will develop if nitrate is present.

8. Blank Preparation: When the timer expires, fill a round sample cell with 10 mL of sample.



9. Wipe the blank and insert it into the cell holder.

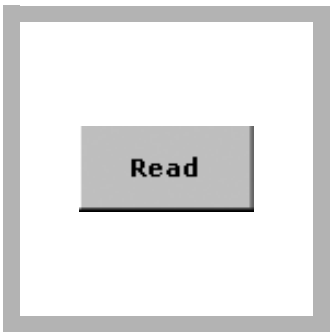


10. Press **ZERO**.

The display will show:

0.0 mg/L NO_3^-–N

11. Within two minutes after the timer expires, wipe the Ampul and insert it into the cell holder.



12. Press **READ**.

Results are in mg/L NO_3^-–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	Interferes at all levels
Nitrite	Interferes at all levels Compensate for nitrite interference as follows: **1.** Add 30-g/L Bromine Water[1] dropwise to the sample before NitraVer 5 Reagent addition until a yellow color remains. **2.** Add one drop of 30-g/L Phenol Solution[1] to destroy the color. **3.** Proceed with the test. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels

[1] See Optional Reagents and Apparatus on page 6.

Sample Collection, Storage, and Preservation

Most reliable results are obtained when samples are analyzed as soon as possible after collection. If prompt analysis is impossible, store samples in clean plastic or glass bottles for up to 24 hours at 4 °C. To preserve samples for longer periods, add 2 mL of Concentrated Sulfuric Acid (H_2SO_4)* per liter and store at 4 °C.

Before analysis, warm the sample to room temperature and adjust the pH to 7 with 5.0 N Sodium Hydroxide Standard Solution*. Do not use mercury compounds as preservatives. Correct the test result for volume additions by dividing the total volume (acid + base + sample) by the original sample volume and multiplying the test result by this factor.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a fresh bottle of Nitrate Nitrogen Standard,100-mg/L NO_3^-–N.

* See Optional Reagents and Apparatus on page 6.

5. Prepare three sample spikes. Fill three sample cells with 10 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

Note: *For AccuVac® Ampuls, fill three Mixing Cylinders with 50 mL of sample and spike with 0.1 mL, 0.2 mL, and 0.3 mL of 500 mg/L Nitrate Nitrogen Ampule Standard Solution. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers*. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. To test accuracy, use a 5.0-mg/L Nitrate Nitrogen Standard Solution instead of the sample and perform the procedure as described above.

2. Prepare a 5.0 mg/L Nitrate Nitrogen Standard by pipetting 5.0 mL of a 100 mg/L Nitrate Nitrogen Standard into a 100 mL volumetric flask. Dilute to volume with deionized water and mix well.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Cadmium metal reduces nitrates in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt. The salt couples with gentisic acid to form an amber colored solution. Test results are measured at 400 nm.

* See Optional Reagents and Apparatus on page 6.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
NitraVer® 5 Nitrate Reagent Powder Pillows (for 10 mL sample)	1	100/pkg	21061-69
OR			
NitraVer® 5 Nitrate Reagent AccuVac® Ampul	1	25/pkg	25110-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper, Neoprene, solid, size no. 2	2	12/pkg	14808-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Flask, volumetric, 100-mL	each	14574-42
Mixed Parameter Drinking Water Standard, for F, NO_3–N, PO_4, SO_4	500 mL	28330-49
Nitrate Nitrogen Standard Solution, 100-mg/L NO_3^-–N	500 mL	1947-49
Nitrate Nitrogen Standard Solution, 500 mg/L NO_3–N, 10-mL ampules	16/pkg	14260-10
Pipet, TenSette, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, 5.00 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Bromine Water, 30-mg/L	2211-20
Cylinder, mixing	20886-41
Phenol Solution, 30-g/L	2112-20
5.0 N Sodium Hydroxide Standard Solution	2450-53
Sulfuric Acid, concentrated	979-49

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Updated November 2005 Edition 1

Nitrite

Method 10019 **Diazotization Method**

Test 'N Tube™ Vials **LR (0.003 to 0.500 mg/L NO_2^-–N)**

Scope and Application: For water, wastewater, and seawater

Test Preparation

Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

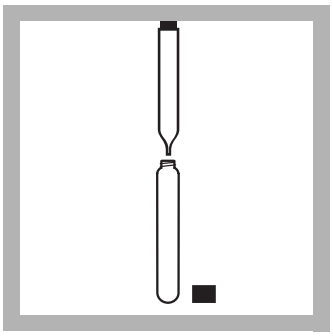
Collect the following items:	**Quantity**
Light Shield	1
Test 'N Tube™ NitriVer® 3 Nitrite Reagent Set	1
Pipet, TenSette®, 1.0 to 10.0 mL, plus tips	1

Note: *Reorder information for consumables and replacement items is on page 3.*

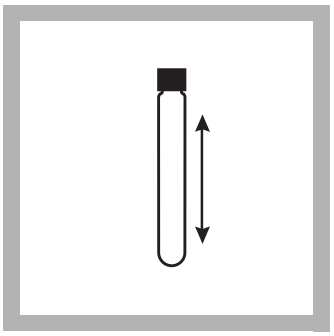
Test 'N Tube Method 10019

1. Select the test.

Install the Light Shield in Cell Compartment #2.



2. Fill a Test 'N Tube NitriVer® 3 Nitrite vial with 5 mL of sample.

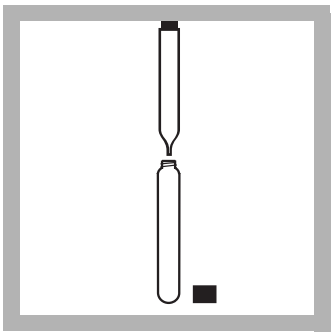


3. Prepared Sample: Cap and shake to dissolve the powder.

A pink color will develop if nitrite-nitrogen is present.

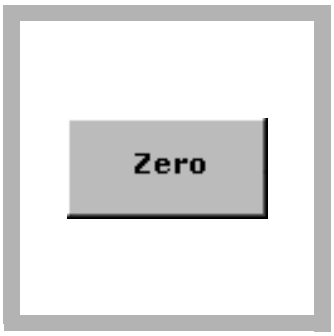
4. Press **TIMER>OK**.

A 20-minute reaction period will begin.



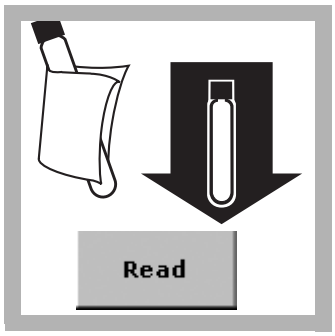
5. Blank Preparation: When the timer expires, fill an empty Test 'N Tube™ vial with 5 mL of sample.

6. Wipe the blank and insert it into the 16-mm round cell holder.



7. Press **ZERO**.

The display will show: 0.000 mg/L NO_2^-–N



8. Insert the prepared sample cell into the 16-mm round cell holder.

Press **READ**. Results are in mg/L NO_2^-–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Antiminous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interfere at all levels

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Store at 4 °C (30 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test.

Accuracy Check

Standard Solution Method
Preparing nitrite standards is difficult. Use the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater*, Method 4500-NO2 B. Prepare a 0.300-mg/L standard. Perform the nitrite test on the standard solution.

Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present. Test results are measured at 507 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Test ‘N Tube™ NitriVer® 3 Nitrite Reagent Set	1	50/pkg	26083-45

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	varies	50/pkg	21997-96

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Handbook, Standard Methods for the Examination of Water and Wastewater	each	22708-00
Pipet Tips, for TenSette Pipet 19700-10	250/pkg	21997-25
Sodium Nitrite, ACS	454 g	2452-01
Water, deionized	4 L	272-56

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Updated November 2005 Edition 1

Nitrite

★Method 8507 — Diazotization Method

Powder Pillows or AccuVac® Ampuls — LR (0.002 to 0.300 mg/L NO_2^-–N)

Scope and Application: For water, wastewater, and seawater; USEPA approved for wastewater analysis[1]

[1] Federal Register, 44(85), 25505 (May 1, 1979)

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample.

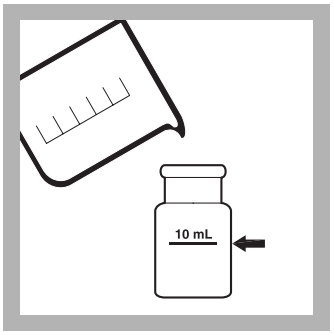
Collect the following items:	Quantity
Powder Pillow Test:	
NitriVer® 3 Nitrite Reagent Powder Pillows	1
Sample Cells, 1-inch square, 10-mL	2
AccuVac Test:	
NitriVer® 3 Nitrite Reagent AccuVac® Ampul.	1
Beaker, 50-mL	1
Sample Cell, 10-mL	1

Note: *Reorder information for consumables and replacement items is on page 4.*

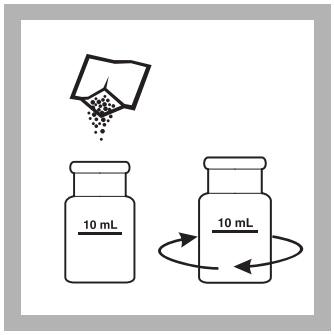
Powder Pillows — Method 8507

1. Press **STORED PROGRAMS**.

2. Select the test.



3. Fill a square sample cell with 10 mL of sample.

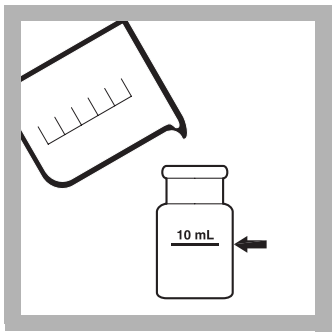


4. Prepared Sample: Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow. Swirl to dissolve.

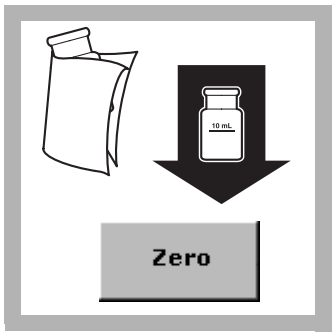
A pink color will develop if nitrite is present.

5. Press **TIMER>OK**.

A 20-minute reaction period will begin.



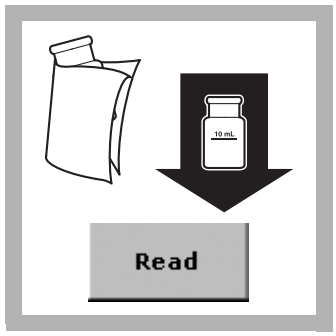
6. Blank Preparation: When the timer expires, fill a second square sample cell with 10 mL of sample.



7. Wipe the blank and insert it into the cell holder with the fill line facing right. Press **ZERO**.

The display will show:

0.000 mg/L NO_2^-–N

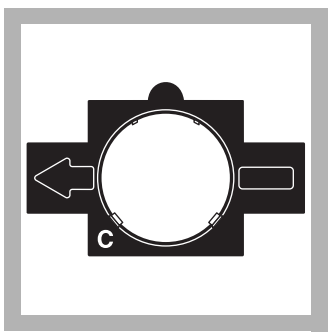


8. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

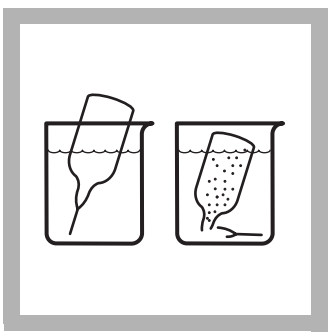
Press **READ**. Results are in mg/L NO_2^-–N.

AccuVac® Ampul — Method 8507

1. Select the test.

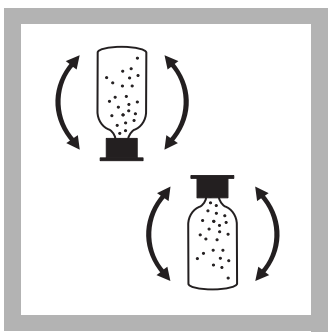


2. Insert Adapter C.



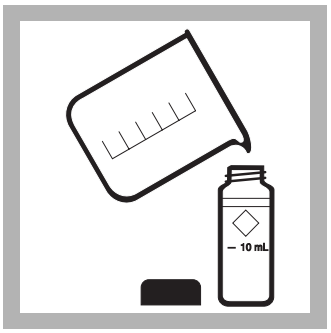
3. Prepared Sample: Collect at least 40 mL of sample into a 50-mL beaker.

Fill a NitriVer 3 Nitrite AccuVac® Ampul with sample. Keep the tip immersed while the Ampul fills.

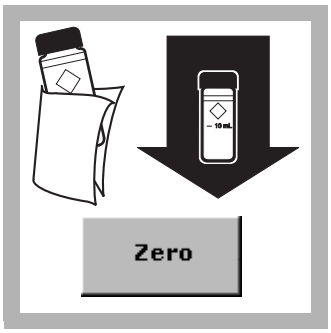


4. Invert the Ampul several times to mix. A pink color will develop if nitrite is present.

5. Press **TIMER**. Press **OK**. A 20-minute reaction period will begin.



6. Blank Preparation: When the timer expires, fill a sample cell with at least 10 mL of sample.



7. Wipe the blank and insert it into the cell holder.

Press **ZERO**.

The display will show: 0.000 mg/L NO_2^-–N

8. Wipe the AccuVac Ampul and insert it into the cell holder.

Press **READ**. Results are in mg/L NO_2^-–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Antimonous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interferes at all levels

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Store at 4 °C (30 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. Do not use acid preservatives.

Accuracy Check

Standard Solution Method

Preparing nitrite standards is difficult. Use the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater*, Method 4500—NO_2-B. Prepare a 0.150-mg/L standard.

Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present. Test results are measured at 507 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
NitriVer® 3 Nitrite Reagent Powder Pillows	1	100/pkg	21071-69
OR			
NitriVer® 3 Nitrite Reagent AccuVac® Ampul	1	25/pkg	25120-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Balance, analytical, SA80, 115 VAC	each	28014-01
Handbook, Standard Methods for the Examination of Water and Wastewater	—	—
Sodium Nitrite, ACS	454 g	2452-01
Water, deionized	4 L	242-56

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Updated November 2005 Edition 1

Nitrite

Method 8153
Powder Pillows

Ferrous Sulfate Method[1]
HR (2 to 250 mg/L NO_2^-)

Scope and Application: For cooling tower waters

[1] Adapted from McAlpine, R. and Soule, B., *Qualitative Chemical Analysis*, New York, 476, 575 (1933)

Test Preparation

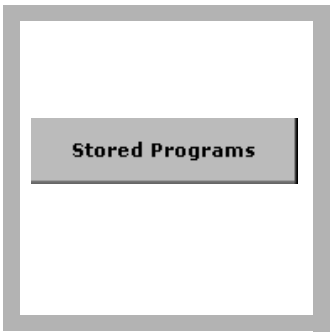
Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
After adding the reagent, a greenish-brown color will develop if nitrite is present.

Collect the following items:	**Quantity**
NitriVer® 2 Nitrite Reagent Powder Pillows	1
Deionized water	varies
Stopper, Neoprene, solid # 1	2
Sample Cells, 1-inch square, 10-mL	2

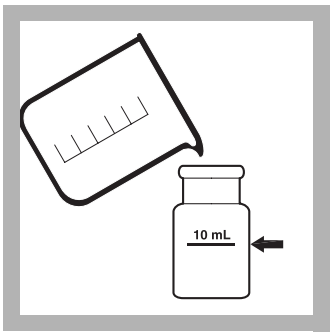
Note: *Reorder information for consumables and replacement items is on page 3.*

Powder Pillows Method 8153

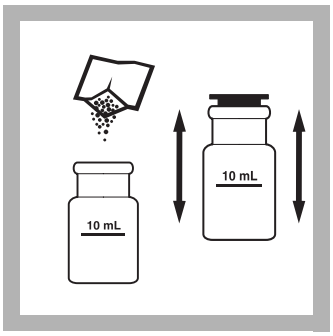


1. Press **STORED PROGRAMS.**

2. Select the test.



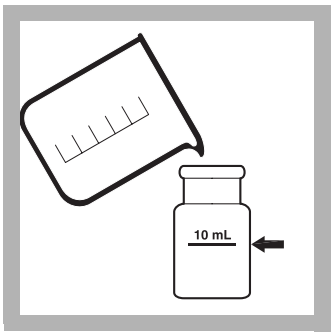
3. Fill a square sample cell with 10 mL of sample.



4. Prepared Sample: Add the contents of one NitriVer® 2 Nitrite Reagent Powder Pillow. Stopper and shake to dissolve.

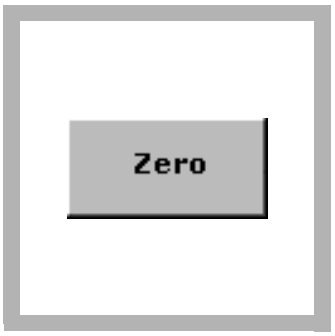
5. Press **TIMER>OK**.

A ten-minute reaction period will begin. To prevent low results, leave the sample on a flat surface and **do not disturb it during the reaction period.**



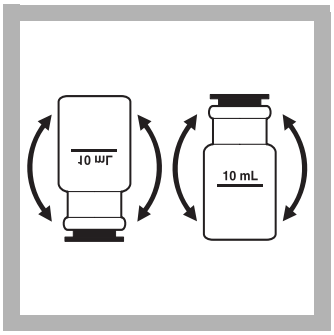
6. Blank Preparation: Fill a second square sample cell with 10 mL of sample.

7. Wipe the blank and insert it into the cell holder will the fill line facing right.



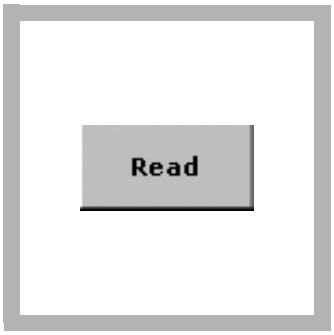
8. Press **ZERO**.

The display will show: 0 mg/L NO_2^-



9. After the timer expires, cap and **gently** invert the prepared sample twice. Avoid excessive mixing, or low results may occur.

10. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.



11. Press **READ**.

Results are in mg/L NO_2^-.

Interferences

This test does not measure nitrates nor is it applicable to glycol-based samples. Dilute glycol-based samples and follow the Low Range Nitrite procedure, Method 8507.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles.

The following storage instructions are necessary only when prompt analysis is impossible. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. Do not use acid preservatives.

Accuracy Check

Standard Solution Method
Preparing nitrite standards is difficult. Use the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater*. Prepare a 200-mg/L standard using Sodium Nitrite, ACS*, reagent grade.

Summary of Method

The method uses ferrous sulfate in an acidic medium to reduce nitrite to nitrous oxide. Ferrous ions combine with the nitrous oxide to form a greenish-brown complex in direct proportion to the nitrite present. Test results are measured at 585 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
NitriVer® 2 Nitrite Reagent Powder Pillows	1	100/ pkg	21075-69
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square. 10-mL matched pair	2	2/pkg	24954-02
Stopper, Neoprene, solid #1	2	12/pkg	14808-01

Optional Reagents and Apparatus

Description	Cat. No.
Balance, Analytical SA80 115 VAC	28014-01
Sodium Nitrite, ACS	2452-01

* See Optional Reagents and Apparatus on page 3.

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Updated November 2005 Edition 1

Nitrite

★Method 10207 — Diazotization Method

TNTplus™ 839

LR (0.015 to 0.600 mg/L NO_2^-–N or 0.05 to 2.00 mg/L NO_2)

Scope and Application: For wastewater, drinking water, surface water and mineral water

Test Preparation

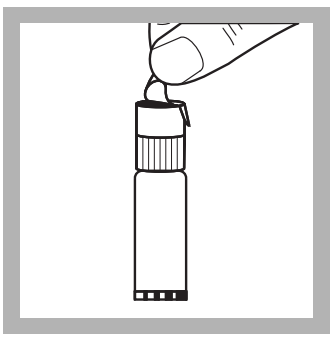
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on the reagent package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 3–10.
Recommended reagent storage temperature is 2–8 °C (35.6–46.4 °F).
TNTplus methods are activated directly from the Main Menu when the sample vial is inserted into the sample cell holder.

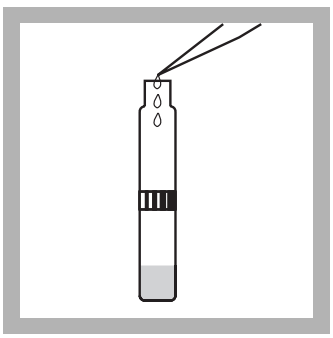
Collect the following items:	**Quantity**
TNT 839 Reagent Set	1
Light Shield	1
Pipettor for 2.0 mL Sample	1
Pipettor Tip	1

Note: *Reorder information for consumables and replacement items is on page 4.*

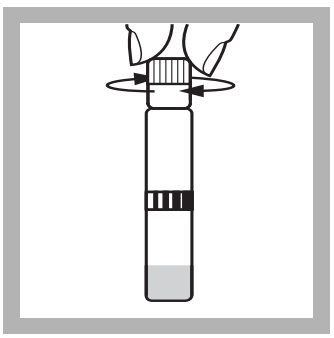
TNTplus — Method 10207



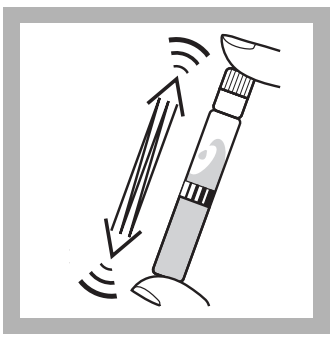
1. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



2. Carefully pipet 2.0 mL of sample into the vial. Immediately proceed to step 3.



3. Flip the DosiCap ***Zip*** over so that the reagent side faces the vial. Screw the cap tightly onto the vial.

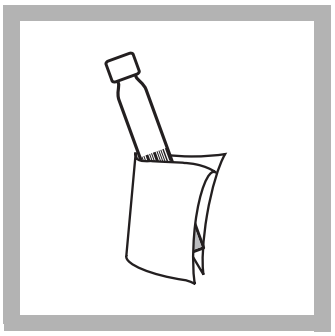


4. Shake the capped vial 2–3 times to dissolve the reagent in the cap.

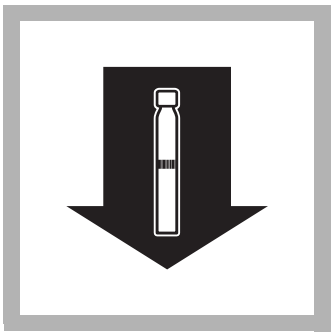
Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

5. Wait 10 minutes.

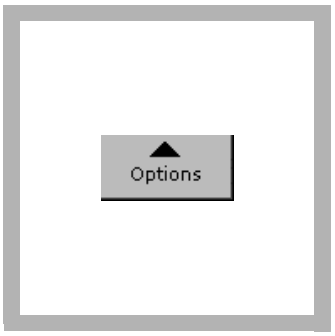
Install the Light Shield in Cell Compartment #2.



6. After 10 minutes, thoroughly clean the outside of the vial.



7. Insert the prepared vial into the cell holder. The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L NO_2^-–N



Note: *Press* ***OPTIONS>MORE> CHEMICAL FORMS*** *to obtain results in other chemical forms.*

Reagent Blanks

A reagent blank can be measured and the value subtracted from the results of each test performed in same reagent lot. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 7. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity the procedure is repeated and the color forming reagent that is present in the DosiCap ***Zip*** is not added.

To determine the sample blank run the procedure as given, but do not remove the foil from the DosiCap ***Zip*** in step 1 and replace the cap in its original position in step 3. The value obtained in step 7 is then subtracted from the value obtained on the original sample to give the corrected sample concentration.

Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually tested up to the given concentrations and do not cause interference. The cumulative effects of these ions or the influence of other ions have not been determined.

Table 1 Interfering Substances and Levels

Interfering Substances	Interference Levels
Cl^-, SO_4^{2-}	2000 mg/L
K^+, NO_3^-	1000 mg/L
NH_4^+, PO_4^{3-}, Ca^{2+}	500 mg/L
Mg^{2+}	100 mg/L
Cr^{3+}	50 mg/L
Co^{2+}, Zn^{2+}, Cd^{2+}, Mn^{2+}, Hg^{2+}	25 mg/L
Ni^{2+}	12 mg/L
Ag^+, Fe^{2+}	10 mg/L
Sn^{4+}, Fe^{3+}	5 mg/L
Cu^{2+}	< 1 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Store at 4 °C (30 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to 15–25 °C (59–77 °F) before running the test. Do not use acid preservatives.

Accuracy Check

Standard Solution Method

Preparing nitrite standards is difficult. Use the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater*, Method 4500—NO_2-B. Prepare a 0.30-mg/L NO_2–N standard.

Summary of Method

Nitrite in the sample reacts with a primary aromatic amine in acidic solution to form a diazonium salt. This couples with an aromatic compound to form a colored complex that is directly proportional to the amount of nitrite present. Test results are measured at 515 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Nitrite, TNT 839 TNTplus™ Reagent Set	1	25/pkg	TNT839

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipet, variable volume, 1–5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	1	100/pkg	27952-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Balance, analytical, SA80, 115 VAC	each	28014-01
Handbook, Standard Methods for the Examination of Water and Wastewater	each	22708-00
Sodium Nitrite, ACS	454 g	2452-01
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
Test Tube Rack for 13-mm vials		

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Updated November 2005 Edition 1

Nitrogen, Ammonia

Method 8155
Powder Pillows

Salicylate Method[1]
(0.01 to 0.50 mg/L NH_3–N)

Scope and Application: For water, wastewater, and seawater

[1] Adapted from *Clin. Chim. Acta., 14*, 403 (1966)

Test Preparation

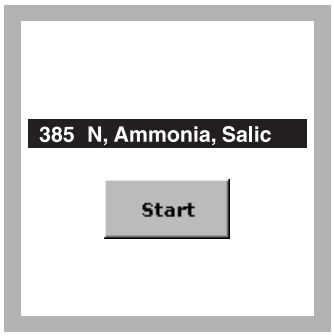
Collect the following items:	Quantity
Ammonia Cyanurate Reagent pillows	2
Ammonia Salicylate Reagent pillows	2
Sample Cells, 1-inch square, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 5.*

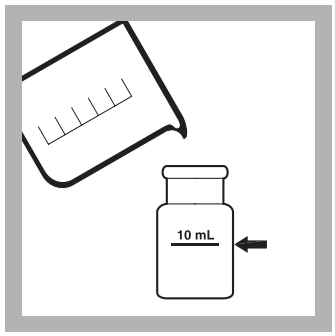
Note: *A green color will develop if ammonia nitrogen is present.*

Powder Pillows — Method 8155

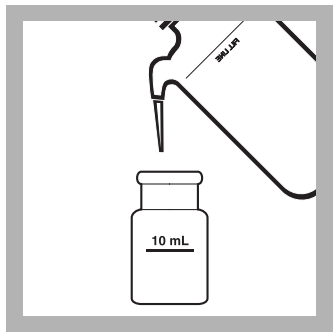
1. Press **STORED PROGRAMS**.



1. Select the test.



2. Prepared Sample: Fill a square sample cell to the 10-mL mark with sample.

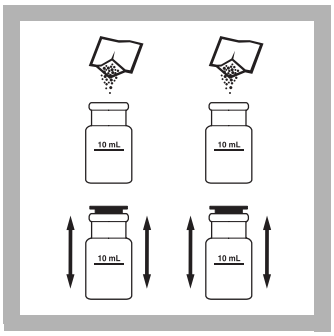


3. Blank Preparation: Fill a second square sample cell to the 10-mL mark with deionized water.

4. Add the contents of one Ammonia Salicylate Powder Pillow to each cell.

Stopper and shake to dissolve.

5. Press **TIMER>OK**. A three-minute reaction period will begin.



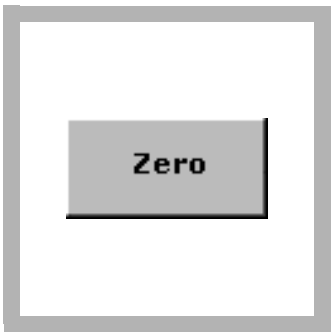
6. When the timer expires, add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each cell.

Stopper and shake to dissolve.

7. Press **TIMER>OK**. A 15-minute reaction period will begin.

A green color will develop if ammonia-nitrogen is present.

8. When the timer expires, insert the blank into the cell holder with the fill line facing right.



9. Press **ZERO**. The display will show: 0.00 mg/L NH_3–N

10. Wipe the sample and insert it into the cell holder with the fill line facing right.

11. Press **READ**.

Results are in mg/L NH_3–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium	Greater than 1000 mg/L as $CaCO_3$
Iron	All levels. Correct for iron interference as follows: **1.** Determine the amount of iron present in the sample by following one of the Iron, Total, procedures. **2.** Add the same iron concentration to the ammonia-free water in step 3. The interference will be successfully blanked out.
Magnesium	Greater than 6000 mg/L as $CaCO_3$
Monochloramine	Monochloramine present in chloraminated drinking water interferes directly at all levels, giving high results. Use Method 10200, Free Ammonia and Monochloramine, to determine free ammonia in these sample matrices.
Nitrate	Greater than 100 mg/L as NO_3^-–N
Nitrite	Greater than 12 mg/L as NO_2^-–N
Phosphate	Greater than 100 mg/L as PO_4^{3-}–P
Sulfate	Greater than 300 mg/L as SO_4^{2-}
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: **1.** Measure about 350 mL of sample in a 500-mL Erlenmeyer flask[1]. **2.** Add the contents of one Sulfide Inhibitor Reagent[1] Powder Pillow. Swirl to mix. **3.** Filter the sample through a Folded Filter Paper[1] and Filter Funnel[1]. **4.** Use the filtered solution in step 3.
Other Substances	Less common interferences such as **hydrazine** and **glycine** will cause intensified colors in the prepared sample. **Turbidity** and **color** will give erroneous high values. Samples with severe interferences require distillation. Use the distillation procedure with the General Purpose Distillation Set.

[1] See Optional Reagents and Apparatus on page 5.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

Adjust the pH to 2 or less with concentrated (about 2 mL per liter) Sulfuric Acid. Store samples at 4 °C or less. Samples preserved in this manner can be stored up to 28 days. Just before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions.

Accuracy Check

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open an Ammonia Nitrogen Standard Solution, 10-mg/L as NH_3–N.

5. Prepare three sample spikes. Fill three mixing cylinders with 25 mL of sample.Use the TenSette® Pipet to add 0.2 mL, 0.4 mL, and 0.6 mL of standard, respectively to the cylinders and mix each thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.2 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method
Prepare a 0.40 mg/L ammonia nitrogen standard as follows:

1. Diluting 4.00 mL of the Ammonia Nitrogen Standard Solution, 10-mg/L, to 100 mL with deionized water. Or, use the TenSette® Pipet to prepare a 0.40 mg/L ammonia nitrogen standard by diluting 0.8 mL of an Ammonia Nitrogen Voluette® Standard Solution, 50-mg/L as NH_3–N, to 100 mL with deionized water.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Ammonia Nitrogen Reagent Set for 10-mL samples (100 tests), includes:	—	—	26680-00
Includes:			
(2) Ammonia Cyanurate Reagent Powder Pillows	2	100/pkg	26531-99
(2) Ammonia Salicylate Reagent Powder Pillows	2	100/pkg	26532-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10-mL, matched pair	2	2/pkg	24954-02

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrogen, Ammonia Standard Solution, 10-mg/L NH_3–N	500 mL	153-49
Nitrogen, Ammonia Standard Solution, 2-mL PourRite® Ampule, 50-mg/L NH_3–N	20/pkg	14791-20
Wastewater, Effluent Inorganics, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49
Pipet, TenSette® 0.1 - 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, volumetric, Class A, 4.00 mL	each	14515-04
Pipet Filler, safety bulb	each	14651-00
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing	20886-40
Distillation Set	22653-00
Erlenmeyer Flask	505-49
Filter Funnel	1083-67
Filter Paper	1894-57
Sodium Hydroxide Standard Solution, 5.0 N	2450-26
Sulfide Inhibitor Reagent Powder Pillow	2418-99
Sulfuric Acid	979-49

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Updated November 2005 Edition 1

Nitrogen, Ammonia

★Method 8038

Nessler Method[1]

(0.02 to 2.50 mg/L NH_3–N)

Scope and Application: For water, wastewater, and seawater; distillation is required for wastewater and seawater; USEPA accepted for wastewater analysis (distillation required); see Distillation on page 4 of this procedure.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater 4500-NH_3 B & C*.

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the user manual for more information.
Nessler Reagent contains mercuric iodide. Both the sample and the blank will contain mercury (D009) at a concentration regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. Refer to a current MSDS for safe disposal and handling instructions.

Collect the following items:	**Quantity**
Ammonia Nitrogen Reagent set	1
Deionized Water	25 mL
Graduated Mixing Cylinders	2
Sample Cells, 1-inch square, 10-mL	2
Serological Pipet, 1-mL	2

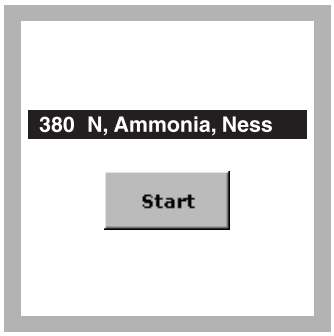
Note: *Reorder information for consumables and replacement items is on page 5.*

Note: *Nessler Reagent is toxic and corrosive. Pipet carefully, using a pipet filler. When dispensing reagent from a dropper bottle, hold the bottle vertically. Do not hold the bottle at an angle.*

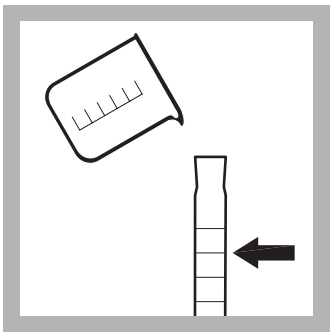
Note: *A yellow color will develop if ammonia is present. (The reagent will cause a faint yellow color in the blank.)*

Nessler Method 8038

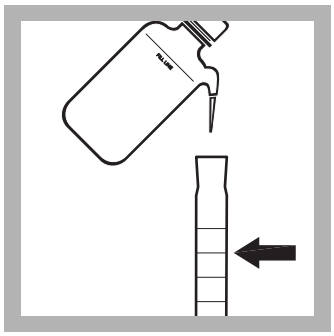
1. Press **STORED PROGRAMS**.



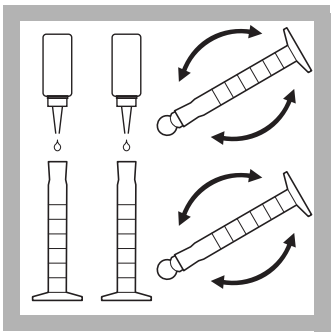
2. Select the test.



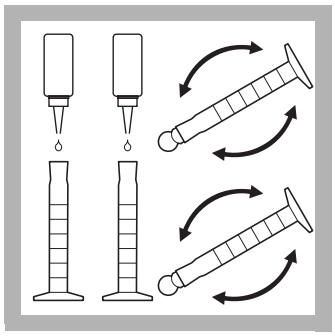
3. Prepared Sample: Fill a 25-mL mixing graduated cylinder to the 25-mL mark with sample.



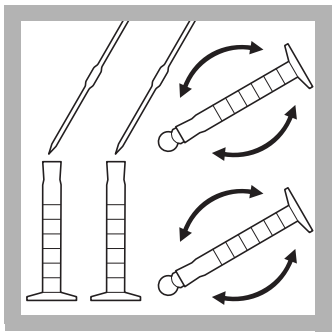
4. Blank Preparation: Fill a 25-mL mixing graduated cylinder to the 25-mL mark with deionized water.



5. Add three drops of Mineral Stabilizer to each cylinder. Stopper and invert several times to mix.



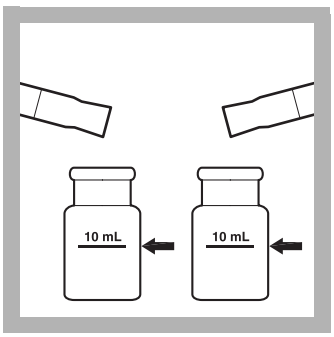
6. Add three drops of Polyvinyl Alcohol Dispersing Agent to each cylinder. Stopper and invert several times to mix.



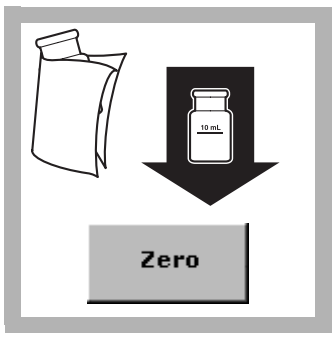
7. Pipet 1.0 mL of Nessler Reagent into each cylinder. Stopper and invert several times to mix.

8. Press **TIMER>OK**.

A one-minute reaction period will begin.



9. Pour 10 mL of each solution into a square sample cell.



10. When the timer expires, insert the blank into the cell holder with the fill line facing right. Press **ZERO**. The display will show:
0.00 mg/L NH_3 –N

11. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

12. Press **READ**.

Results are in mg/L NH_3–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chlorine	Remove residual chlorine by adding 2 drops of sodium arsenite for each mg/L chlorine (Cl_2) from a 250 mL sample. Sodium thiosulfate can be used instead of sodium arsenite. See Sample Collection, Storage, and Preservation.
Hardness	A solution containing a mixture of 500 mg/L $CaCO_3$ and 500 mg/L Mg as $CaCO_3$ does not interfere. If the hardness concentration exceeds these concentrations, add extra Mineral Stabilizer.
Iron	Interferes at all levels by causing turbidity with Nessler Reagent.
Seawater	May be analyzed by adding of 1.0 mL (27 drops) of Mineral Stabilizer to the sample before analysis. This complexes the high magnesium concentrations found in sea water, but the sensitivity of the test is reduced by 30 percent due to the high chloride concentration. For best results, perform a calibration, using standards spiked to the equivalent chloride concentration, or distill the sample as described below.
Sulfide	Interferes at all levels by causing turbidity with Nessler Reagent.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Glycine, various aliphatic and aromatic amines, organic chloramines, acetone, aldehydes and alcohols	May cause greenish or other off colors or turbidity. Distill the sample if these compounds are present.

Sample Collection, Storage, and Preservation

Collect samples in clean glass or plastic bottles. If chlorine is present, add one drop of 0.1 N Sodium Thiosulfate* for each 0.3 mg/L Cl_2 in a 1-liter sample. Preserve the sample by reducing the pH to 2 or less with sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide* before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off a Nitrogen Ammonia Voluette® Ampule Standard, 50-mg/L NH_3–N.
5. Prepare three sample spikes. Fill three mixing cylinders with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of the 50 mg/L standard, respectively, to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Standard Solutions Method

1. To check accuracy, use a 1.0-mg/L Nitrogen Ammonia Standard Solution. Or, prepare a 1.0-mg/L ammonia nitrogen standard solution by pipetting 1.00 mL of Nitrogen Ammonia Voluette® Ampule Standard, 50-mg/L, into a 50-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the Nessler procedure as described above.

* See Optional Reagents and Apparatus on page 5.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Distillation

1. Measure 250 mL of sample into a 250-mL graduated cylinder and pour into a 400-mL beaker. Destroy chlorine, if necessary, by adding 2 drops of Sodium Arsenite Solution per mg/L Cl_2.

2. Add 25 mL of Borate Buffer Solution and mix. Adjust the pH to about 9.5 with 1 N sodium hydroxide solution. Use a pH meter.

3. Set up the General Purpose Distillation Apparatus as shown in the *Distillation Apparatus Manual*. Pour the solution into the distillation flask. Add a stir bar.

4. Use a graduated cylinder to measure 25 mL of deionized water into a 250-mL Erlenmeyer flask. Add the contents of one Boric Acid Powder Pillow. Mix thoroughly. Set the flask under the still drip tube. Elevate so the end of the tube is immersed in the solution.

5. Turn on the heater power switch. Set the stir control to 5 and the heat control to 10. Turn on the water and adjust to maintain a constant flow through the condenser.

6. Turn off the heater after collecting 150 mL of distillate. Immediately remove the collection flask to avoid sucking solution into the still. Measure the distillate to ensure 150 mL was collected (total volume = 175 mL).

7. Adjust the pH of the distillate to about 7 with 1 N sodium hydroxide. Use a pH meter.

8. Pour the distillate into a 250-mL volumetric flask; rinse the Erlenmeyer with deionized water. Add the rinsings to the volumetric flask. Dilute to the mark. Stopper. Mix thoroughly. Analyze as described above.

Summary of Method

The Mineral Stabilizer complexes hardness in the sample. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color is formed proportional to the ammonia concentration. Test results are measured at 425 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Ammonia Nitrogen Reagent Set, includes:	—	—	24582-00
Nessler Reagent	2 mL	500 mL	21194-49
Mineral Stabilizer	6 drops	50 mL SCDB	23766-26
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	23765-26
Water, deionized	25 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, mixing, 25-mL	2	each	20886-40
Pipet, serological, 1-mL	2	each	9190-02
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10-mL, matched pair	2	2/pkg	24954-02

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Flask, volumetric, Class A, 50 mL	each	14574-41
Nitrogen, Ammonia Standard Solution, 1-mg/L NH_3–N	500 mL	1891-49
Nitrogen, Ammonia Standard Solution, 10-mL Voluette® Ampule, 50-mg/L NH_3–N	16/pkg	14791-10
Pipet, TenSette® 0.1 - 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Wastewater, Effluent Inorganics, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49

Optional Reagents and Apparatus

Description	Cat. No.
Distillation Apparatus, General	22653-00
Heater and Support Apparatus, 115 VAC, 60 Hz	22744-00
Heater and Support Apparatus, 230 VAC, 50 Hz	22744-02
Mixing Cylinders	20886-40
Pour-Thru Cell Kit	59404-00
Sodium Thiosulfate, 0.1 N	323-32
Sodium Hydroxide, 5 N	2450-32

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Updated November 2005 Edition 1

Nitrogen, Ammonia

Method 10031 **Salicylate Method**

Test ‘N Tube™ Vials **HR (0.4 to 50.0 mg/L NH_3–N)**

Scope and Application: For water, wastewater, and seawater

Test Preparation

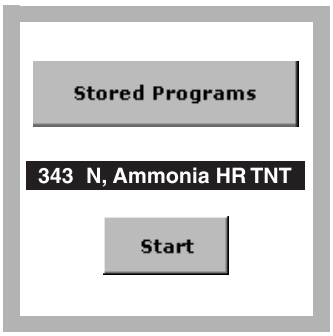
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Small sample sizes (such as 0.1 mL) may not be representative of the entire sample. Mix the sample well before testing or repeat the test, sampling from different portions of the sample.
Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

Collect the following items:	**Quantity**
High Range Test ‘N Tube AmVer™ Nitrogen Ammonia Reagent	2
Light Shield	1
Funnel, micro (for adding reagent)	1
Pipet, TenSette®, 0.1–1.0 mL	1
Pipet Tips, for TenSette Pipet	varies

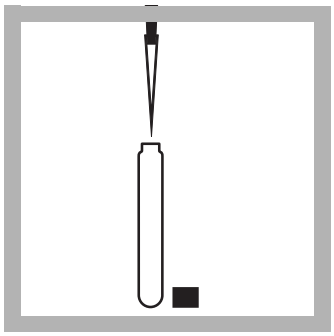
Note: *Reorder information for consumables and replacement items is on page 4.*

Test ‘N Tube Method 10031

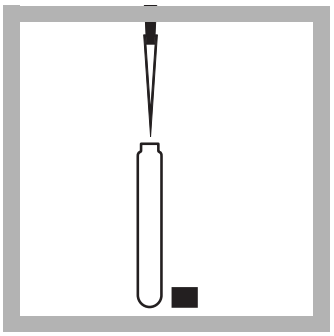


1. Select the test.

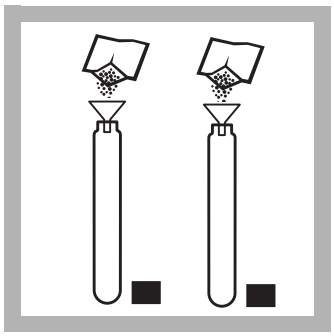
Install the Light Shield in Cell Compartment #2.



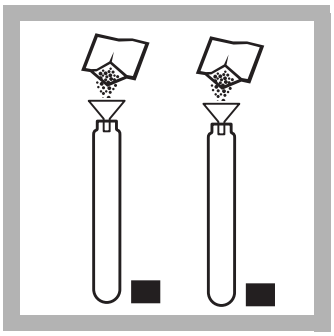
2. Prepared Sample: Add 0.1 mL of sample to one AmVer™ Diluent Reagent Test ‘N Tube for High Range Ammonia Nitrogen.



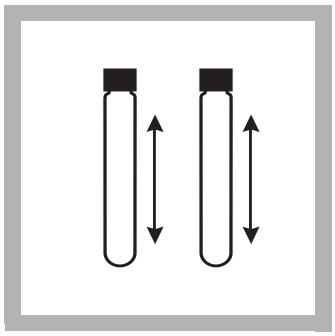
3. Blank Preparation: Add 0.1 mL of ammonia-free water to one AmVer™ Diluent Reagent Test ‘N Tube for High Range Ammonia Nitrogen.



4. Add the contents of one Ammonia Salicylate Reagent Powder Pillow for 5 mL sample to each vial.

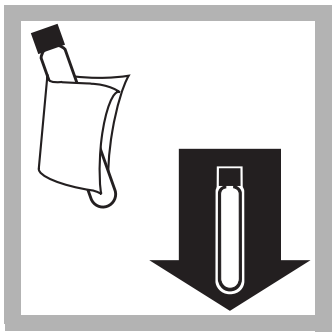


5. Add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each vial.

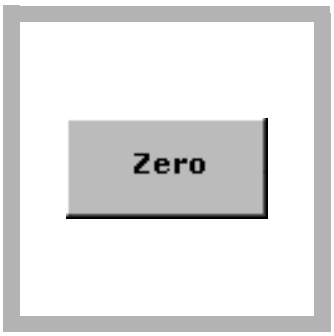


6. Cap the vials tightly and shake thoroughly to dissolve the powder.

7. Press **TIMER>OK**. A 20-minute reaction period will begin.

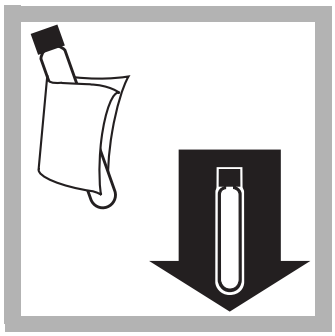


8. After the waiting period, wipe the blank and insert it into the 16-mm round cell holder.



9. Press **ZERO**.

The display will show: 0.0 mg/L NH_3–N



10. Wipe the sample vial and insert it into the 16-mm round cell holder.

11. Press **READ**.

Results are in mg/L NH_3–N.

Interferences

In some lab environments, airborne cross contamination of the blank is possible. Complete preparation of the blank before opening or handling any samples or standards to avoid transfer of ammonia. If sample or standard containers have already been opened, move to a separate area of the lab to prepare the blank.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Acidic or Basic Samples	Adjust to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution[1] for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Calcium	50,000 mg/L as $CaCO_3$
Glycine, hydrazine	Will cause intensified colors in the prepared sample.
Magnesium	300,000 mg/L as $CaCO_3$
Monochloramine	Monochloramine present in chloraminated drinking water interferes directly at all levels giving high results. Use Method 10200, Free Ammonia and Monochloramine, to determine free ammonia in these sample matrices.
Iron	Eliminate iron interference as follows: **1.** Determine the amount of iron present in the sample using one of the total iron procedures. **2.** Add the same iron concentration to the deionized water in step 3. The interference will then be successfully blanked out.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Nitrite	600 mg/L as NO_2^-–N
Nitrate	5000 mg/L as NO_3^-–N
Orthophosphate	5000 mg/L as PO_4^{3-}–P
Sulfate	6000 mg/L as SO_4^{2-}
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: **1.** Measure about 350 mL of sample in a 500-mL Erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow[1]. Swirl to mix. **3.** Filter the sample through folded filter paper[1]. Use the solution in step 2.
Turbidity and color	Give erroneous high values. Samples with severe interferences require distillation. The manufacturer recommends the distillation procedure using the General Purpose Distillation Set.

[1] See Optional Reagents and Apparatus on page 4.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. Preserve samples by reducing the pH to 2 or less with at least 2 mL of Hydrochloric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature. Neutralize to a pH of 7.0 with 5.0 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off a Nitrogen, Ammonia PourRite® Ampule Standard, 150-mg/L NH_3–N.
5. Prepare three sample spikes. Fill three mixing cylinders with 25 mL of sample. Use the TenSette® Pipet to add 0.2, 0.4 mL, and 0.6 mL of standard, respectively, to each sample and mix each thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.2 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. To check accuracy, prepare a 40.0-mg/L ammonia nitrogen standard solution by pipetting 20.00 mL of 100-mg/L Ammonia Nitrogen standard into a 50-mL, Class A volumetric flask. Dilute to the mark with deionized water.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue colored compound. The blue color is masked by the yellow color from the excess reagent present to give a green-colored solution. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Reagent Set, High Range Test 'N Tube™ AmVer™ Nitrogen Ammonia	50 tests	26069-45

Required Apparatus

Description		Unit	Cat. No.
Funnel, micro (for adding reagent)		each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for TenSette® Pipet 19700-01		50/pkg	21856-96

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrogen Ammonia Standard Solution, 10-mg/L NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Solution, 100-mg/L NH_3–N	500 mL	24065-49
Nitrogen Ammonia Standard Solution, 150-mg/L NH_3–N, 10-mL PourRite® Ampules	16/pkg	21284-10
Nitrogen Ammonia Standard Solution, 50-mg/L NH_3–N, 10-mL Voluette® Ampules	16/pkg	14791-10
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Wastewater, Effluent Inorganics Standard, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49
Water, deionized	4L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinders, mixing	20886-40
Distillation Set, general purpose	22653-00
Filter Paper	692-57
Hydrochloric Acid Standard Solution, 1 N	134-49
Sodium Hydroxide Standard Solution, 1 N	1045-32
Sulfide Inhibitor Reagent Powder Pillows	2418-99

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Updated November 2005 Edition 1

Nitrogen, Ammonia

Method 10023 **Salicylate Method[1]**

Test ‘N Tube™ Vials **LR (0.02 to 2.50 mg/L NH_3–N)**

Scope and Application: For water, wastewater, and seawater

[1] Adapted from *Clin. Chim. Acta*, 14, 403 (1966)

Test Preparation

Before starting the test:

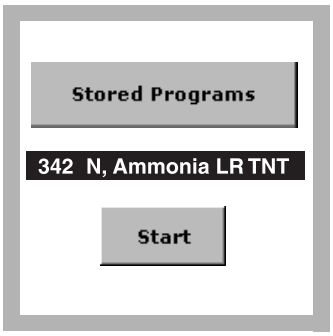
Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data sheet (MSDS) for information specific to the reagent used.

Collect the following items:	**Quantity**
Light Shield	1
Low Range Test ‘N Tube AmVer™ Nitrogen Ammonia Reagent	2
Funnel, micro (for adding reagent)	1
TenSette® Pipet, 1.0–10.0 mL	1
Pipet Tips for TenSette Pipet	varies

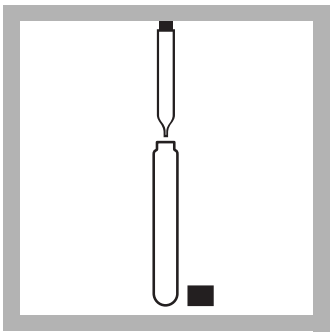
Note: *Reorder information for consumables and replacement items is on page 4.*

Test ‘N Tube Method 10023

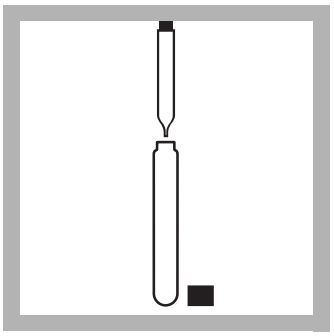


1. Select the test.

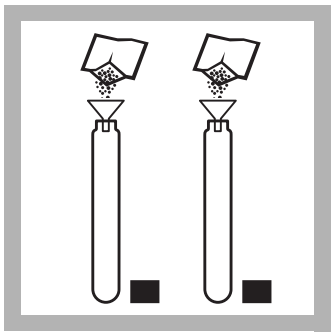
Install the Light Shield in Cell Compartment #2.



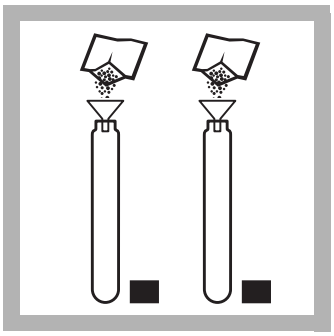
2. Prepared Sample: Add 2.0 mL of sample to one AmVer™ Diluent Reagent Test ‘N Tube™ for Low Range Ammonia Nitrogen.



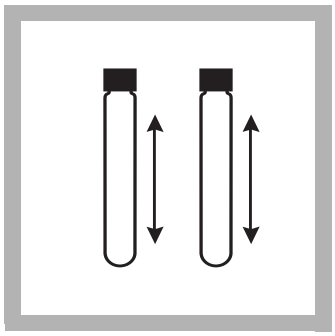
3. Blank Preparation: Add 2.0 mL of ammonia-free water to another AmVer™ Diluent Reagent Test ‘N Tube for Low Range Ammonia Nitrogen.



4. Add the contents of one Ammonia Salicylate Reagent Powder Pillow to each vial.

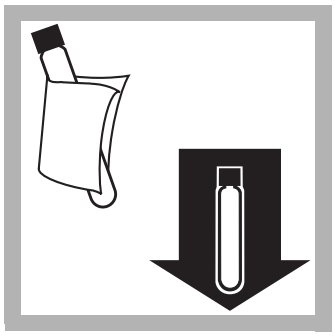


5. Add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each vial.

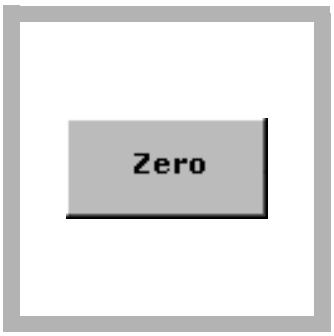


6. Cap the vials tightly and shake thoroughly to dissolve the powder.

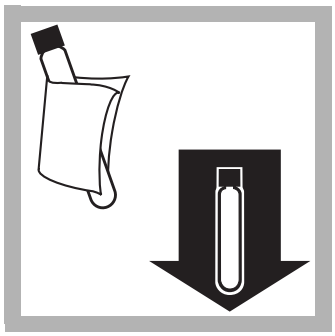
7. Press **TIMER>OK**. A 20-minute reaction period will begin.



8. After the waiting period, wipe the blank and insert it into the 16-mm round cell holder.



9. Press **ZERO**. The display will show: 0.00 mg/L NH_3–N



10. Wipe the sample vial and insert it into the 16-mm round cell holder.

11. Press **READ**.

Results are in mg/L NH_3–N.

Interference

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium	2500 mg/L as $CaCO_3$
Iron	Determine the amount of iron present in the sample by using one of the Iron, Total, procedures. Add the same iron concentration to the ammonia-free water in step 3. The interference will then be successfully blanked out.
Magnesium	15,000 mg/L as $CaCO_3$
Monochloramine	Monochloramine present in chloraminated drinking water Interferes directly at all levels giving high results. Use Method 10200, Free Ammonia and Monochloramine, to determine free ammonia in these sample matrices.
Nitrite	30 mg/L as NO^2_–N
Nitrate	250 mg/L as NO^3_–N
Orthophosphate	250 mg/L as PO_4^{3-}–P
pH	Acidic or basic samples should be adjusted to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution[1] for acidic samples and 1 N Hydrochloric Acid Standard Solution[1] for basic samples.
Sulfate	300 mg/L as SO_4^{2-}

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Sulfide	**1.** Measure about 350 mL of sample in a 500-mL Erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow[1]. Swirl to mix. **3.** Filter the sample through a folded filter paper[1]. **4.** Use the filtered solution in step 3.
Other	Less common interferences such as **hydrazine** and **glycine** will cause intensified colors in the prepared sample. **Turbidity** and **color** will give erroneous high values. Samples with severe interferences require distillation. Use the distillation procedure with the General Purpose Distillation Set.

[1] See Optional Reagents and Apparatus on page 4.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. Preserve the samples by reducing the pH to 2 or less with at least 2 mL of Hydrochloric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize to pH 7.0 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off an Ammonia Nitrogen Ampule Standard*, 50-mg/L as NH_3–N.
5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. To check accuracy, use the Nitrogen Ammonia Standard Solution, 1.0 mg/L. Or, dilute 1 mL of 50-mg/L Nitrogen Ammonia Standard Solution to 50 mL with deionized water in a 50-mL volumetric flask to prepare a 1.0 mg/L solution.
2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

* See Optional Reagents and Apparatus on page 4.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green solution. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Reagent Set, Low Range Test 'N Tube™ AmVer™ Nitrogen Ammonia	25 tests	26045-45

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Funnel, micro (for adding reagent)	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	varies	50/pkg	21997-96
Tube Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrogen Ammonia Standard Solution, 1.0-mg/L NH_3–N	500 mL	1891-49
Nitrogen Ammonia Standard Solution, 50-mg/L NH_3–N, 10-mL Voluette® Ampules	16/pkg	14791-10
Pipet Tips, for TenSette Pipet 19700-10	250/pkg	21856-25
Wastewater, Effluent Inorganics Standard, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49
Water, deionized	4L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinders, mixing	20886-40
Distillation Set, general purpose	22653-00
Filter Funnel	1083-67
Filter Paper	1894-57
Hydrochloric Acid Standard Solution, 1 N	23213-53
Hydrochloric Acid, concentrated ACS	134-49
Sodium Hydroxide Standard Solution, 1 N	1045-32
Sulfide Inhibitor Reagent Powder Pillows	2418-99

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Updated November 2005 Edition 1

Nitrogen, Ammonia

Method 10205 **Salicylate Method**

TNTplus™ 830 **ULR (0.015 to 2.000 mg/L NH_3–N)**

Scope and Application: For municipal and industrial wastewaters, environmental waters, and watershed protection monitoring.

Test Preparation

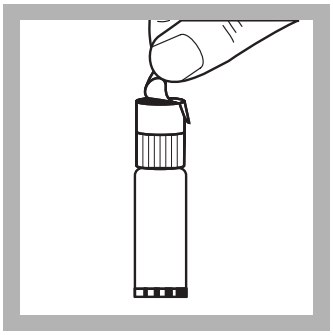
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample pH is 4–8.
Recommended sample and reagent temperature is 20–23 °C (68–73.4 °F). Incorrect results may be obtained if test is not performed at the recommended temperature.
Recommended reagent storage is 2–8 °C (35.6–46.4 °F).
Analyze samples as soon as possible for best results.
TNTplus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

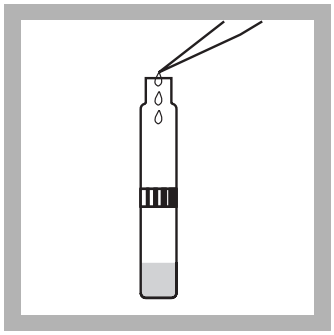
Collect the following items:	**Quantity**
Nitrogen, Ammonia, TNT 830 Reagent Set	1 vial
Light Shield	1
Pipettor for 5.0 mL Sample	1
Pipettor Tip	varies

Note: *Reorder information for consumables and replacement items is on page 4.*

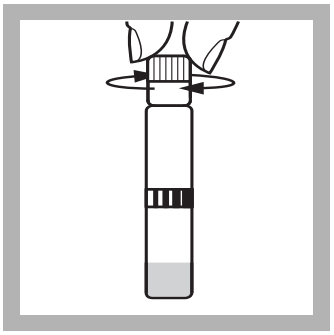
TNTplus Method 10205



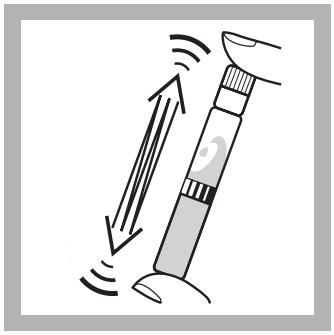
1. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



2. Carefully pipet 5.0 mL of sample into the vial. Immediately proceed to step 3.



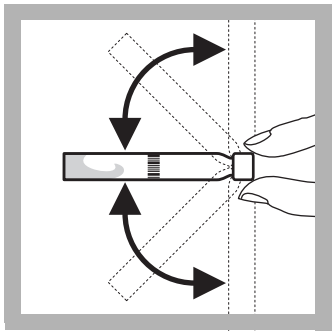
3. Flip the DosiCap ***Zip*** over so that the reagent side faces the vial. Screw the cap tightly onto the vial.



4. Shake the capped vial 2–3 times to dissolve the reagent in the cap.

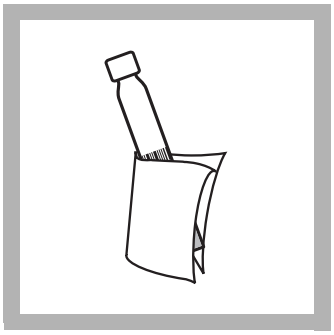
Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

5. Wait 15 minutes.



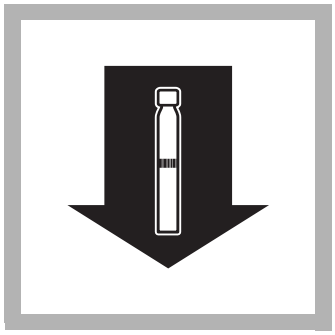
6. After 15 minutes, invert the sample an additional 2–3 times to mix.

The color remains constant for an additional 15 minutes after the timer expires.



7. Thoroughly clean the outside of the vial.

Install the Light Shield in Cell Compartment #2.



8. Insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L NH_3–N.

No instrument Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 8. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity the procedure is repeated and the color forming reagent that is present in the DosiCap ***Zip*** is not added.

To determine the sample blank run the procedure as given, but do not remove the foil from the DosiCap ***Zip*** in step 1 and replace the cap in its original position in step 3. The value obtained in step 8 is then subtracted from the value obtained on the original sample to give the corrected sample concentration.

Alternatively, samples that contain turbidity only may be first filtered through a membrane filter and then analyzed.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Primary amines are determined and cause high-bias results. A 10000-fold excess of urea does not interfere. All reducing agents interfere and cause low-bias results.

Important Note: *An analyte concentration greatly in excess of the stated range will adversely affect color formation, resulting in a false reading within the method range.*

Measurement results can be verified using sample dilutions or standard additions.

Samples with severe interferences require distillation. The manufacturer recommends the distillation procedure using the Hach General Purpose Distillation Set.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels
Cl^-, SO_4^{2-}	1000 mg/L
K^+, Na^+, Ca^{2+}	500 mg/L
CO_3^{2-}, NO_3^-, Fe^{3+}, Cr^{3+}, Cr^{6+}, Zn^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Hg^{2+}	50 mg/L
Fe^{2+}	25 mg/L
Sn^{2+}	10 mg/L
Pb^{2+}	5 mg/L
Ag^+	2 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. Preserve the samples by reducing the pH to 2 or less with at least 2 mL of Hydrochloric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to 20–23 °C (68–73.4 °F) and neutralize to pH 7.0 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the method with a 1.0 mg/L ammonia nitrogen standard. Use 5.0 mL of this 1.0 mg/L standard in place of the sample in step 2.

2. Alternately, use 5.0 mL of a Wastewater Effluent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 2 mg/L ammonia nitrogen in the presence of other ions such as nitrate, phosphate and sulfate.

Summary of Method

Ammonium ions react at pH 12.6 with hypochlorite ions and salicylate ions in the presence of sodium nitroprusside as a catalyst to form indophenol. The amount of color formed is directly proportional to the ammonia nitrogen present in the sample. Test results are measured at 690 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Ammonia, ULR TNT830 Reagent Set	25 tests	TNT830

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 1-5 mL	1	each	27951-00
Pipettor Tips, for 27951-00 pipettor	1	100/pkg	27952-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrogen Ammonia Standard Solution, 1.0-mg/L NH_3–N	500 mL	1891-49
Wastewater, Effluent Inorganics Standard, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49
Water, deionized	4L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL, 12/pkg	20870-79
Distillation Set, general purpose	22653-00
Filter Holder, glass for vacuum filtration (SUVA)	2340-00
Filter membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	546-53
Hydrochloric Acid Standard Solution, 1 N	23213-53
Hydrochloric Acid, concentrated ACS	134-49
Sodium Hydroxide Standard Solution, 1 N	1045-32
Test Tube Rack for 13-mm vial	24979-00
Tubing, rubber, 12-ft	560-19

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Updated November 2005 Edition 1

Nitrogen, Ammonia

Method 10205 **Salicylate Method**

TNTplus™ 831 **LR (1 to 12 mg/L NH_3–N)**

Scope and Application: For surface waters, municipal and industrial wastewaters.

Test Preparation

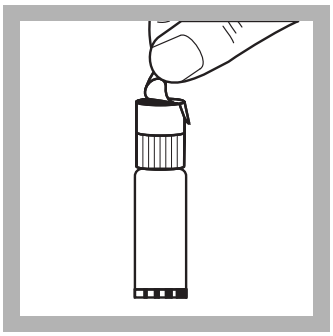
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample pH is 4–8.
Recommended sample and reagent temperature is 20–23 °C (68–73.4 °F). Incorrect results may be obtained if test is not performed at the recommended temperature.
Recommended reagent storage is 2–8 °C (35.6–46.4 °F).
Analyze samples as soon as possible for best results.
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.

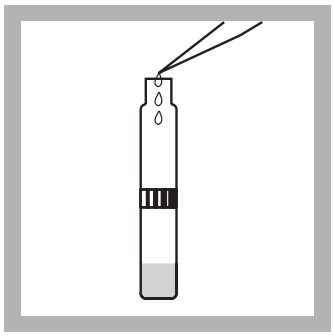
Collect the following items:	**Quantity**
Ammonia, TNTplus LR TNT831 Reagent Set	1 vial
Light Shield	1
Pipettor for 0.5 mL sample	1
Pipettor Tip	varies

Note: *Reorder information for consumables and replacement items is on page 4.*

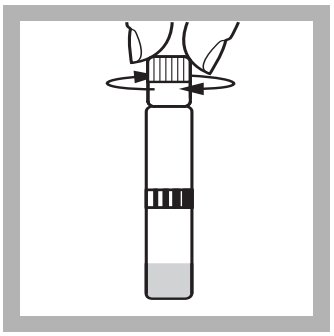
TNTplus Method 10205



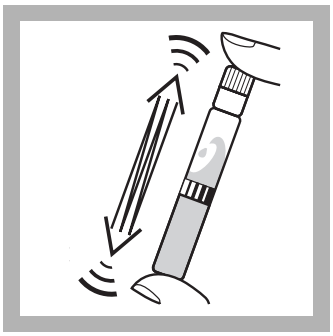
1. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



2. Carefully pipet 0.5 mL (500 µL) of sample into the vial. Immediately proceed to step 3.



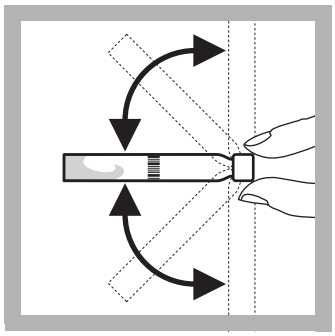
3. Flip the DosiCap ***Zip*** over so that the reagent side faces the vial. Screw the cap tightly onto the vial.



4. Shake the capped vial 2–3 times to dissolve the reagent in the cap.

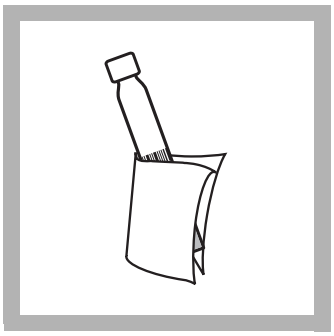
Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

5. Wait 15 minutes.



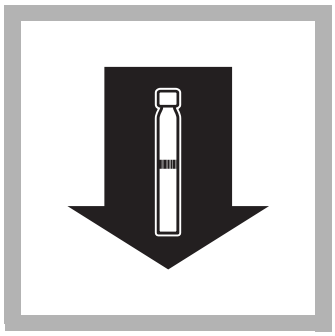
6. After 15 minutes, invert the sample an additional 2–3 times to mix.

The color remains constant for an additional 15 minutes after the timer expires.



7. Thoroughly clean the outside of the vial.

Install the Light Shield in Cell Compartment #2.



8. Insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L NH_3–N.

No instrument Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 8. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity the procedure is repeated and the color forming reagent that is present in the DosiCap ***Zip*** is not added.

To determine the sample blank run the procedure as given, but do not remove the foil from the DosiCap ***Zip*** in step 1 and replace the cap in its original position in step 3. The value obtained in step 8 is then subtracted from the value obtained on the original sample to give the corrected sample concentration.

Alternatively, samples that contain turbidity only may be first filtered through a membrane filter and then analyzed.

Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Primary amines are determined and cause high-bias results. A 10000-fold excess of urea does not interfere. All reducing agents interfere and cause low-bias results.

Important Note: *An analyte concentration greatly in excess of the stated range will adversely affect color formation, resulting in a false reading within the method range.*

Measurement results can be verified using sample dilutions or standard additions.

Samples with severe interferences require distillation. The manufacturer recommends the distillation procedure using the Hach General Purpose Distillation Set.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels
Cl^-, SO_4^{2-}	1000 mg/L
K^+, Na^+, Ca^{2+}	500 mg/L
CO_3^{2-}, NO_3^-, Fe^{3+}, Cr^{3+}, Cr^{6+}, Zn^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Hg^{2+}	50 mg/L
Fe^{2+}	25 mg/L
Sn^{2+}	10 mg/L
Pb^{2+}	5 mg/L
Ag^+	2 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. Preserve the samples by reducing the pH to 2 or less with at least 2 mL of Hydrochloric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to 20–23 °C (68–73.4 °F) and neutralize to pH 7.0 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method
Check the accuracy of the method with a 10 mg/L ammonia nitrogen standard. Use 0.5 mL of this 10 mg/L standard in place of the sample in step 2.

Summary of Method

Ammonium ions react at pH 12.6 with hypochlorite ions and salicylate ions in the presence of sodium nitroprusside as a catalyst to form indophenol. The amount of color formed is directly proportional to the ammonia nitrogen present in the sample. Test results are measured at 690 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Ammonia, TNTplus ULR TNT831 Reagent Set	25 tests	TNT831

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27950-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrogen Ammonia Standard Solution, 10 mg/L NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Solution, 100 mg/L NH_3–N	500 mL,	24065-49
Wastewater, Effluent Inorganics Standard, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49
Water, deionized	4L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL, 12/pkg	20870-79
Distillation Set, general purpose	22653-00
Filter Holder, glass for vacuum filtration (SUVA)	2340-00
Filter membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	546-53
Hydrochloric Acid Standard Solution, 1 N	23213-53
Hydrochloric Acid, concentrated ACS	134-49
Sodium Hydroxide Standard Solution, 1 N	1045-32
Test Tube Rack for 13-mm vial	24979-00
Tubing, rubber, 12-ft	560-19

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Updated November 2005 Edition 1

Nitrogen, Ammonia

Method 10205 **Salicylate Method**

TNTplus™ 832 **HR (2 to 47 mg/L NH_3–N)**

Scope and Application: For surface waters, municipal and industrial wastewaters.

Test Preparation

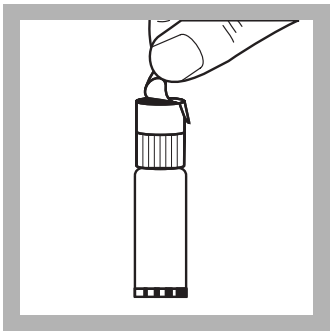
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample pH is 4–8.
Recommended sample and reagent temperature is 20–23 °C (68–73.4 °F). Incorrect results may be obtained if test is not performed at the recommended temperature.
Recommended reagent storage is 2–8 °C (35.6–46.4 °F).
Analyze samples as soon as possible for best results.
TNTplus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

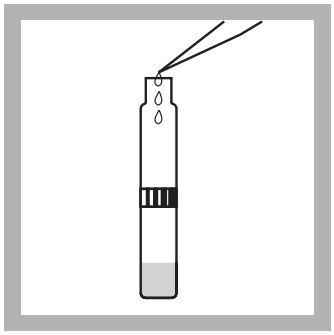
Collect the following items:	**Quantity**
Ammonia, HR TNT832 Reagent Set	1 vial
Light Shield	1
Pipettor for 0.2 mL sample	1
Pipettor Tip	varies

Note: *Reorder information for consumables and replacement items is on page 4.*

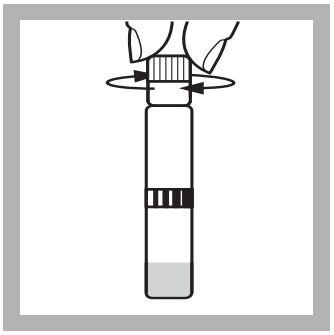
TNTplus Method 10205



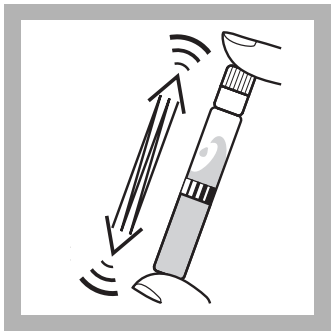
1. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



2. Carefully pipet 0.2 mL (200 µL) of sample into the vial. Immediately proceed to step 3.



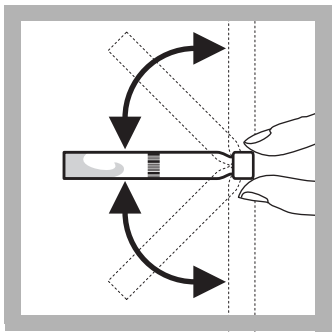
3. Flip the DosiCap ***Zip*** over so that the reagent side faces the vial. Screw the cap tightly onto the vial.



4. Shake the capped vial 2–3 times to dissolve the reagent in the cap.

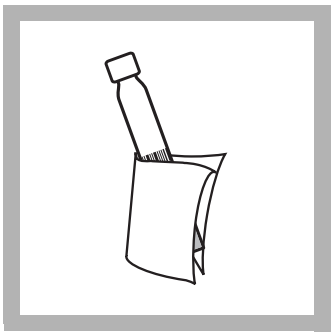
Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.

5. Wait 15 minutes.



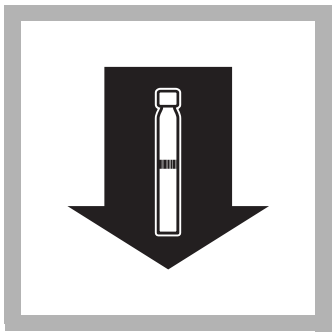
6. After 15 minutes, invert the sample an additional 2–3 times to mix.

The color remains constant for an additional 15 minutes after the timer expires.



7. Thoroughly clean the outside of the vial.

Install the Light Shield in Cell Compartment #2.



8. Insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L NH_3–N.

No instrument Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 8. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Colored or turbid samples can cause high results. To compensate for color or turbidity the procedure is repeated and the color forming reagent that is present in the DosiCap ***Zip*** is not added.

To determine the sample blank run the procedure as given, but do not remove the foil from the DosiCap ***Zip*** in step 1 and replace the cap in its original position in step 3. The value obtained in step 8 is then subtracted from the value obtained on the original sample to give the corrected sample concentration.

Alternatively, samples that contain turbidity only may be first filtered through a membrane filter and then analyzed.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined.

Primary amines are determined and cause high-bias results. A 10000-fold excess of urea does not interfere. All reducing agents interfere and cause low-bias results.

Important Note: *An analyte concentration greatly in excess of the stated range will adversely affect color formation, resulting in a false reading within the method range.*

Measurement results can be verified using sample dilutions or standard additions.

Samples with severe interferences require distillation. The manufacturer recommends the distillation procedure using the Hach General Purpose Distillation Set.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels
Cl^-, SO_4^{2-}	1000 mg/L
K^+, Na^+, Ca^{2+}	500 mg/L
CO_3^{2-}, NO_3^-, Fe^{3+}, Cr^{3+}, Cr^{6+}, Zn^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Hg^{2+}	50 mg/L
Fe^{2+}	25 mg/L
Sn^{2+}	10 mg/L
Pb^{2+}	5 mg/L
Ag^+	2 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. Preserve the samples by reducing the pH to 2 or less with at least 2 mL of Hydrochloric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to 20–23 °C (68–73.4 °F) and neutralize to pH 7.0 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the method with a 10 mg/L ammonia nitrogen standard. Use 0.2 mL of this 10 mg/L standard in place of the sample in step 2.

2. Alternately, use 0.2 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 15 mg/L ammonia nitrogen in the presence of other ions such as nitrate, phosphate and sulfate.

Summary of Method

Ammonium ions react at pH 12.6 with hypochlorite ions and salicylate ions in the presence of sodium nitroprusside as a catalyst to form indophenol. The amount of color formed is directly proportional to the ammonia nitrogen present in the sample. Test results are measured at 690 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Ammonia, ULR TNT832 Reagent Set	25 tests	TNT832

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27950-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrogen Ammonia Standard Solution, 10 mg/L NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Solution, 100 mg/L NH_3–N	500 mL,	24065-49
Wastewater, Influent Inorganics Standard, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49
Water, deionized	4L	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL, 12/pkg	20870-79
Distillation Set, general purpose	22653-00
Filter Holder, glass for vacuum filtration (SUVA)	2340-00
Filter membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	546-53
Hydrochloric Acid Standard Solution, 1 N	23213-53
Hydrochloric Acid, concentrated ACS	134-49
Sodium Hydroxide Standard Solution, 1 N	1045-32
Test Tube Rack for 13-mm vial	24979-00
Tubing, rubber, 12-ft	560-19

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Updated November 2005 Edition 1

Nitrogen, Free Ammonia

Method 10201 **Indophenol Method[1]**

Powder Pillows **(0.01 to 0.50 mg/L NH_3–N)**

Scope and Application: For controlling free ammonia levels during the production of chloramines, at booster stations and for monitoring free ammonia levels in potable distribution system waters.

[1] U.S. Patent 6,315,950

Test Preparation

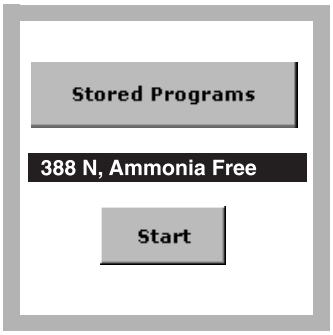
Before starting the test:

Use Method 10200, Nitrogen, Free Ammonia and Chloramine (Mono) to determine free ammonia and monochloramine simultaneously on the same sample.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

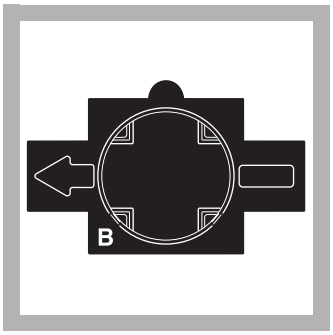
Collect the following items:	**Quantity**
Free Ammonia Reagent Set	—
Free Ammonia Reagent Solution	1 drop
Monochlor F Reagent Pillows	2
Sample Cell, 1-cm, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 6.*

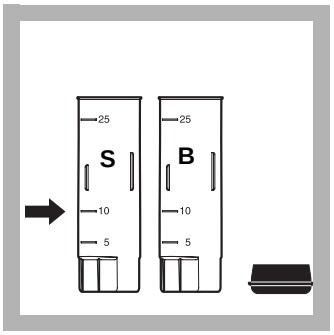
Multi-path Cell Method 10201



1. Select the Free Ammonia test.

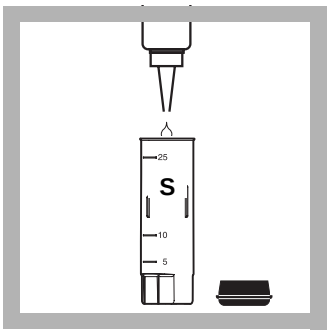


2. Insert Adapter B.



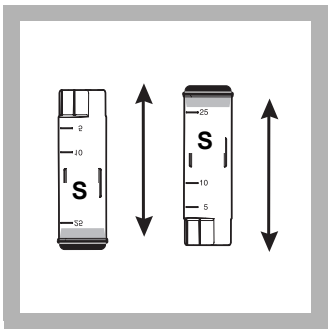
3. Fill two 1-cm cells to the 10-mL line with sample.

Label one cell ‘sample’ and one cell ‘blank’.



4. Prepared Sample: Add one drop of Free Ammonia Reagent Solution to the sample.

5. Cap the reagent bottle to maintain reagent performance and stability.



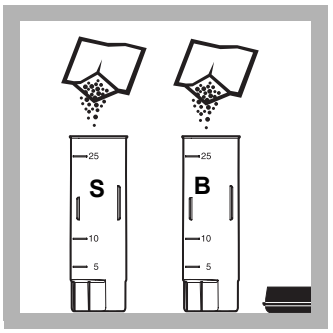
6. Cap and invert the sample to mix.

If the sample becomes cloudy by the end of the reaction period, pretreat the sample and retest. See Interferences on page 3.

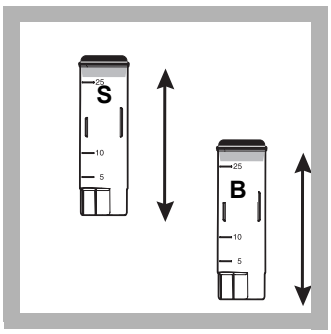
7. Press **TIMER>OK**.

A 5-minute reaction period will begin.

Color development time depends on sample temperature. For accurate results allow the full reaction period to occur. See Table 2 on page 4.



8. When the timer expires, add the contents of one MonoChlor F powder pillow to each cell.



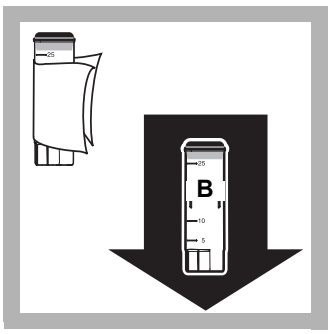
9. Cap and shake both cells about 20 seconds to dissolve the reagent.

A green color will develop if monochloramine is present.

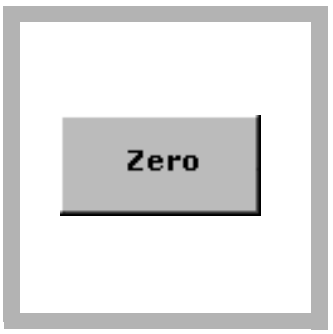
10. Press **TIMER>OK**.

A 5-minute reaction period will begin.

Color development time depends on sample temperature. For accurate results allow the full reaction period to occur. See Table 2 on page 4.



11. When the timer expires, insert the vial into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.

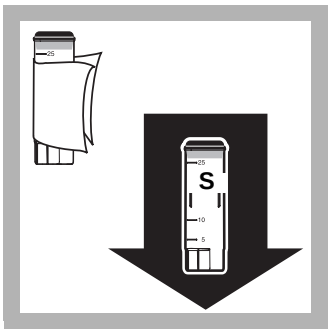


12. Press **ZERO**.

The display will show:

0.00 mg/L NH_3–N f

Remove the blank.



13. Insert the sample into the cell holder with the 1-cm (flat) path in line with the indicator arrow on the adapter.

14. Press **READ**.

Results are in mg/L NH_3–N f.

Interferences

This method is intended for finished, chloraminated drinking water samples that have a measurable combined (total) chlorine disinfectant residual. Samples where the disinfectant residual has disappeared and samples which exhibit a chlorine demand may produce low ammonia test results. Blanks and ammonia standards analyzed without a disinfectant residual must be prepared using high quality, reagent grade water.

The following do not interfere in free ammonia determination at or below the stated concentration:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	0.2 mg/L
Chloride	1200 mg/L Cl
Copper	1 mg/L Cu
Iron	0.3 mg/L Fe
Manganese	0.05 mg/L Mn
Nitrate	10 mg/L NO_3–N
Nitrite	1 mg/L NO_2–N
Phosphate	2 mg/L o–PO_4
Silica	100 mg/L SiO_2
Sulfate	1600 ppm as $CaCO_3$
Zinc	5 ppm Zn

Samples containing high levels of both Total Hardness and Alkalinity may become cloudy after the addition of the Free Ammonia Reagent Solution. If this occurs by the end of the first reaction period, the sample for Free Ammonia measurement must be pretreated as follows:

Note: *The blank does not need pretreatment.*

1. Measure 10 mL of sample into the cell for the labeled sample.

2. Add the contents of one Hardness Treatment Reagent Powder Pillow to the sample.

3. Cap the cell and invert until the reagent is dissolved.

4. Remove the cap.

5. Continue with the analysis at step 3 using the pretreated sample as the sample cell.

Color Development Time

Test results are strongly influenced by sample temperature. **Both reaction periods in the procedure are the same and depend on the temperature of the sample.** The reaction periods indicated in the procedure are for a sample temperature of 18–20 °C (64–68 °F). Adjust both reaction periods according to Table 2.

Table 2 Color Development Based on Sample Temperature

Sample Temperature		Development Time (minutes)
°C	°F	
5	41	10
7	45	9
9	47	8
10	50	8
12	54	7
14	57	7
16	61	6
18	64	5
20	68	5
23	73	2.5
25	77	2
greater than 25	greater than 77	2

Sampling and Storage

Collect samples in clean glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

Accuracy Check

Dilution water is required when testing a diluted sample and preparing standard solutions. Dilution water must be free of ammonia, chlorine and chlorine demand. A convenient source is a recirculating, deionizer system with carbon filtration which produces 18 megaohm-cm water.

Standard Additions Method

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentrations, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare three spiked samples. Measure 50 mL of sample into three 50-mL mixing cylinders.

5. Use the TenSette® Pipet to add 0.3, 0.6, and 1.0 mL of Ammonium Nitrogen Standard, 10 mg/L as NH_3-N to the three samples. Mix well.

6. Analyze each spiked sample starting with the 0.3 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery. Follow all steps in Method 10201.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Prepare a 0.20 mg/L ammonia nitrogen standard by diluting 2.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with dilution water. Or, using the TenSette Pipet, prepare a 0.20 mg/L ammonia nitrogen standard by diluting 0.4 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3–N, to 100 mL with dilution water. Analyze the Standard Solution, following all steps in Method 10201.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Monochloramine (NH_2Cl) and free ammonia (NH_3 and NH_4^+) can exist in the same water sample. Added hypochlorite combines with free ammonia to form more monochloramine. In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Free ammonia is determined by comparing the color intensities, with and without added hypochlorite. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Free Ammonia Reagent Set (50 tests), includes:	—	—	28797-00
(1) 28022-99, (1) 28773-36			
Free Ammonia Reagent Solution	1 drop	4 mL SCDB	28773-36
Monochlor F Reagent Pillows	2	100/pkg	28022-99

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Sample cell, multi-path	2	2/pkg	59405-06

Recommended Standards and Reagents

Description	Unit	Cat. No.
Hardness Treatment Reagent Pillows (1 per test)	50/pkg	28823-46
Nitrogen Ammonia Standard Solution, 10 mg/L as NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Ampule, 50 mg/L as NH_3–N, 10 mL	16/pkg	14791-10
Water, organic-free water	500-mL	26415-49

Recommended Apparatus

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet Filler, Safety Bulb	each	14651-00
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Thermometer, –10 to 110 °C	each	1877-01
Wipers, Disposable Kimwipes®, 30 x 30 cm, 280/box	box	20970-00

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Updated November 2005 Edition 1

Nitrogen, Total

Method 10072

Persulfate Digestion Method

Test ‘N Tube™ Vials

HR (2 to 150 mg/L N)

Scope and Application: For water and wastewater

Test Preparation

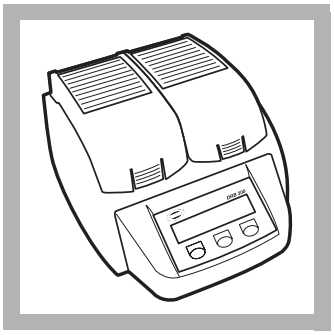
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Digestion is required for determining total nitrogen.
This test is technique-sensitive. Invert the vials as described here to avoid low results: Hold the vial in a vertical position with the cap pointing up. Turn the vial upside-down. Wait for all of the solution to flow down to the cap. Pause. Return the vial to an upright position. Wait for all the solution to flow to the bottom of the vial. This process equals one inversion.
If the test overranges, repeat the digestion and measurement with diluted sample. The digestion must be repeated for accurate results.
Use the deionized water provided in the reagent set or Organic-free Water to prepare the standards and perform the procedure.

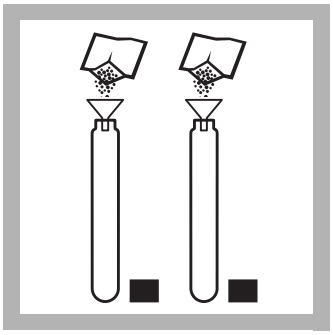
Collect the following items:	**Quantity**
Test ‘N Tube™ HR Total Nitrogen Reagent Set	1
DRB200 Reactor	1
Funnel, micro	1
Light Shield	1
Pipet, TenSette®, 0.1 to 1.0 mL plus tips	1
Pipet, TenSette®, 1.0 to 10.0 mL plus tips	1
Test Tube Cooling Rack	1–3

Note: *Reorder information for consumables and replacement items is on page 7.*

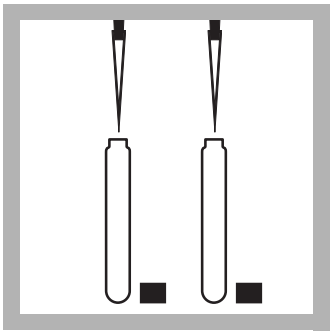
Test ‘N Tube Method 10072



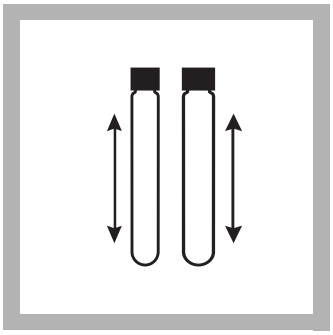
1. Turn on the DRB200 Reactor and heat to 105 °C.



2. Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two HR Total Nitrogen Hydroxide Digestion Reagent vials. Wipe off any reagent that may get on the lid or the tube threads.

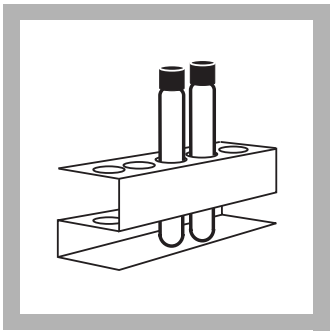


3. Add 0.5 mL of sample to a vial (this is the prepared sample). Add 0.5 mL of the deionized water included in the kit to a second vial (this is the reagent blank). Use only water that is free of all nitrogen-containing species as a substitute for the deionized water provided.

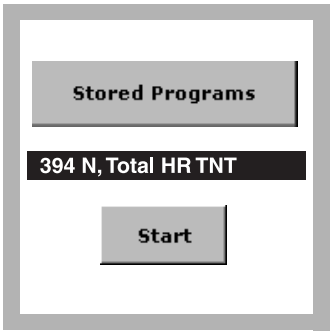


4. Cap both vials. Shake vigorously for at least 30 seconds to mix. The persulfate reagent may not dissolve completely after shaking. This will not affect accuracy.

5. Insert the vials in the reactor. Heat for exactly 30 minutes.

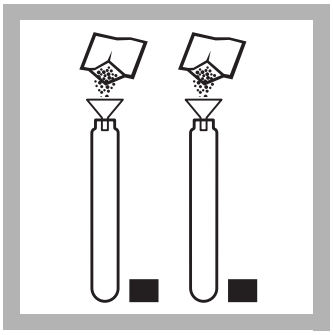


6. Using finger cots, immediately remove the hot vials from the reactor. Cool the vials to room temperature.

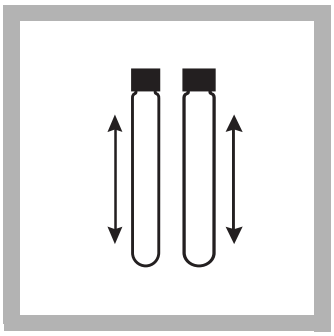


7. Select the test.

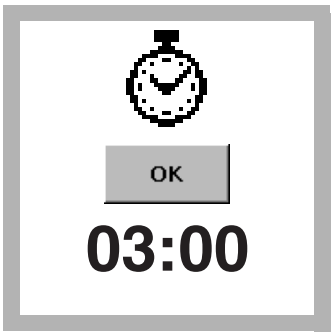
Install the Light Shield in Cell Compartment #2.



8. Remove the caps from the digested vials and add the contents of one Total Nitrogen (TN) Reagent A Powder Pillow to each vial.

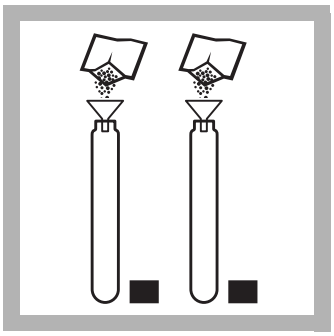


9. Cap the tubes and shake for 15 seconds.

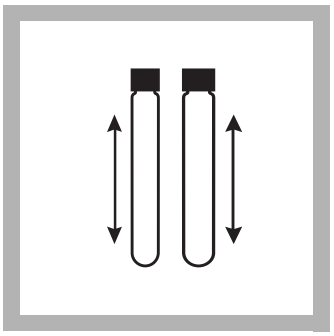


10. Press **TIMER>OK**.

A three-minute reaction period will begin.



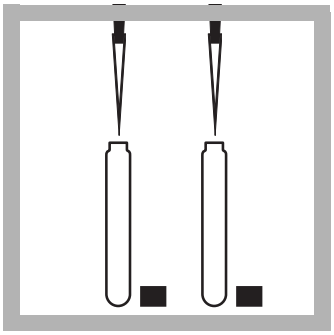
11. After the timer expires, remove the caps from the vials and add one TN Reagent B Powder Pillow to each vial.



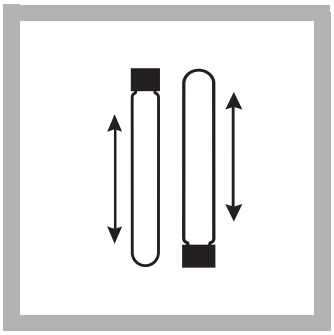
12. Cap the tubes and shake for 15 seconds. The reagent will not completely dissolve. This will not affect accuracy. The solution will begin to turn yellow.

13. Press **TIMER>OK**.

A two-minute reaction period will begin.



14. After the timer expires, remove the caps from two TN Reagent C vials and add 2 mL of digested, treated sample to one vial. Add 2 mL of digested, treated reagent blank to the second TN Reagent C vial.



15. Cap the vials and invert ten times to mix. Use slow, deliberate inversions for complete recovery.

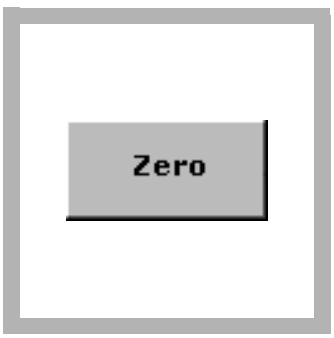
The tubes will be warm to the touch.

16. Press **TIMER>OK**.

A five-minute reaction period will begin.

The yellow color will intensify.

17. Wipe the reagent blank and insert it into the 16-mm round cell holder.



18. Press **ZERO**.

The display will show:

0.0 mg/L N

19. Wipe the reagent vial and insert it into the 16-mm round cell holder.

20. Press **READ**.

Results are in mg/L N.

Blanks for Colorimetric Measurement

The reagent blank may be used up to seven days for measurements using the same lots of reagents. Store it in the dark at room temperature (18–25 °C). If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

Interferences

The substances in Table 1 have been tested and found not to interfere up to the indicated levels (in mg/L). Interfering substances that resulted in a concentration change of ±10% appear in Table 2.

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Barium	10.4 mg/L
Calcium	1200 mg/L
Chromium ($^{3+}$)	2 mg/L
Iron	8 mg/L
Lead	26.4 µg/L
Magnesium	2000 mg/L
Organic Carbon	600 mg/L
pH	13 pH units
Phosphorus	400 mg/L
Silica	600 mg/L
Silver	3.6 mg/L
Tin	6 mg/L

Table 2 Interfering Substances

Substance	Level and Effect
Bromide	> 240 mg/L; positive interference
Chloride	> 3000 mg/L; positive interference

This test performed with standard nitrogen solutions prepared from the following compounds obtained 95% recovery:

- Ammonium chloride
- Ammonium sulfate
- Ammonium acetate
- Urea
- Glycine

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in ≥ 95% recovery.

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

This method generally yields 95–100% recovery on organic nitrogen standards. A set of three Kjeldahl Nitrogen Primary Standards is available for proof of accuracy.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 120-mg/L N standard. Use the deionized water included in the kit or water that is free of all organic and nitrogen-containing species.

   a. Weigh 1.6208 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   b. Weigh 2.1179 g of Glycine p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   c. Weigh 2.5295 g of Nicotinic p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{25(120)} \times 100$$

The percent recovery should be:

Table 3 % Recovery

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare three sample spikes. Fill three Mixing Cylinders with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of an Ammonia Nitrogen Standard Solution, 1000-mg/L as NH_3–N, respectively, to each sample and mix thoroughly.

5. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

6. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. For proof of accuracy, substitute 0.5 mL of a 100-mg/L ammonia nitrogen standard solution for the sample in the procedure.

2. To adjust the calibration curve using the reading obtained with a 100-mg/L N standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration. Press **OK**. Press **ADJUST**.

Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum at 410 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Test 'N Tube™ Total HR Nitrogen Reagent Set	50 vials	27141-00

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Funnel, micro	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg	21856-96
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	2	50/pkg	21997-96
Test Tube Cooling Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Ammonia Nitrogen Standard Sol., 1000-mg/L NH_3–N	1 L	23541-53
Ammonia Nitrogen Standard Sol., 100-mg/L NH_3–N	500 mL	24065-49
Balance, analytical SA80, 115 VAC	each	28014-01
Cylinder, mixing with stopper, 25 mL	each	20886-40
Flask, volumetric, Class A, 1000 mL	each	14574-53
Pipet tips for 19700-01	1000/pkg	21856-28
Pipet tips for 19700-10	250-pkg	21997-25
Primary Standard Set, for Kjeldahl Nitrogen	set of 3	22778-00
Sodium Hydroxide, 5 N	50 mL	2450-26
Sulfuric Acid	500 mL	979-49
Wastewater Mixed Inorganic Standard for NH_3-H, NO_3-N, PO4, COD, SO_4,TOC	500 mL	28331-49
Water, deionized	500 mL	272-49
Water, organic-free	500 mL	26415-49

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Updated November 2005 Edition 1

Nitrogen, Total

Method 10071

Persulfate Digestion Method

Test ‘N Tube™ Vials

LR (0.5 to 25.0 mg/L N)

Scope and Application: For water and wastewater.

Test Preparation

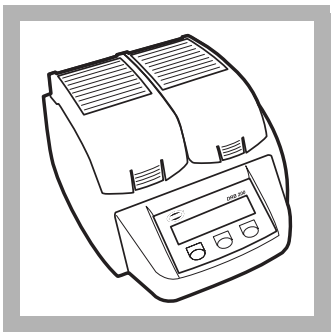
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Digestion is required for determining total nitrogen.
This test is technique-sensitive. Invert the vials as described here to avoid low results: Hold the vial in a vertical position with the cap pointing up. Turn the vial upside-down. Wait for all of the solution to flow down to the cap. Pause. Return the vial to an upright position. Wait for all the solution to flow to the bottom of the vial. This process equals one inversion.
If the test overranges, repeat the digestion and measurement with diluted sample. The digestion must be repeated for accurate results.
Use the deionized water provided in the reagent set or Organic-free Water to prepare the standards and perform the procedure.

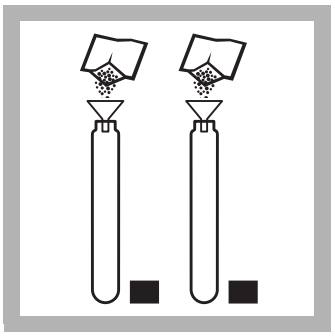
Collect the following items:	**Quantity**
Test ‘N Tube™ LR Total Nitrogen Reagent Set	1
DRB200 Reactor	1
Funnel, micro	1
Light Shield	1
Pipet, TenSette®, 1.0 to 10.0 mL plus tips	1
Test Tube Cooling Rack	1–3

Note: *Reorder information for consumables and replacement items is on page 7.*

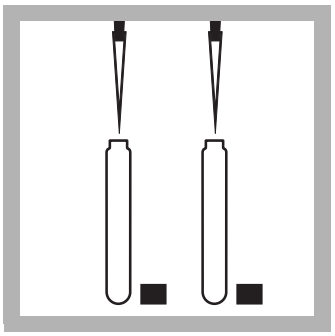
Test ‘N Tube Method 10071



1. Turn on the DRB200 Reactor and heat to 105 °C.



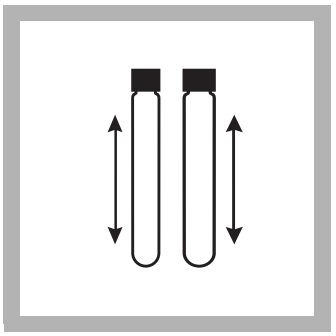
2. Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two Total Nitrogen Hydroxide Digestion Reagent vials. Wipe off any reagent that may get on the lid or the tube threads.



3. Prepared Sample: Add 2 mL of sample to one vial.

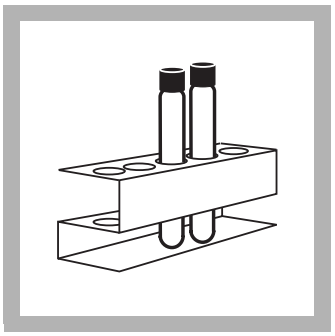
Blank Preparation: Add 2 mL of the deionized water included in the kit to a second vial.

Note: *Use only water that is free of all nitrogen-containing species as a substitute for the provided deionized water.*

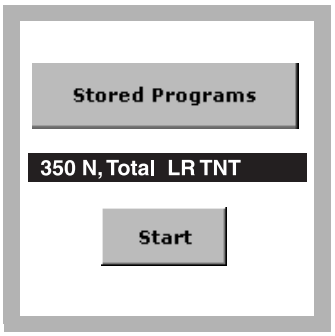


4. Cap both vials. Shake vigorously for at least 30 seconds to mix. The persulfate reagent may not dissolve completely after shaking. This will not affect accuracy.

5. Insert the vials in the reactor. Heat for exactly 30 minutes.

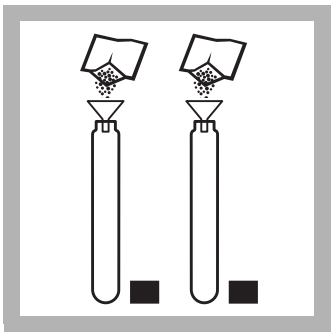


6. Using finger cots, immediately remove the hot vials from the reactor. Cool the vials to room temperature.

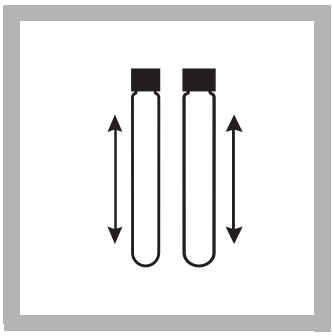


7. Select the test.

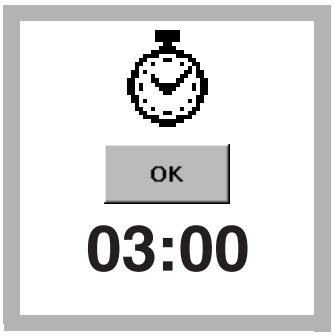
Install the Light Shield in Cell Compartment #2.



8. Remove the caps from the digested vials and add the contents of one Total Nitrogen (TN) Reagent A Powder Pillow to each vial.

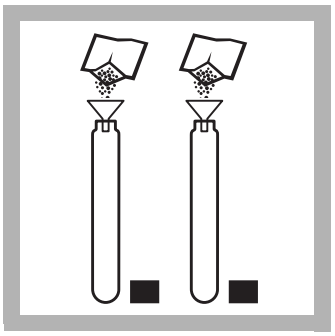


9. Cap the tubes and shake for 15 seconds.

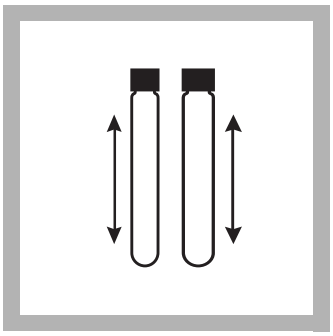


10. Press **TIMER>OK**.

A three-minute reaction period will begin.



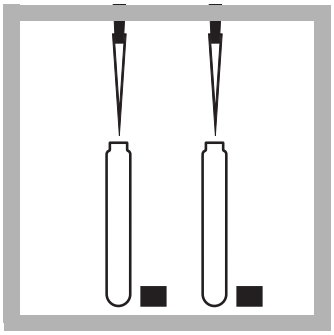
11. After the timer expires, remove the caps from the vials and add one TN Reagent B Powder Pillow to each vial.



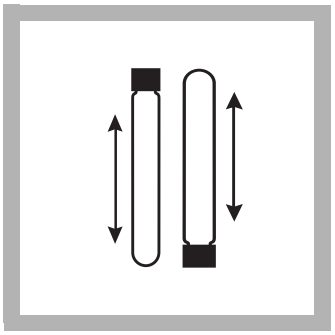
12. Cap the tubes and shake for 15 seconds. The reagent will not completely dissolve. This will not affect accuracy. The solution will begin to turn yellow.

13. Press **TIMER>OK**.

A two-minute reaction period will begin.



14. After the timer expires, remove the caps from two TN Reagent C vials and add 2 mL of digested, treated sample to one vial. Add 2 mL of digested, treated reagent blank to the second TN Reagent C vial.



15. Cap the vials and invert ten times to mix. Use slow, deliberate inversions for complete recovery.

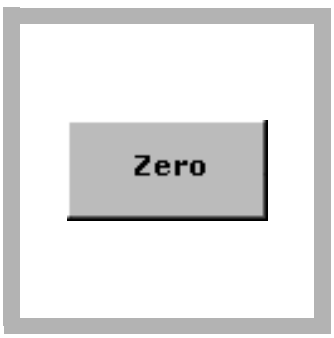
The tubes will be warm to the touch.

16. Press **TIMER>OK**.

A five-minute reaction period will begin.

The yellow color will intensify.

17. Wipe the reagent blank and insert it into the 16-mm round cell holder.

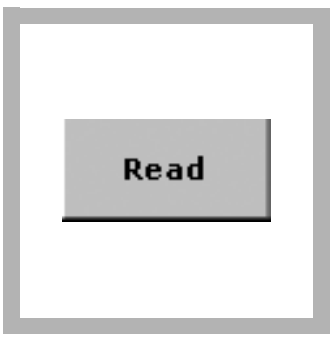


18. Press **ZERO**.

The display will show:

0.0 mg/L N

19. Wipe the reagent vial and insert it into the 16-mm round cell holder.



20. Press **READ**.

Results are in mg/L N.

Blanks for Colorimetric Measurement

The reagent blank may be used up to seven days for measurements using the same lots of reagents. Store it in the dark at room temperature (18–25 °C). If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

Interferences

The substances in Table 1 have been tested and found not to interfere up to the indicated levels (in mg/L). Interfering substances that resulted in a concentration change of ±10% appear in Table 2.

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Barium	2.6 mg/L
Calcium	300 mg/L
Chromium ($^{3+}$)	0.5 mg/L
Iron	2 mg/L
Lead	6.6 µg/L
Magnesium	500 mg/L
Organic Carbon	150 mg/L
pH	13 pH units
Phosphorus	100 mg/L
Silica	150 mg/L
Silver	0.9 mg/L
Tin	1.5 mg/L

Table 2 Interfering Substances

Substance	Level and Effect
Bromide	> 60 mg/L; positive interference
Chloride	> 1000 mg/L; positive interference

This test performed with standard nitrogen solutions prepared from the following compounds obtained 95% recovery:

- Ammonium chloride
- Ammonium sulfate
- Ammonium acetate
- Urea
- Glycine

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in ≥ 95% recovery.

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

This method generally yields 95–100% recovery on organic nitrogen standards. For proof of accuracy use Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 25-mg/L N standard. Use the deionized water included in the kit or water that is free of all organic and nitrogen-containing species.

   a. Weigh 0.3379 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   b. Weigh 0.4416 g of Glycine p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   c. Weigh 0.5274 g of Nicotinic p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{25} \times 100$$

The percent recovery is listed in Table 3

Table 3 Percent Recovery

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a fresh bottle of Ammonia Nitrogen Standard Solution, 1000-mg/L as NH_3–N.

5. Prepare three sample spikes. Fill three mixing cylinders with 50 mL of sample. Use the TenSette® Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Add 2 mL of each prepared solution, respectively, to three Total Nitrogen Hydroxide Reagent Vials.

7. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Standard Solution Method

1. For proof of accuracy, substitute 2 mL of a 10-mg/L ammonia nitrogen standard solution for the sample in the procedure. A single analyst should obtain less than 5% variation on replicates.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum at 410 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Test 'N Tube™ Total Nitrogen Reagent Set	50 vials	26722-45

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Funnel, micro	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	2	50/pkg	21997-96
Test Tube Cooling Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Ammonia Nitrogen Standard Solution, 1000-mg/L NH_3–N	1 L	23541-53
Ammonia Nitrogen Standard Solution, 10-mg/L NH_3–N	500 mL	153-49
Ammonia Nitrogen Standard Solution as N, 10 mg/L	500 mL	153-49
Primary Standard Set, for Kjeldahl Nitrogen	set of 3	22778-00
Wastewater Mixed Inorganic Standard for NH_3-H, NO_3-N, PO4, COD, SO_4,TOC	500 mL	28331-49
Water, deionized	500 mL	272-49
Water, organic-free	500 mL	26415-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Balance, analytical SA80, 115 VAC	each	28014-01
Cylinder, mixing with stopper, 50 mL	each	20886-41
Flask, volumetric, Class A, 1000 mL	each	14574-53
Pipet, TenSette, 0.1 to 1.0 mL	1	each
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg
Pipet tips for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet tips for TenSette Pipet 19700-10	250-pkg	21997-25
Sodium Hydroxide, 5 N	50 mL	2450-26
Sulfuric Acid, concentrated	500 mL	979-49

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Updated November 2005 Edition 1

Nitrogen, Total

Method 10208 **Persulfate Digestion Method**

TNTplus™ 826 **LR (1 to 16 mg/L N)**

Scope and Application: For water and wastewater

Test Preparation

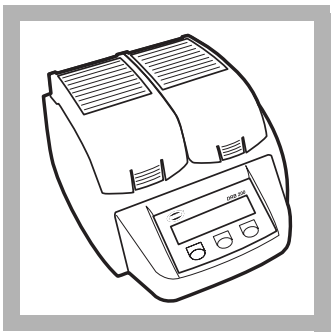
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Read the Safety Advice and Expiration Date on the package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 3–12.
Digestion is required for determining total nitrogen.
If test is not performed at the recommended temperature an incorrect result may be obtained.
Use only high quality deionized water or Organic Free Water for preparing nitrogen standards or making sample dilutions and reagent blanks.
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.
Important Note:
Sodium hydroxide solution A / Oxidant tablet B / MicroCap C: After addition of reagents A, B and C the reagent bottles must be reclosed **immediately**.
Reaction Tubes (Ø 20 mm): Do not use reaction tubes more than **13 times**. After use, clean thoroughly with a brush and water, then rinse well with nitrogen-free distilled water and dry.
Turbidity: Slight turbidity does not interfere; high turbidity after the addition of the MicroCap C should be allowed to settle before pipetting the digested sample.

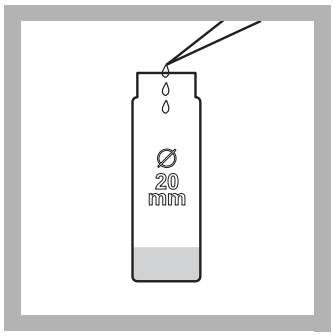
Collect the following items:	**Quantity**
Nitrogen, Total, LR TNT826 Reagent Set	1
DRB200 Reactor, 20-mm wells	1
Light Shield	1
Pipettor for 1–5 mL volumes	1
Pipettor Tips for 1–5 mL pipettor	2
Pipettor for 100–1000 µL sample	1
Pipettor Tips for 100–1000 µL pipettor	2

Note: *Reorder information for consumables and replacement items is on page 4.*

TNTplus Method 10208

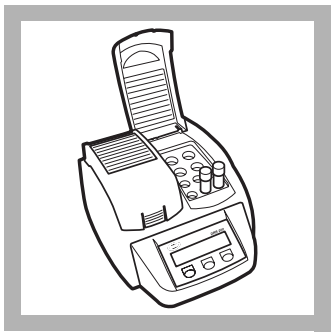


1. Turn on the DRB200 Reactor and heat to 100 °C.

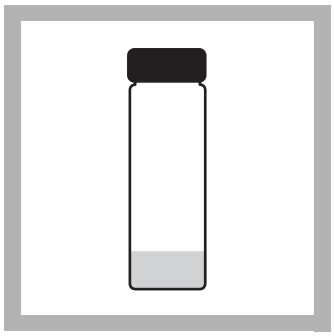


2. Add **1.3 mL** of sample, **1.3 mL** of Solution A, and **1** Reagent B tablet in quick succession to a dry 20-mm reaction tube.

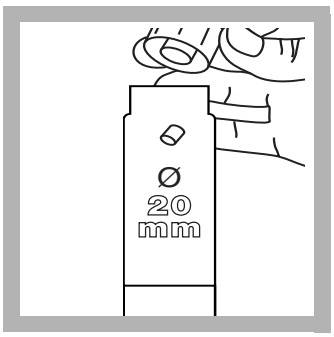
Close the reaction tube immediately. **Do not invert.**



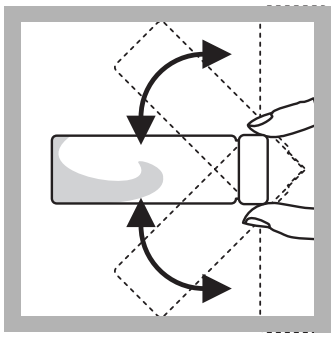
3. Insert the reaction tubes in the reactor. Heat for one hour.



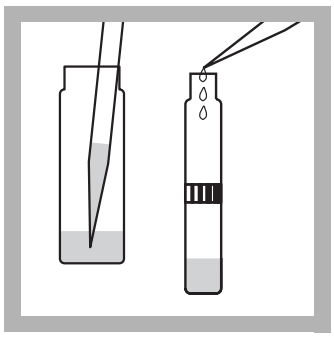
4. Remove the hot reaction tubes from the reactor. Cool the vials to room temperature (15–20 °C).



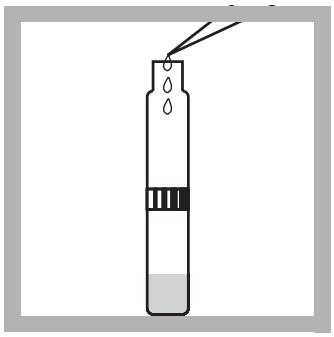
5. After the reaction tube has cooled, remove the cap and add 1 Micro Cap C to the tube.



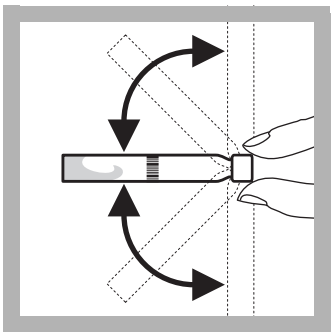
6. Cap and invert the reaction tube 2–3 times until no more streaks can be seen in the reaction tube solution.



7. Pipet 0.5 mL (500 µL) of the digested sample from the reaction tube into a test vial.



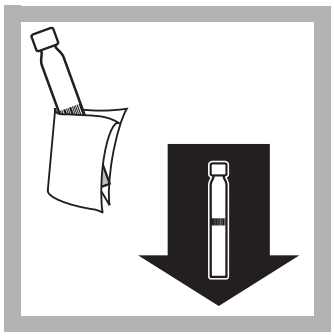
8. Pipet 0.2 mL (200 µL) of Solution D into the test vial.



9. Quickly cap and invert the test vial 2–3 times until no more streaks can be seen in the vial solution.

10. Wait 15 minutes.

Install the Light Shield in Cell Compartment #2.



11. After the timer expires wipe the vial and Insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L N.

No instrument zero is required.

Reagent Blanks

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use nitrogen-free deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 11. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. The cumulative effects of these ions or the influence of other ions have not been determined.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
COD	400 mg/L
Chloride	800 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to 15–25 °C and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of method with a 10 mg/L ammonia nitrogen standard. Use 1.3 mL this 10 mg/L standard in place of the sample in step 2.

2. Alternately, use 1.3 mL of a Wastewater Effluent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 2 mg/L ammonia nitrogen and 4 mg/L nitrate nitrogen to give a combined standard of 6 mg/L as total nitrogen.

Summary of Method

Inorganically and organically bonded nitrogen is oxidized to nitrate by digestion with peroxodisulphate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulphuric and phosphoric acid to form a nitrophenol. Test results are measured at 345 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Nitrogen Total, LR TNT826 Reagent Set	25 vials	TNT826

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable, 1–5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	2	100/pkg	27952-00
Pipet, variable volume, 100–1000 µL	1	each	27949-00
Pipet Tips, for 27949-00 pipet	1	400/pkg	27950-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Ammonia Nitrogen Standard Sol., 1000-mg/L NH_3–N	1 L	23541-53
Ammonia Nitrogen Standard Sol., 10-mg/L NH_3–N	500 mL	153-49
Sodium Hydroxide, 5 N	50 mL	2450-26
Sulfuric Acid	500 mL	979-49
Wastewater Mixed Inorganic Standard for NH_3-H, NO_3-N, PO_4, COD, SO_4,TOC	500 mL	28332-49
Water, deionized	500 mL	272-49
Water, organic-free	500 mL	26415-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
DRB200 Reactor, 115 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-08
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05

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Updated November 2005 Edition 1

Nitrogen, Total

Method 10208 **Persulfate Digestion Method**

TNTplus™ 827 **HR (5 to 40 mg/L N)**

Scope and Application: For water and wastewater

Test Preparation

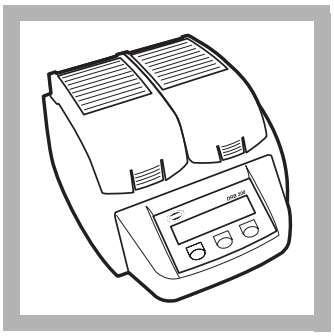
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Read the Safety Advice and Expiration Date on the package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 3–12.
Digestion is required for determining total nitrogen.
If test is not performed at the recommended temperature an incorrect result may be obtained.
Use only high quality deionized water or Organic Free Water for preparing nitrogen standards or making sample dilutions and reagent blanks.
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.
Important Note:
Sodium hydroxide solution A / Oxidant tablet B / MicroCap C: After addition of reagents A, B and C the reagent bottles must be reclosed **immediately**.
Reaction Tubes (20-mm): Do not use reaction tubes more than **13 times**. After use, clean thoroughly with a brush and water, then rinse well with nitrogen-free distilled water and dry.
Turbidity: Slight turbidity does not interfere; high turbidity after the addition of the MicroCap C should be allowed to settle before pipetting the digested sample.

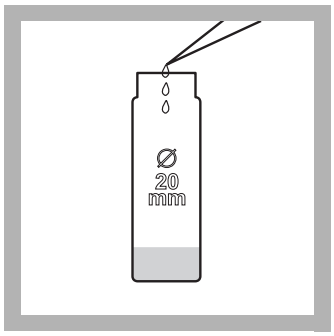
Collect the following items:	**Quantity**
Nitrogen, Total, HR TNT827 Reagent Set	1
DRB200 Reactor, 20-mm wells	1
Light Shield	1
Pipettor for 1–5 mL volumes	1
Pipettor Tips for 1–5 mL pipettor	2
Pipettor for 100–1000 µL sample	1
Pipettor Tips for 100–1000 µL pipettor	2

Note: *Reorder information for consumables and replacement items is on page 4.*

TNTplus Method 10208

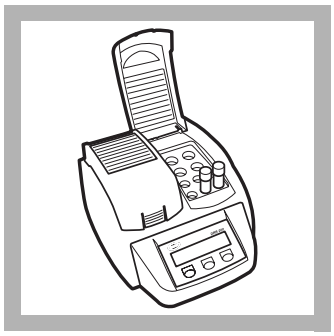


1. Turn on the DRB200 Reactor and heat to 100 °C.

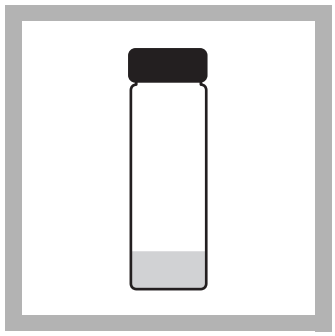


2. Add **10.5 mL** (500 µL) of sample, **2.0 mL** of Solution A, and **1** Reagent B tablet in quick succession to a dry 20-mm reaction tube.

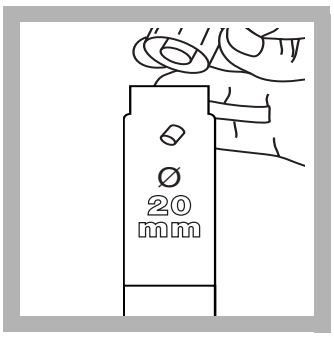
Close the reaction tube immediately. **Do not invert.**



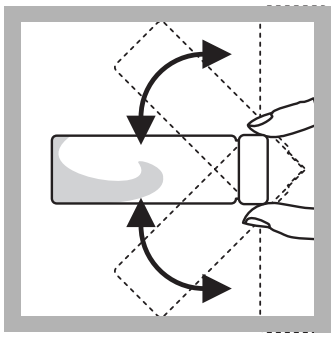
3. Insert the reaction tubes in the reactor. Heat for one hour.



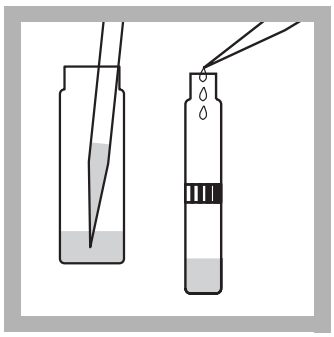
4. Remove the hot reaction tubes from the reactor. Cool the vials to room temperature (15–20 °C).



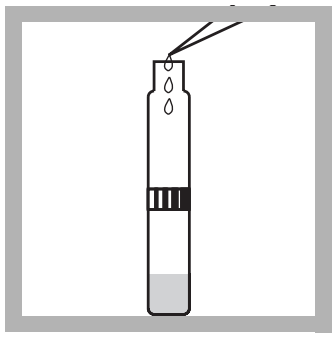
5. After the reaction tube has cooled, remove the cap and add 1 Micro Cap C to the tube.



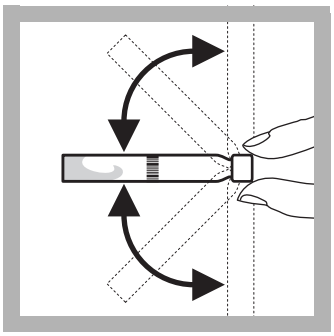
6. Cap and invert the reaction tube 2–3 times until no more streaks can be seen in the reaction tube solution.



7. Pipet 0.5 mL (500 µL) of the digested sample from the reaction tube into a test vial.



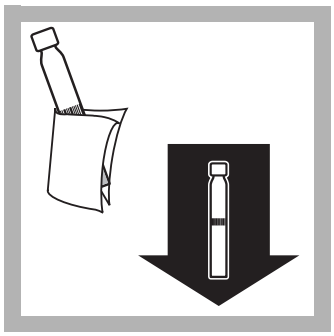
8. Pipet 0.2 mL (200 µL) of Solution D into the test vial.



9. Quickly cap and invert the test vial 2–3 times until no more streaks can be seen in the vial solution.

10. Wait 15 minutes.

Install the Light Shield in Cell Compartment #2.



11. After the timer expires wipe the vial and insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L N.

No instrument zero is required.

Reagent Blanks

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use nitrogen-free deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 11. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. The cumulative effects of these ions or the influence of other ions have not been determined.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
COD	1000 mg/L
Chloride	2000 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to 15–25 °C and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of method with a 10 mg/L ammonia nitrogen standard. Use 0.5 mL this 10 mg/L standard in place of the sample in step 2.

2. Alternately, use 0.5 mL of a Wastewater Effluent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 15 mg/L ammonia nitrogen and 10 mg/L nitrate nitrogen to give a combined standard of 25 mg/L as total nitrogen.

Summary of Method

Inorganically and organically bonded nitrogen is oxidized to nitrate by digestion with peroxodisulphate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulphuric and phosphoric acid to form a nitrophenol. Test results are measured at 345 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Nitrogen Total, HR TNT827 Reagent Set	25/pkg	TNT827

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable, 1–5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	2	100/pkg	27952-00
Pipet, variable volume, 100–1000 µL	1	each	27949-00
Pipet Tips, for 27949-00 pipet	1	400/pkg	27950-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Ammonia Nitrogen Standard Sol., 1000-mg/L NH_3–N	1 L	23541-53
Ammonia Nitrogen Standard Sol., 10-mg/L NH_3–N	500 mL	153-49
Sodium Hydroxide, 5 N	50 mL	2450-26
Sulfuric Acid	500 mL	979-49
Wastewater, Influent, Mixed Inorganic Standard for NH_3-H, NO_3-N, PO_4, COD, SO_4,TOC	500 mL	28331-49
Water, deionized	500 mL	272-49
Water, organic-free	500 mL	26415-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
DRB200 Reactor, 115 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-08
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05

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Updated November 2005 Edition 1

Nitrogen, Total

Method 10208 **Persulfate Digestion Method**

TNTplus™ 828 **UHR (20 to 100 mg/L N)**

Scope and Application: For water and wastewater

Test Preparation

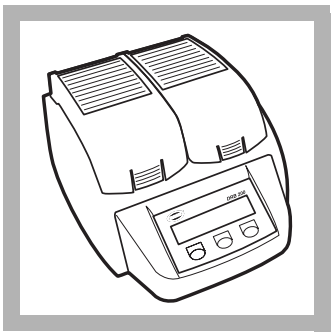
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Read the Safety Advice and Expiration Date on the package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 3–12.
Digestion is required for determining total nitrogen.
If test is not performed at the recommended temperature an incorrect result may be obtained.
Use only high quality deionized water or Organic Free Water for preparing nitrogen standards or making sample dilutions and reagent blanks.
TNTplus methods are activated from the Main Menu when the sample vial is inserted into the sample cell holder.
Important Note:
Sodium hydroxide solution A / Oxidant tablet B / MicroCap C: After addition of reagents A, B and C the reagent bottles must be reclosed **immediately**.
Reaction Tubes (20-mm): Do not use reaction tubes more than **13 times**. After use, clean thoroughly with a brush and water, then rinse well with nitrogen-free distilled water and dry.
Turbidity: Slight turbidity does not interfere; high turbidity after the addition of the MicroCap C should be allowed to settle before pipetting the digested sample.

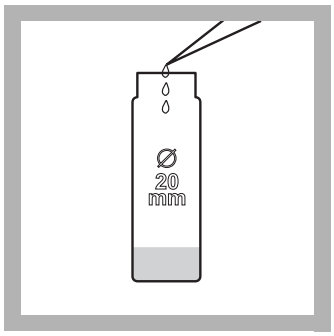
Collect the following items:	**Quantity**
Nitrogen, Total, UHR TNT828 Reagent Set	1
DRB200 Reactor, 20-mm wells	1
Light Shield	1
Pipet for 1–5 mL volumes	1
Pipet Tips for 1–5 mL pipettor	2
Pipet for 100–1000 µL sample	1
Pipet Tips for 100–1000 µL pipettor	2

Note: *Reorder information for consumables and replacement items is on page 4.*

TNTplus Method 10208

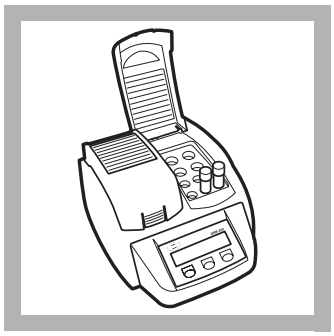


1. Turn on the DRB200 Reactor and heat to 100 °C.

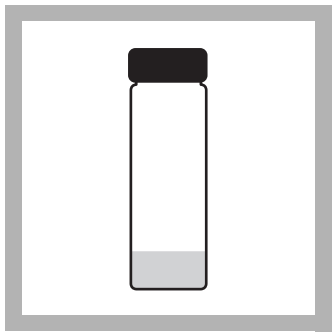


2. Add **0.2 mL** (200 µL) of sample, **2.3 mL** of Solution A, and **1** Reagent B tablet in quick succession to a dry 20-mm reaction tube.

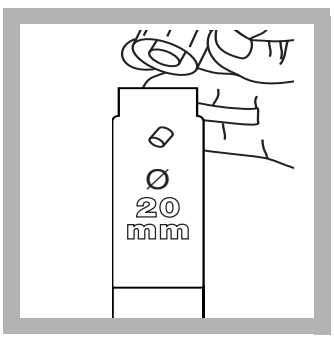
Close the reaction tube immediately. Do not invert.



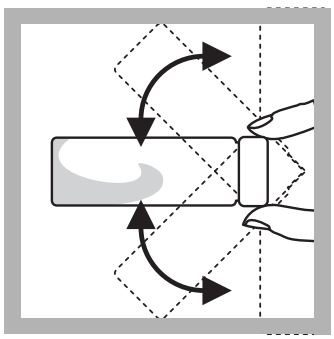
3. Insert the reaction tubes in the reactor. Heat for one hour.



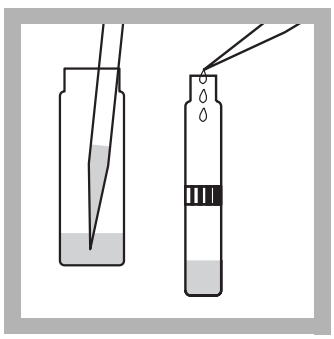
4. Remove the hot reaction tubes from the reactor. Cool the vials to room temperature (15–20 °C).



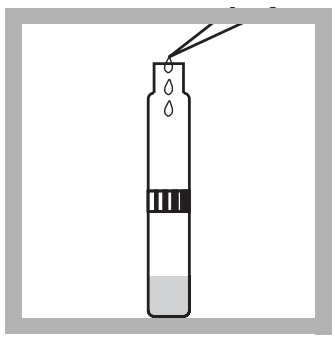
5. After the reaction tube has cooled, remove the cap and add **one** Micro Cap C to the tube.



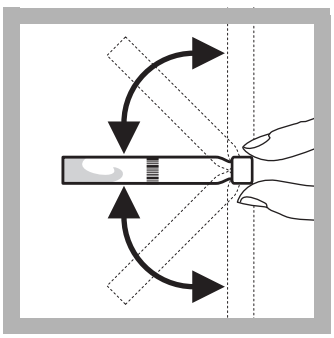
6. Cap and invert the reaction tube 2–3 times until no more streaks can be seen in the reaction tube solution.



7. Pipet 0.5 mL (500 µL) of the digested sample from the reaction tube into a test vial.



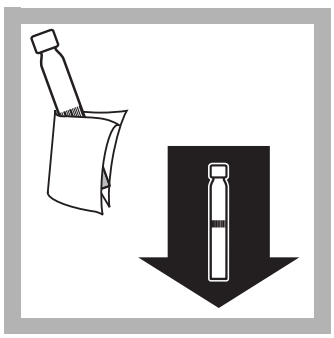
8. Pipet 0.2 mL (200 µL) of Solution D into the test vial.



9. Quickly cap and invert the test vial 2–3 times until no more streaks can be seen in the vial solution.

10. Wait 15 minutes.

Install the Light Shield in Cell Compartment #2.



11. After the timer expires wipe the vial and insert the prepared vial into the cell holder.

The instrument reads the barcode, then selects and performs the correct test. Results are in mg/L N.

No instrument zero is required.

Reagent Blanks

A reagent blank can be measured and the value subtracted from the results of each test performed using the same reagent lot number. Use nitrogen-free deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 11. Press **OPTIONS>MORE>REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. The cumulative effects of these ions or the influence of other ions have not been determined.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
COD	2500 mg/L
Chloride	5000 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL/L) Sulfuric Acid. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to 15–25 °C and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of method with a 50 mg/L ammonia nitrogen standard. Prepare the standard by pipetting 5.0 mL of a 1000 mg/L ammonia nitrogen standard into a 100 mL volumetric flask. Dilute to volume with deionized water, stopper and invert to mix. Use 0.2 mL this 50 mg/L standard in place of the sample in step 2. A 50-mg/L ammonia nitrogen standard solution is also available.

2. Alternately, use 0.2 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 15 mg/L ammonia nitrogen and 10 mg/L nitrate nitrogen to give a combined standard of 25 mg/L as total nitrogen.

Summary of Method

Inorganically and organically bonded nitrogen is oxidized to nitrate by digestion with peroxodisulphate. The nitrate ions react with 2,6-dimethylphenol in a solution of sulphuric and phosphoric acid to form a nitrophenol. Test results are measured at 345 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
Nitrogen Total, UHR TNT828 Reagent Set	25/pkg	TNT828

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13 mm + 2x20 mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13 mm + 2x20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable, 1–5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	2	100/pkg	27952-00
Pipet, variable volume, 100–1000 µL	1	each	27949-00
Pipet Tips, for 27949-00 pipet	1	400/pkg	27950-00
Test Tube Cooling Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Ammonia Nitrogen Standard Sol., 1000-mg/L NH_3–N	1 L	23541-53
Ammonia Nitrogen Standard, 50 mg/L NH_3 –N, 2-mL ampules	20/pkg	14791-20
Ammonia Nitrogen Standard, 50 mg/L NH_3 –N, 10-mL ampules	16/pkg	14791-10
Sulfuric Acid	500 mL	979-49
Wastewater Influent Mixed Inorganic Standard for NH_3-H, NO_3-N, PO4, COD, SO_4,TOC	500 mL	28331-49
Water, deionized	500 mL	272-49
Water, organic-free	500 mL	26415-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-08
Pipet, volumetric 5.0 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00
Flask, volumetric, 100 mL	each	14574-42
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05

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Updated November 2005 Edition 1

Nitrogen, Total Inorganic

Method 10021 **Titanium Trichloride Reduction Method**

Test ‘N Tube™ Vials **(0.2 to 25.0 mg/L N)**

Scope and Application: For water, wastewater, and seawater

Test Preparation

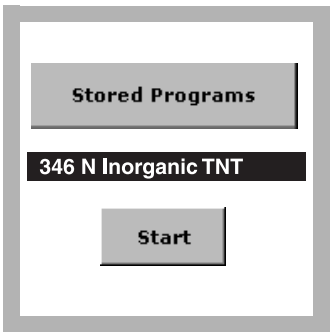
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
For safety, wear gloves while breaking ampules.
The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. Refer to the current MSDS for safe handling and disposal information.

Collect the following items:	**Quantity**
Total Inorganic Nitrogen Pretreatment Reagent Set ($TiCl_3$ Reduction Method)	1
Test ‘N Tube™ AmVer™ Nitrogen-Ammonia Reagent Set	1
Water, deionized	1 mL
Centrifuge	1
Funnel, micro	1
Light Shield	1
Pipet, TenSette®, 1.0–10.0 mL with tips	1
Pipette, volumetric, Class A, 1.00-mL	1
Test Tube Rack	1–3

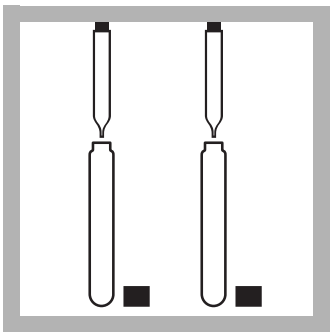
Note: *Reorder information for consumables and replacement items is on page 6.*

Test ‘N Tube Method 10021

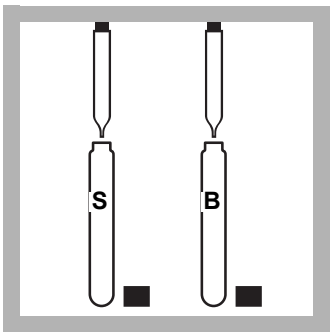


1. Select the test.

Install the Light Shield in Cell Compartment #2.

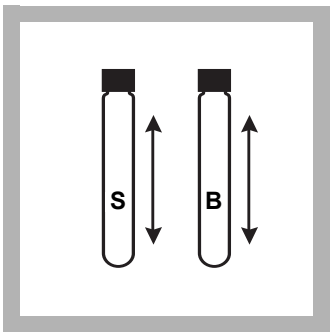


2. Pipet 1 mL of Total Inorganic Nitrogen Pretreatment Base concentrate into each of two Total Inorganic Nitrogen Pretreatment Diluent Vials.

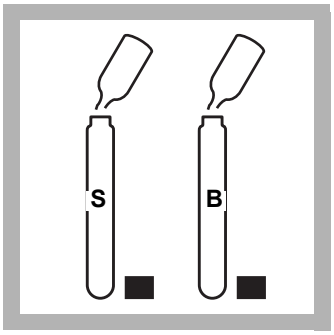


3. Prepared Sample: Pipet 1 mL of sample into one vial.

Blank Preparation: Pipet 1 mL of deionized water into the second vial.



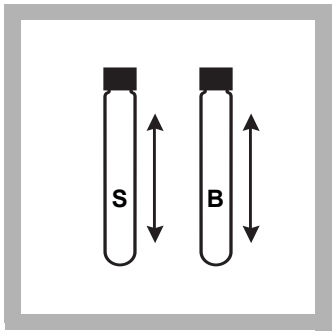
4. Cap the vials and shake for 30 seconds to mix.



5. Pour the contents of one Total Inorganic Nitrogen Reductant ampule into the vial containing the sample.

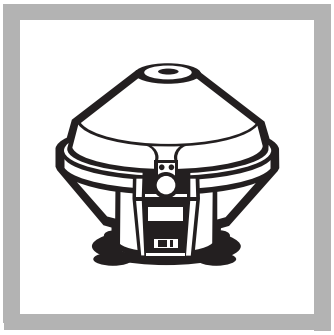
Pour the contents of another Total Inorganic Nitrogen Reductant ampule into the vial containing the blank.

A black precipitate will form immediately.



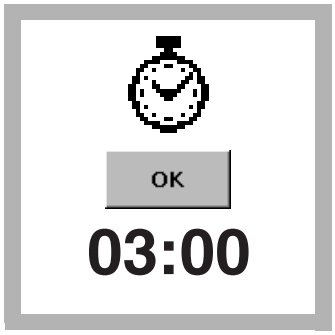
6. Immediately cap the vials. Shake gently for 30 seconds to mix the reagents. Allow the vials to stand for at least one minute.

The precipitate should remain black after shaking. Excessive shaking will cause the precipitate to turn white and cause low results.



7. Place the vials in a centrifuge.

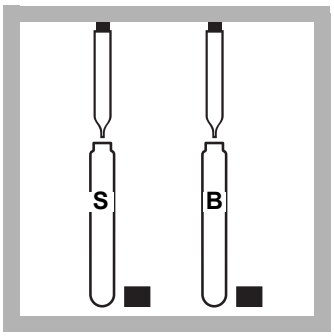
(If you do not have a centrifuge, wait at least 30 minutes for the solids to settle at the bottom of the vial. Proceed to step 9.)



8. Press **TIMER>OK**.

A three-minute timer will begin.

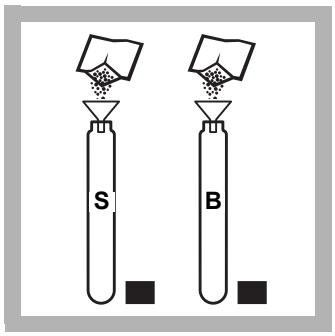
Centrifuge the vials for three minutes.



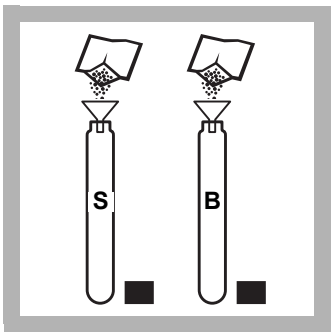
9. Using a pipet, add 2 mL of centrifuged sample to an AmVer™ Diluent Reagent Test 'N Tube™ for Low Range Ammonia Nitrogen.

Add 2 mL of centrifuged blank to another AmVer™ Dilutent Reagent Test 'N Tube™ for Low Range Ammonia Nitrogen.

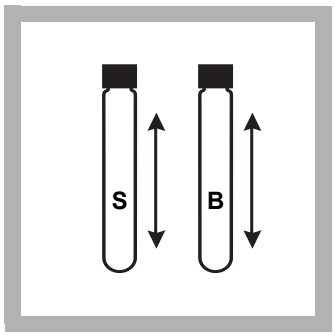
Pipet carefully to avoid disturbing the sediment.



10. Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow (for 5-mL samples) to each vial.



11. Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow (for 5-mL samples) to each vial.



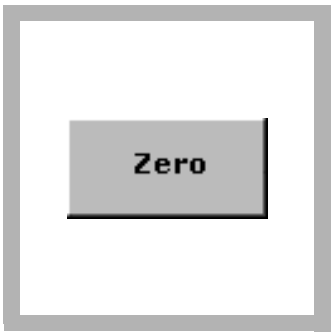
12. Cap the vials tightly and shake thoroughly to dissolve the powder.

A green color will develop if nitrogen is present.

13. Press **TIMER>OK**.

A 20-minute reaction period will begin.

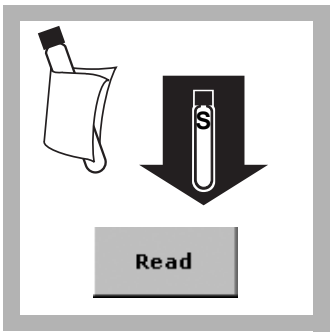
14. When the timer expires, wipe the blank and insert it into the 16-mm round cell holder.



15. Press **ZERO**.

The display will show:

0.0 mg/L N



16. Wipe the prepared sample and insert it into the 16-mm round cell holder.

Press **READ**. Results are in mg/L N.

Interferences

The substances in Table 1 may interfere when present. The substances in Table 2 do not interfere below the levels listed.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium	Causes a positive interference at 1000 mg/L as $CaCO_3$.
Manganese (IV)	Causes a negative interference at 3 mg/L.
Magnesium	Causes a positive interference at 1000 mg/L as $CaCO_3$.
Sulfide	Causes a negative interference at 3 mg/L.
Sulfate	Causes a negative interference at 250 mg/L.

Table 2 Non-interfering Substances and Levels

Substance	Levels Tested
Al^{3+}	8 mg/L
Ba^{2+}	40 mg/L
Cu^{2+}	40 mg/L
Fe^{3+}	8 mg/L
Zn^{2+}	80 mg/L
F^-	40 mg/L
PO_4^{3-}–P	8 mg/L
SiO_2	80 mg/L
EDTA	80 mg/L

Sample Collection, Preservation, and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N Sodium Thiosulfate* for each 0.3 mg/L Cl_2 in a one-liter sample. Preserve the sample by reducing the pH to 2 or less with concentrated (at least 2 mL) Hydrochloric Acid*. Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature. Neutralize with 5 N Sodium Hydroxide* before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a fresh HR Nitrate Nitrogen PourRite Ampule Standard, 500-mg/L NO_3^-–N.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. To check accuracy, use a 10.0-mg/L Nitrate Nitrogen Standard Solution. Alternatively, prepare this by diluting 1 mL of solution from a Nitrate Nitrogen Voluette® Ampule Standard, 500-mg/L NO_3^-–N, to 50 mL with deionized water. Perform the procedure are described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 6.

Method Performance

The total inorganic nitrogen test is designed to provide an estimate of the total nitrite, nitrate, and ammonia nitrogen load present in a water or wastewater sample. This test is most applicable to the monitoring of samples taken from an industrial process stream or a wastewater treatment stream where it is important to track the inorganic nitrogen load as it passes through the treatment process. The test does exhibit different recoveries of each of the three nitrogen species, as summarized below. The test is not recommended for use when quantifying only one of the three species. In that case, specific procedures for each particular analyte would be more appropriate.

Species Recovery

Nitrogen Form	Recovery
NH_3–N	112%
NO_3^-–N	100%
NO_2^-–N	77%

Summary of Method

Titanium (III) ions reduce nitrate and nitrite to ammonia in a basic environment. After centrifugation to remove solids, the ammonia is combined with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green colored solution. Test results are measured at 655 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Total Inorganic Nitrogen Pretreatment Reagent Set ($TiCl_3$ Reduction Method)	—	25 tests	26049-45
Test 'N Tube™ AmVer™ Nitrogen-Ammonia Reagent Set	—	25 tests	26045-45
Water, deionized	1 mL	100 mL	272-42

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Centrifuge, 115 VAC, 6 x 15 mL	1	each	26765-00
OR			
Centrifuge, 220 VAC, 6 x 15 mL	1	each	26765-02
Funnel, micro	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 1.0–10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	varies	50/pkg	21997-96
Pipette, volumetric, Class A, 1.00-mL	1	each	14515-35
Test Tube Rack	1	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Flask, volumetric Class A, 50-mL	each	14574-41
Nitrate Nitrogen Standard Solution, 10-mg/L NO_3^-–N	500 mL	307-49
Nitrate Nitrogen Standard Solution, 2-mL Ampule, 500-mg/L NO_3^-–N	20/pkg	14260-20
Pipet Filler, safety bulb	each	14651-00
Pipet, TenSette, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet Tips, for TenSette Pipet 19700-10	250-pkg	21997-28
Pipet, volumetric Class A, 1.00 mL	each	14515-35
Water, deionized	4 liters	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing	20886-40
Hydrochloric Acid, concentrated	134-49
Sodium Hydroxide, 5.0 N	2450-26
Sodium Thiosulfate, 0.1 N	323-32

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Updated November 2005 Edition 1

Nitrogen, Total Kjeldahl

Method 8075

Nessler Method[1] (Digestion Required)

(1 to 150 mg/L)

Scope and Application: For water, wastewater, and sludge; digestion is required.

[1] Adapted from Hach, et. al., *Journal of Association of Official Analytical Chemists*, 70(5) 783-787 (1987); Hach, et. al., *Journal of Agricultural and Food Chemistry*, 33(6) 1117-1123 (1985); *Standard Methods for the Examination of Water and Wastewater*

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
If using the Pour-Thru Module, periodically clean the cell by pouring a few sodium thiosulfate pentahydrate crystals into the cell funnel. Flush it through the funnel and cell with enough deionized water to dissolve. Rinse out the crystals.
Hold droppers and dropper bottles vertically, **not at an angle**, when dispensing reagent.
Nessler reagent contains mercuric iodide. Both the sample and blank will contain mercury (D009) at concentrations regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. Refer to the current MSDS for safe handling and disposal instructions.

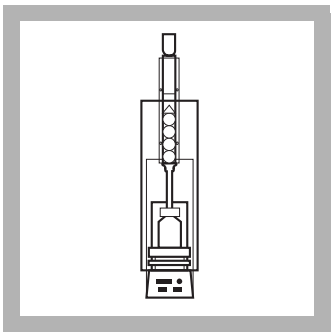
Collect the following items:	**Quantity**
Boiling Chips, silica carbide	2–3
Cots, finger	2
Cylinder, graduated mixing, 25 mL	2
Digesdahl Digestion Apparatus	1
Hydrogen Peroxide, 50%	20 mL
Mineral Stabilizer	6 drops
Nesslers Reagent	2 mL
Polyvinyl Alcohol Dispersing Agent	varies
Potassium Hydroxide Standard Solution, 1.0 N	varies
Potassium Hydroxide Standard Solution, 8.0 N	varies
Sulfuric Acid, ACS, concentrated	6 mL
TKN Indicator Solution	2 drops
Pipet, TenSette®, 0.1–1.0 mL, plus tips	1
Safety Shield	1
Sample Cell, 1-inch square, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 6.*

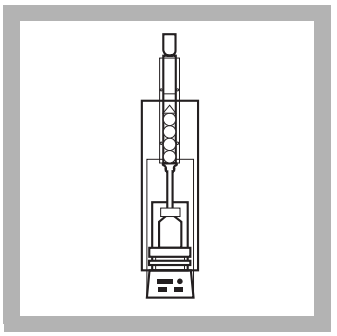
Nessler Method 8075

1. Press **STORED PROGRAMS**.

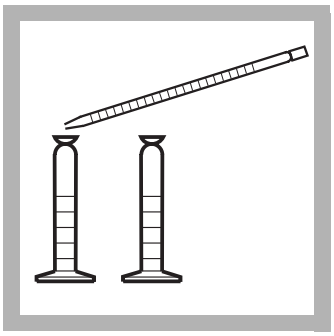
2. Select the test.



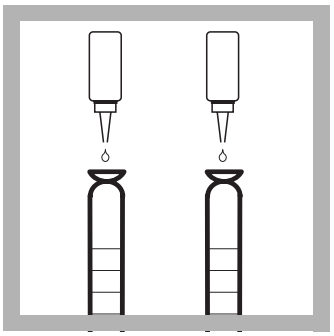
3. Prepared Sample: Digest the sample amount as described in the Digesdahl® Digestion Apparatus Instruction Manual.



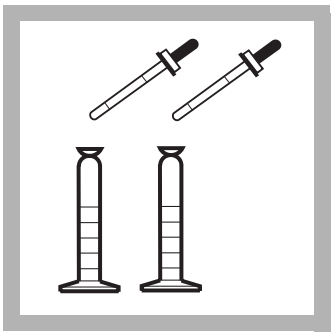
4. Blank Preparation: Digest an equal amount of deionized water as the blank.



5. Select the appropriate analysis volume of the digested sample given in Table 3 on page 4. Pipet the analysis volume from the sample and the blank into separate 25-mL mixing graduated cylinders.

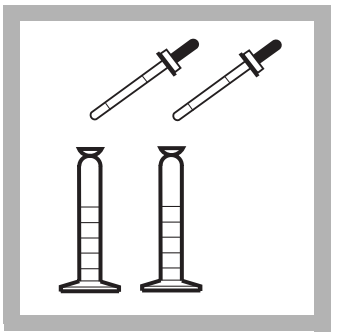


6. Add one drop of TKN Indicator to each cylinder.

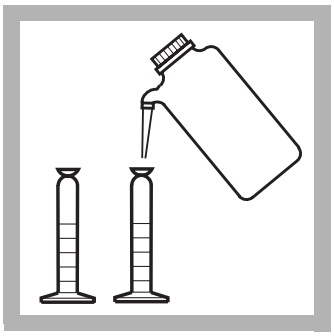


7. If the aliquot is less than 1 mL, proceed to step 8.

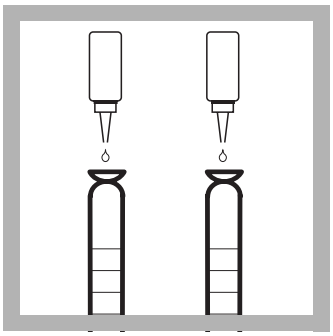
If it is greater than 1 mL, add drops of 8.0 N KOH to each cylinder until the first flash of blue color appears. Stopper and invert the cylinder after each addition. Proceed to the next step.



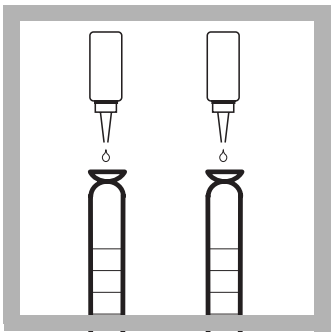
8. Add 1.0 N KOH to each cylinder, one drop at a time, mixing after each addition. Continue until the first permanent blue color appears.



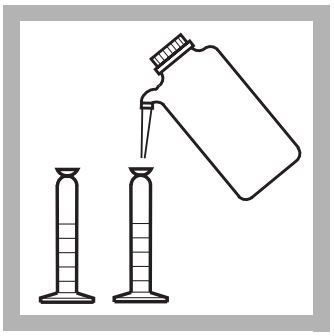
9. Fill both cylinders to the 20-mL mark with deionized water.



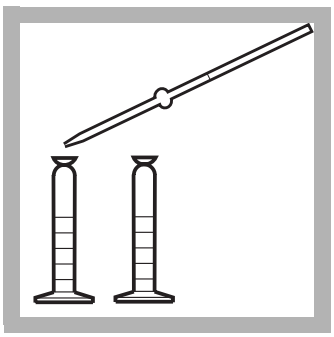
10. Add three drops of Mineral Stabilizer to each cylinder. Stopper and invert several times to mix.



11. Add three drops of Polyvinyl Alcohol Dispersing Agent to each cylinder. Stopper and invert several times to mix.



12. Fill both cylinders to the 25-mL mark with deionized water. Stopper and invert several times to mix.

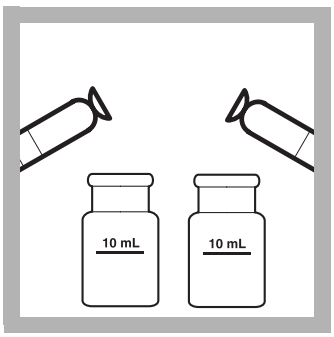


13. Pipet 1.00 mL of Nesslers Reagent to each cylinder. Stopper and invert repeatedly.

The solution should not be hazy. Any haze (turbidity) will cause incorrect results.

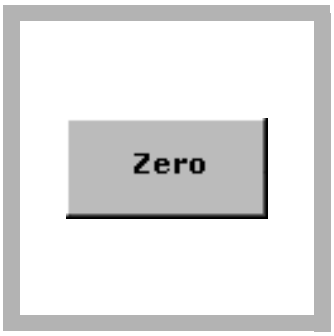
14. Press **TIMER>OK**.

A two-minute reaction period will begin.

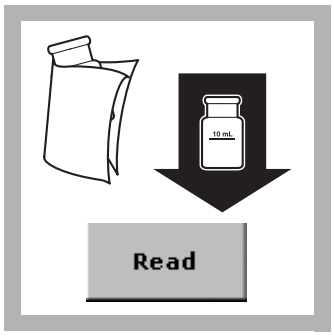


15. When the timer expires, pour the contents of each cylinder into separate square sample cells.

16. Wipe the blank and insert it into the cell holder with the fill line facing right.

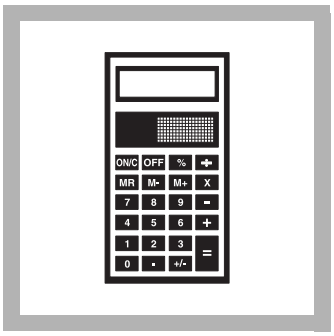


17. Press **ZERO**. The display will show:
0 mg/L TKN



18. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L TKN.



19. Calculate sample TKN as follows:

$$\text{ppm TKN} = \frac{75 \times A}{B \times C}$$

Where:

A = mg/L read from the display
B = g (or mL of water) sample taken for digest
C = mL analysis volume of digested sample

Interferences

Table 1 AQUEOUS SAMPLES (Solutions of suspensions in water—less than 1% solids)

Expected Nitrogen Concentration (mg/L)	Analysis Volume (mL)
0.5–28	10.0
2–112	5.0
11–560	2.00
45–2250	1.00
425–22500	0.50

Table 2 DRY SAMPLES

Expected Nitrogen Concentration (mg/L)	Analysis Volume (mL)
42–2200	10.0
106–5600	5.00
350–18,000	2.00
1000–56,000	1.00
4200–220,000	0.50

Table 3 OILS AND FATS

Expected Nitrogen Concentration (mg/L)	Analysis Volume (mL)
85–4500	10.0
210–11,000	5.00
2100–110,000	1.00

Sample Collection, Storage, and Preservation

Collect samples in clean glass or plastic containers. Adjust the pH to 2 or less with Sulfuric Acid (about 2 mL per liter) and cool to 4 °C (39 °F). Preserved samples can be stored up to 28 days.

Accuracy Check

Kjeldahl Nitrogen Standard Method
This procedure checks digestion efficiency and indicates that amount of bound nitrogen that is freed during digestion. The methods and standards available to check digestion technique are found in the *Accuracy Check* section following the procedure in the *Digesdahl® Digestion Apparatus Instruction Manual*. Using the digested Kjeldahl standard, perform the above TKN analysis on the colorimeter. The TKN value should come within ± 3% of the value of the prepared Kjeldahl standard.

Standard Solution Method (to check calibration accuracy only)

1. Add one drop of TKN Indicator to each 25-mL graduated mixing cylinder.
2. Fill one cylinder to the 20-mL mark with deionized water. Fill the other cylinder to the 20-mL mark with a 1.0-mg/L NH_3–N solution.
3. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix.
4. Add 3 drops of Polyvinyl Alcohol Dispersing agent to each cylinder.
5. Perform the TKN procedure as described in step 12 to step18. The display should show 26–27 mg/L TKN.

Summary of Method

The term *Total Kjeldahl Nitrogen* refers to the combination of ammonia and organic nitrogen. However, only the organic nitrogen compounds appearing as organically bound nitrogen in the trinegative state are determined in this test. Nitrogen in this form is converted into ammonium salts by the action of sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a modified Nessler method test. Test results are measured at 460 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Nitrogen Reagent Set, 0–150 mg/L, Nesslers Method, includes:	—	250 tests	24953-00
Hydrogen Peroxide, 50%	20 mL	490 mL	21196-49
Mineral Stabilizer	6 drops	50 mL SCDB	23766-26
Nesslers Reagent	2 mL	500 mL	21194-49
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	23765-26
Potassium Hydroxide Standard Solution, 1.0 N	varies	50 mL SCDB	23144-26
Potassium Hydroxide Standard Solution, 8.0 N	varies	100 mL MDB	282-32H
Sulfuric Acid, ACS, concentrated	6 mL	500 mL	979-49
TKN Indicator Solution	2 drops	50 mL SCDB	22519-26

Required Apparatus

Description	Quantity/Test	Unit	Cat. No,
Boiling Chips, silicon carbide	2–3	500 g	20557-34
Cots, finger	2	2/pkg	14647-02
Cylinder, graduated mixing, 25-mL	2	each	26362-40
Digesdahl® Digestion Apparatus, 115 VAC	1	each	23130-20
OR			
Digesdahl® Digestion Apparatus, 220 VAC	1	each	23130-21
Pipet, TenSette®, 0.1–1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg	21856-96
Safety Shield	1	each	50030-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Nitrogen, Primary Standard Set for TKN	each	22778-00
Nitrogen Standard Solution, 1-mg/L NH_3–N	500 mL	1891-49
Nitrogen Standard Solution, Voluette® Ampule, 150-mg/L NH_3–N, 10-mL	16/pkg	21284-10
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Pour-Thru Cell Kit	each	59404-00
Sodium Thiosulfate, Pentahydrate	—	460-01

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Updated November 2005 Edition 1

Organic Carbon, Total

Method 10129

Direct Method[1]

LR (0.3 to 20.0 mg/L C)

Scope and Application: For water, drinking water, and wastewater

[1] U.S. Patent 6,368,870

Test Preparation

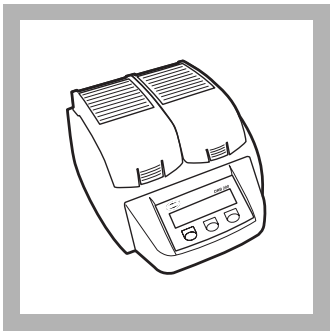
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
A reagent blank is required for each series of samples.
To test for higher ranges of TOC use Method 100173 or method 10128.

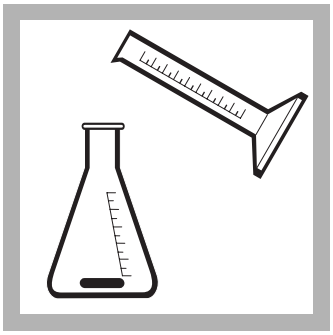
Collect the following items:	**Quantity**
Total Organic Carbon Direct Method Low Range Test 'N Tube™ Reagent Set	1
Cylinder, graduated, 10-mL	1
DRB200 Reactor	1
Light Shield	1
pH Paper	1
Flask, Erlenmeyer, 50-mL	1
Magnetic Stirrer	1
Pipet, TenSette®, 0.1 to 1.0 mL plus tips	1
Pipet, TenSette, 1.0 to 10.0 mL plus tips	1
Stir Bar, magnetic	1
Test Tube Rack	1–3
Water, organic-free	3.0 mL
Wipes, disposable, Kimwipes®	1

Note: *Reorder information for consumables and replacement items is on page 6.*

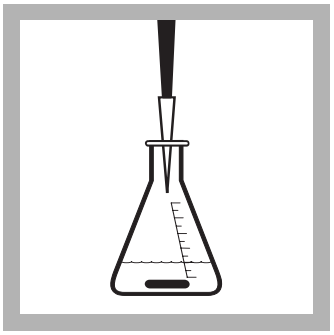
Direct Method — Method 10129



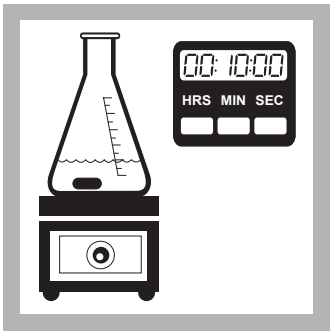
1. Turn on the DRB 200 reactor. Select the TOC program.



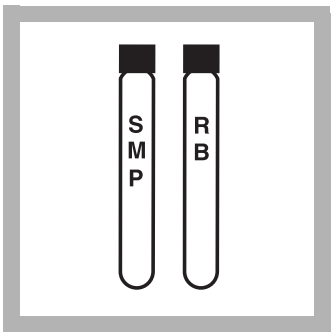
2. Use a graduated cylinder to add 10 mL of sample to a 50-mL Erlenmeyer flask that contains a stir bar.



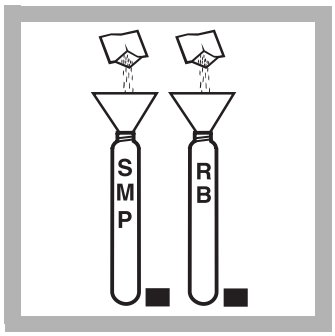
3. Add 0.4 mL of Buffer Solution, pH 2.0. Use pH paper to make sure the sample pH is 2.



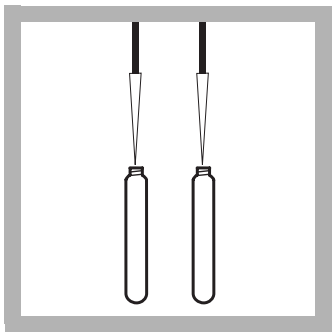
4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.



5. Label two Low Range Acid Digestion vials *sample* and *reagent blank*.



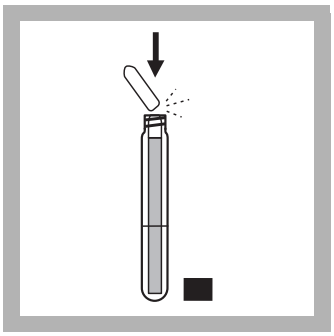
6. Use a funnel to add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).



7. Use a TenSette® Pipet to add 3.0 mL of organic-free water to the reagent blank vial and 3.0 mL of prepared sample to the sample vial. Swirl to mix.

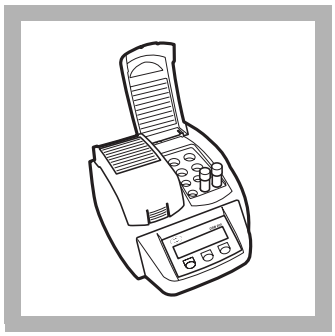
8. Rinse two blue Low Range Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

Do not touch the ampules sides after wiping. Pick them up by the top.

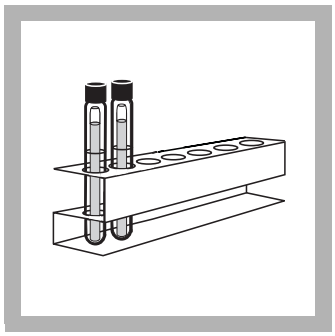


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Do not invert or tilt the vial after inserting the ampule.



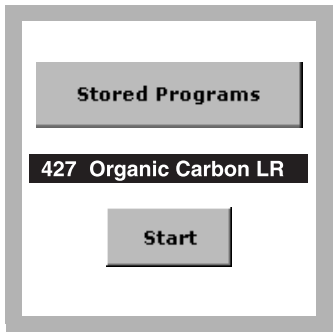
10. Cap the vial assemblies tightly and insert them in the COD reactor for 2 hours at 103–105 °C.



11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

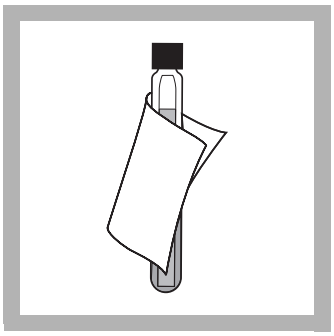
Allow the vials to cool for one hour for accurate results.

The liquid in the reagent blank vial should be dark blue.

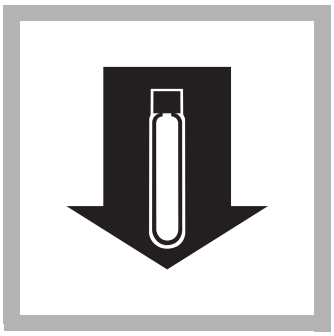


12. Select the test.

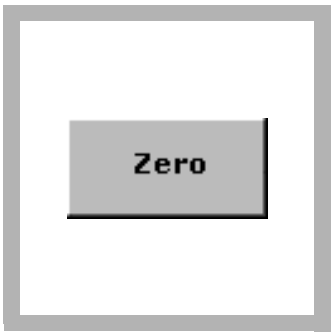
Install the Light Shield in Cell Compartment #2.



13. Wipe the reagent blank with a damp towel, followed by a dry one, to remove fingerprints or other marks.



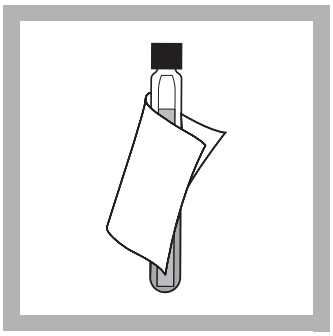
14. Insert the reagent blank vial assembly in the 16-mm round cell holder.



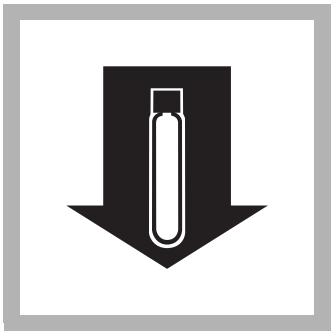
15. Press **ZERO**.

The display will show:

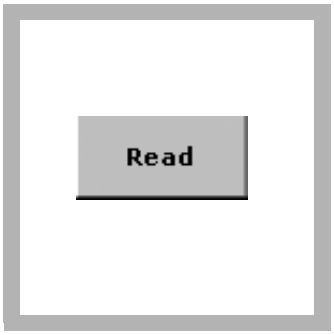
0.0 mg/L C.



16. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.



17. Insert the sample vial assembly in the 16-mm round cell holder.



18. Press **READ**.

Results are in mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Interfering Substances and Levels

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	500 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L

Table 1 Interfering Substances and Levels (continued)

Substance	Maximum Level Tested
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 600 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding Sulfuric Acid Solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle completely full before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare a 150-g/L C standard by transferring 15.00mL of 1000-mg/L C stock solution to a 100-mL Class A volumetric flask. Dilute to volume with organic-free water. Mix.

5. Use the TenSette® Pipet to add 0.1, 0.2, and 0.3 mL of the 150-mg/L C standard to each of three Acid Digestion Vials.

6. Add the contents of one TOC Persulfate Powder Pillow to each vial.

7. Add 3.0 mL of sample to each vial. Swirl to mix. Continue the test starting at step 8 on page 2 of this procedure.

8. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

9. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solutions Method

1. Prepare a 1000-mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

   Alternatively, open one ampule of TOC Standard Solution, 1000 mg/L C. Perform the TOC procedure as described above.

2. Prepare a 10.0-mg/L C standard by transferring 10.00 mL of the stock standard to a 1000-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample. Test results are measured at 598 and 430 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Reagent Set, Total Organic Carbon Direct Method Low Range Test 'N Tube™, includes:	—	50 vials	27603-45
Acid Digestion Solution Vials, High Range TOC[1]	1	50/pkg	—
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampule, Low Range TOC[1]	1	10/pkg	—
TOC Persulfate Powder Pillows[1]	1	50/pkg	—
pH Paper	1	5/pkg	391-33
Water, Organic-free	3.0 mL	500 mL	26415-49

[1] Not sold separately.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 10-mL	1	each	508-38
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Flask, Erlenmeyer, 50-mL	1	each	505-41
Light Shield	1	each	LZV646
Magnetic Stirrer	1	each	28812-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg	21856-96
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	2	50/pkg	21997-96
Stir Bar, magnetic	1	each	45315-00
Test Tube Rack	1–3	each	18641-00
Wipes, Disposable, Kimwipes®	1	280/pkg	20970-00

Recommended Standards

Description	Unit	Cat. No.
Potassium Acid Phthalate	500 g	315-34
TOC Standard Solution (KHP Standard, 1000-mg/L C)	5/pkg	27915-05

Optional Reagents

Description	Cat. No.
Sulfuric Acid Solution	2449-32

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

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Updated November 2005 Edition 1

Organic Carbon, Total

Method 10173

Direct Method[1]

MR (15 to 150 mg/L C)

Scope and Application: For wastewater and industrial waters

[1] U.S. Patent 6,368,870

Test Preparation

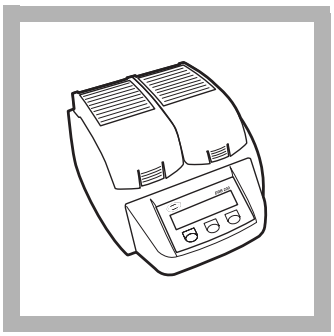
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
A reagent blank is required for each series of samples.
To test for TOC levels below 15 mg/L C, use Method 10129; to test for TOC levels above 150 mg/L C, use Method 10128.

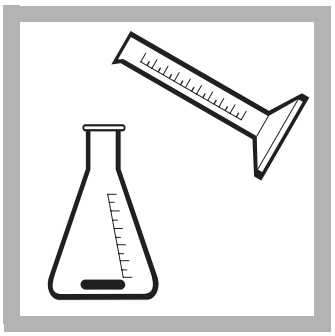
Collect the following items:	**Quantity**
Total Organic Carbon Direct Method Mid Range Test 'N Tube™ Reagent Set	1
Cylinder, graduated, 10-mL	1
DRB200 Reactor	1
Flask, Erlenmeyer, 50-mL	1
Light Shield	1
Magnetic Stirrer	1
pH Paper	1
Pipet, TenSette®, 0.1 to 1.0 mL plus tips	1
Pipet, TenSette, 1.0 to 10.0 mL plus tips	1
Stir Bar, magnetic	1
Test Tube Rack	1–3
Water, organic-free	3.0 mL
Wipes, disposable, Kimwipes®	1

Note: *Reorder information for consumables and replacement items is on page 6.*

Direct Method — Method 10173

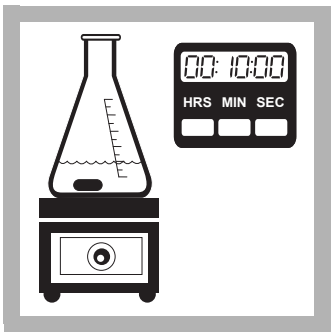


1. Turn on the DRB 200 reactor. Select the TOC program.

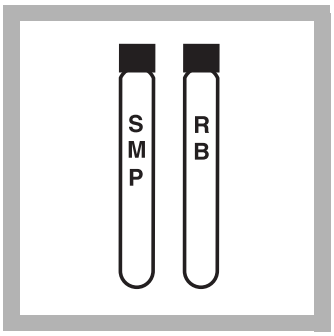


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL Erlenmeyer flask that contains a stir bar.

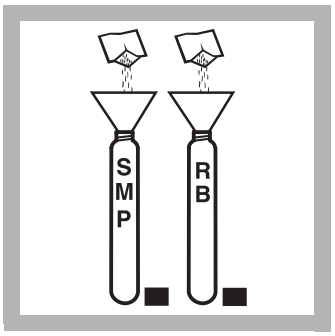
3. Add 0.4 mL of Buffer Solution, pH 2.0. Use pH paper to make sure the sample pH is 2.



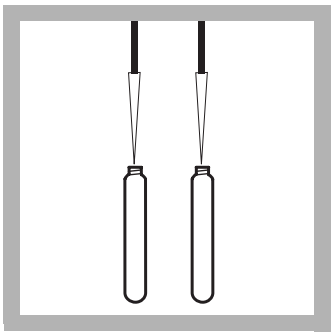
4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.



5. Label two Mid Range Acid Digestion vials *sample* and *reagent blank*.



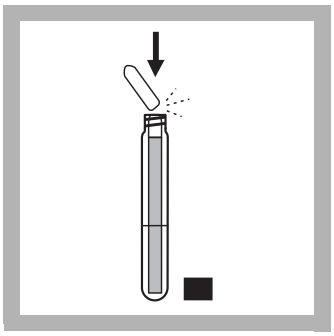
6. Use a funnel to add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).



7. Use a TenSette® Pipet to add 1.0 mL of organic-free water to the reagent blank vial and 1.0 mL of prepared sample to the sample vial. Swirl to mix.

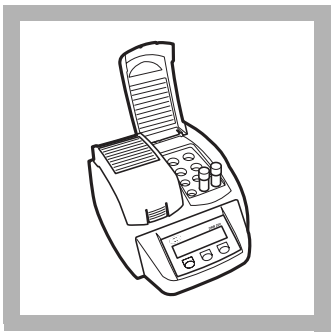
8. Rinse two blue MR/HR Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

Do not touch the ampules sides after wiping. Pick them up by the top.

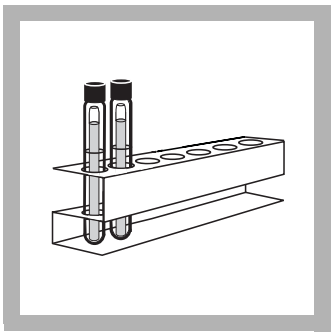


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Do not invert or tilt the vial after inserting the ampule.



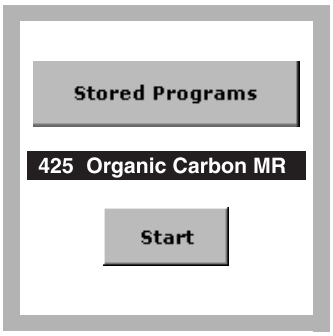
10. Cap the vial assemblies tightly and insert them in the DRB 200 reactor for 2 hours at 103–105 °C.



11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for one hour for accurate results.

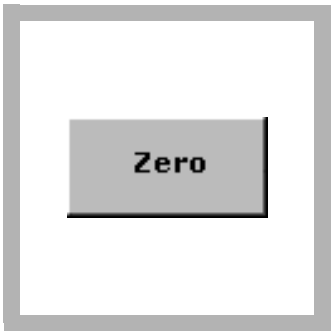
The liquid in the blank vial should be dark blue.



12. Select the test.

Install the Light Shield in Cell Compartment #2.

13. Wipe the reagent blank vial assembly and insert it into the 16-mm round cell holder.



14. Press **ZERO**.

The display will show:

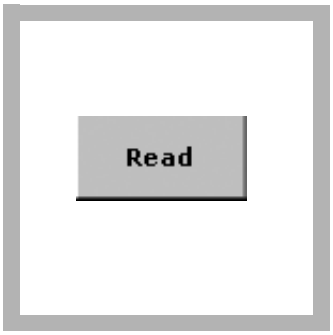
0 mg/L C

then Underrange until the sample is read.

15. Wipe the reagent vial assembly and insert it into the 16-mm round cell holder.

Results are in mg/L C.

The display will show Underrange when the mg/L C value is below the lower limit of the method.



16. Press **READ**.

Results are in mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Interfering Substances and Levels

Substance	Maximum Level Tested
Aluminum	10 mg/L

Table 1 Interfering Substances and Levels (continued)

Substance	Maximum Level Tested
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	1500 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 1000 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding Sulfuric Acid Solution*.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle completely full before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

* See Optional Reagents on page 6.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare a 300-mg/L C standard by transferring 15.00mL of a 1000-mg/L organic carbon to a 50 mL Class A volumetric flask. Dilute to volume using Organic-free Reagent Water. Stopper and mix thoroughly.

5. Use the TenSette® Pipet to add 0.1, 0.2, and 0.3 mL of the 300-mg/L C standard to each of three Acid digestion vials.

6. Add the contents of one TOC Persulfate Powder Pillow to each vial.

7. Add 1.0 mL sample to each vial. Swirl to mix. Proceed with the procedure starting at step 8 on page 2.

8. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

9. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solutions Method

1. Prepare a 1000-mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

   Alternatively, open one ampule of TOC Standard Solution, 1000 mg/L C. Perform the TOC procedure as described above.

2. Prepare a 100-mg/L C standard by transferring 5.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution

which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample. Test results are measured at 598 and 430 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Reagent Set, Total Organic Carbon Direct Method Mid Range Test 'N Tube™, includes:	—	50 vials	28159-45
Acid Digestion Solution Vials, High Range TOC[1]	1	50/pkg	—
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, MR/HR TOC[1]	1	10/pkg	—
TOC Persulfate Powder Pillows[1]	1	50/pkg	—
pH Paper	1	5/pkg	391-33
Water, Organic-free	3.0 mL	500 mL	26415-49

[1] Not sold separately.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 10-mL	1	each	508-38
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Flask, Erlenmeyer, 50-mL	1	each	505-41
Light Shield	1	each	LZV646
Magnetic Stirrer	1	each	28812-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg	21856-96
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	2	50/pkg	21997-96
Stir Bar, magnetic	1	each	45315-00
Test Tube Rack	1–3	each	18641-00
Wipes, disposable, Kimwipes®	1	280/pkg	20970-00

Recommended Standards

Description	Unit	Cat. No.
Potassium Acid Phthalate	500 g	315-34
TOC Standard Solution (KHP Standard, 1000-mg/L C)	5/pkg	27915-05

Optional Reagents

Description	Cat. No.
Sulfuric Acid Solution	2449-32

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Updated November 2005 Edition 1

Organic Carbon, Total

Method 10128

Direct Method[1]

HR (100 to 700 mg/L C)

Scope and Application: For wastewater and industrial waters

[1] U.S. Patent 6,368,870

Test Preparation

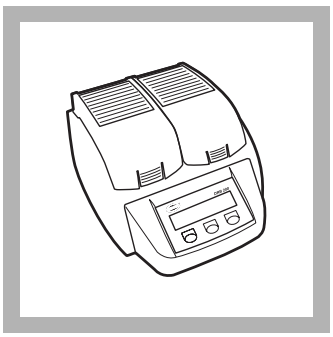
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
A reagent blank is required for each series of samples.
To test for TOC levels below 100 mg/L C, use Method 10173.

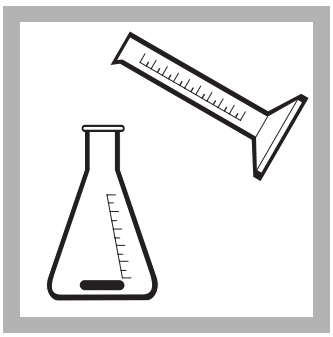
Collect the following items:	**Quantity**
Total Organic Carbon Direct Method High Range Test 'N Tube™ Reagent Set	1
Cylinder, graduated, 10-mL	1
DRB200 Reactor	1
Flask, Erlenmeyer, 50-mL	1
Light Shield	1
Magnetic Stirrer	1
Pipet, TenSette®, 0.1 to 1.0 mL plus tips	1
Pipet, TenSette, 1.0 to 10.0 mL plus tips	1
Stir Bar, magnetic	1
Test Tube Rack	1–3
Water, organic-free	3.0 mL
Wipes, disposable, Kimwipes®	1

Note: *Reorder information for consumables and replacement items is on page 6.*

Direct Method — Method 10128

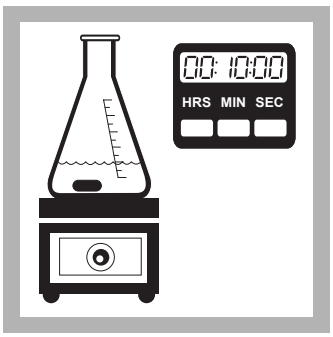


1. Turn on the DRB200 Reactor. Select the TOC program.

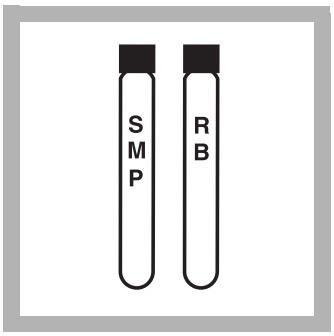


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL Erlenmeyer flask that contains a stir bar.

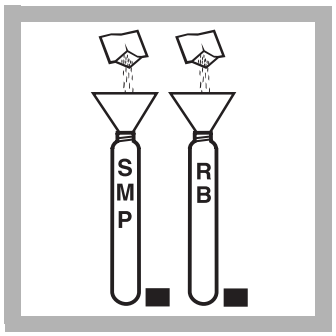
3. Add 0.4 mL of Buffer Solution, pH 2.0. Use pH paper to make sure the sample pH is 2.



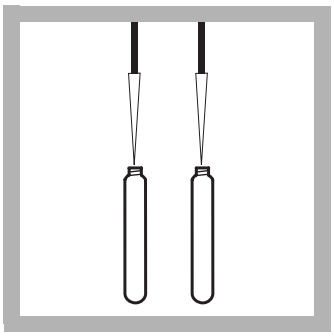
4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.



5. Label two High Range Acid Digestion vials *sample* and *reagent blank*.



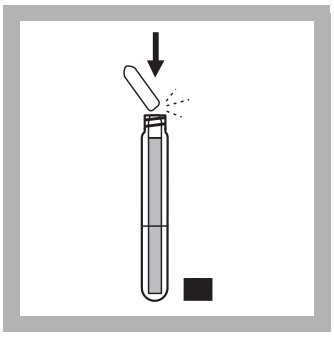
6. Use a funnel to add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).



7. Use a TenSette® Pipet to add 0.3 mL of organic-free water to the reagent blank vial and 0.3 mL of prepared sample to the sample vial. Swirl to mix.

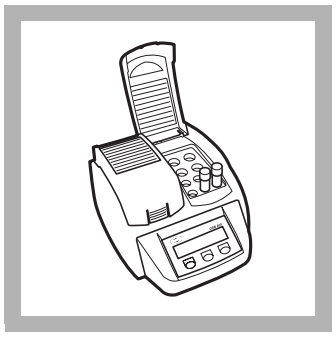
8. Rinse two blue MR/HR Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

Do not touch the ampules sides after wiping. Pick them up by the top.

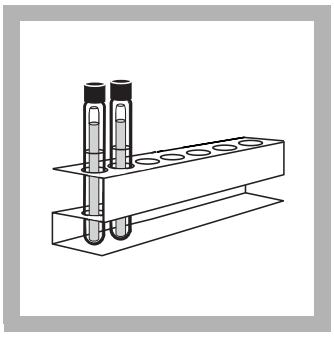


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Do not invert or tilt the vial after inserting the ampule.



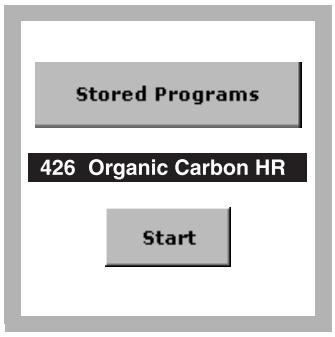
10. Cap the vial assemblies tightly and insert them in the DRB 200 reactor for 2 hours at 103–105 °C.



11. Carefully remove the vial assemblies from the reactor. Insert them in a test tube rack.

Allow the vials to cool for one hour for accurate results.

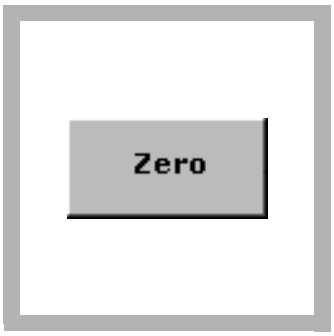
The liquid in the reagent blank vial should be dark blue.



12. Select the test.

Install the Light Shield in Cell Compartment #2.

13. Wipe the reagent blank vial assembly and insert it into the 16-mm round cell holder.

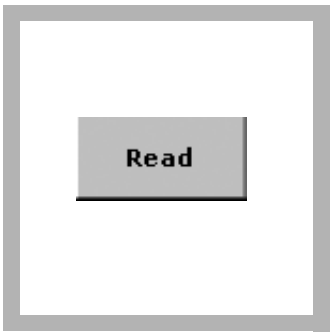


14. Press **ZERO**.

The display will show: 0 mg/L C.

"Underrange" will be displayed until the sample is read.

15. Wipe the reagent vial assembly and insert it into the 16-mm round cell holder.



16. Press **READ**.

Results are in mg/L C.

The display will show Underrange when the mg/L C value is below the lower limit of the method.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Interfering Substances and Levels

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	5000 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}

Table 1 Interfering Substances and Levels (continued)

Substance	Maximum Level Tested
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 1000 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding Sulfuric Acid Solution*.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 900 NTU have been tested without interference.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle completely full before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Prepare a 300-mg/L C standard as described in Standard Solutions Method, below.
5. Use the TenSette pipet to add 0.1, 0.2, and 0.3 mL of the 300-mg/L C standard to each of three Acid Digestion vials.
6. Add the contents of one TOC Persulfate powder pillow to each vial.
7. Add 0.3 mL sample to each vial. Swirl to mix. Proceed with the procedure starting at step 8 on page 2.
8. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
9. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents on page 6.

Standard Solutions Method

1. Prepare a 1000-mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

   Alternatively, open one ampule of TOC Standard Solution, 1000 mg/L C*.

2. Prepare a 300-mg/L C standard by transferring 15.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily. Perform the TOC procedure as described above.

3. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

4. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample. Test results are measured at 598 and 430 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Total Organic Carbon Direct Method High Range Test 'N Tube™ Reagent Set, includes:	—	50 vials	27604-45
Acid Digestion Solution Vials, High Range TOC[1]	1	50/pkg	—
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, MR/HR TOC[1]	1	10/pkg	—
TOC Persulfate Powder Pillows[1]	1	50/pkg	—
pH Paper	1	5/pkg	391-33
Water, organic-free	3.0 mL	500 mL	26415-49

[1] Not sold separately.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 10-mL	1	each	508-38
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Flask, Erlenmeyer, 50-mL	1	each	505-41
Light Shield	1	each	LZV646
Magnetic Stirrer	1	each	28812-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	2	50/pkg	21856-96
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	2	50/pkg	21997-96
Stir Bar, magnetic	1	each	45315-00
Test Tube Rack	1–3	each	18641-00
Wipes, disposable, Kimwipes®	1	280/pkg	20970-00

Recommended Standards

Description	Units	Cat. No.
Potassium Acid Phthalate	500 g	315-34
TOC Standard Solution (KHP Standard, 1000-mg/L C)	5/pkg	27915-05

Optional Reagents

Description	Cat. No.
Sulfuric Acid Solution	2449-32

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Updated November 2005 Edition 1

Oxygen, Dissolved

★Method 8166 **HRDO Method**

AccuVac® Ampuls **HR (0.3 to 15.0 mg/L O_2)**

Scope and Application: For water and wastewater

Test Preparation

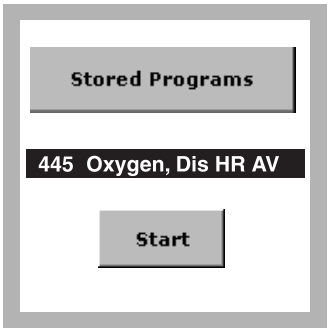
Before starting the test:

Analyze samples on-site. Do not store for later analysis

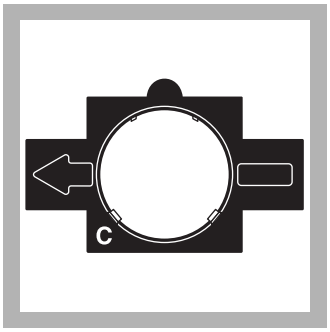
Collect the following items:	**Quantity**
High Range Dissolved Oxygen AccuVac® Ampuls with reusable Ampul caps	1
Polypropylene Beaker, 50-mL	1
Sample Cell, 10-mL	1

Note: *Reorder information for consumables and replacement items is on page 4.*

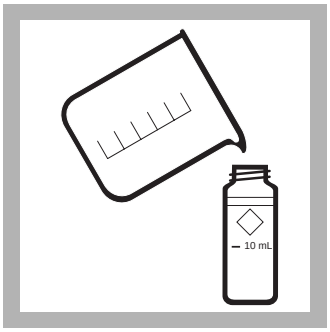
AccuVac Ampul® Method 8166



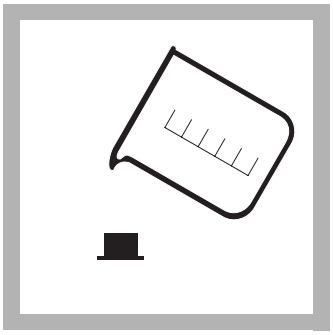
1. Select the test.



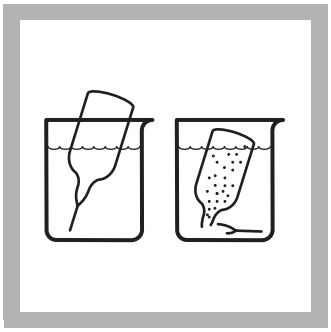
2. Insert Adapter C.



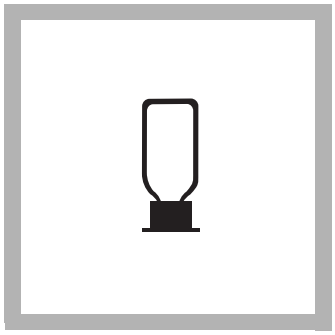
3. Blank Preparation: Fill a round sample cell with 10 mL of sample.



4. Fill a blue Ampul cap with sample.

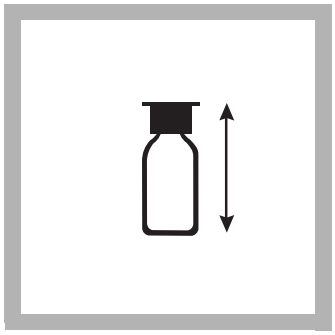


5. Prepared Sample: Fill a High Range Dissolved Oxygen AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.



6. Hold the Ampul with the tip pointing down and immediately insert the Ampul into the Ampul cap.

The cap prevents contamination from atmospheric oxygen.

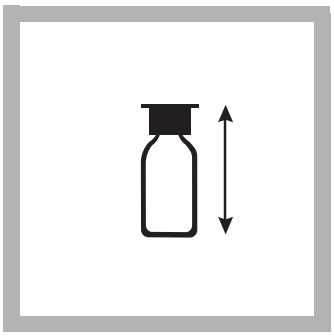


7. Shake the Ampul for 30 seconds.

A small amount of undissolved reagent will not affect results.

8. Press **TIMER>OK**.

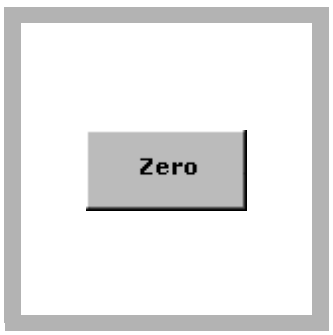
A two-minute reaction period will begin. This enables the oxygen that was degassed during aspiration to redissolve and react.



9. When the timer expires, shake the Ampul for 30 seconds.

Allow any bubbles to dissipate before proceeding.

10. Insert the blank in the cell holder.



11. Press **ZERO**.

The display will show:

0.0 mg/L O_2

12. Insert the prepared sample into the cell holder.

Press **READ**. Results will appear in mg/L O_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Cr^{3+}	Greater than 10 mg/L
Cu^{2+}	Greater than 10 mg/L
Fe^{2+}	Greater than 10 mg/L
Mg^{2+}	Magnesium is commonly present in seawater and causes a negative interference. If the sample contains more than 50% seawater, the oxygen concentration obtained by this method will be 25% less than the true oxygen concentration. If the sample contains less than 50% seawater, the interference will be less than 5%.
Mn^{2+}	Greater than 10 mg/L
Ni^{2+}	Greater than 10 mg/L
NO^{2-}	Greater than 10 mg/L

Sample Collection, Preservation, and Storage

The main consideration in sampling with the High Range Dissolved Oxygen Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen between breaking open the Ampul and reading the absorbance. This is accomplished by capping the Ampul with an Ampul cap. If the Ampul is securely capped, the Ampul should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards.

Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested may change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time, and other factors. A single dissolved oxygen test rarely reflects the accurate overall condition of a body of water. Several samples taken at different times, locations, and depths are recommended for most reliable results. Samples must be tested immediately upon collection, although only a small error results if the absorbance reading is taken several hours later.

Accuracy Check

The results of this procedure may be compared with the results of a titrimetric procedure (request Lit. Code 8042), or by using any of the following dissolved oxygen meters: sens***ion***™6 Dissolved Oxygen Meter*, HQ10 Portable LDO Dissolved Oxygen Meter*, or HQ20 Portable LDO Dissolved Oxygen/pH Meter*.

Summary of Method

The High Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum-sealed in a 14-mL Ampul. When the AccuVac Ampul is opened in a sample containing dissolved oxygen, it forms a yellow color which turns purple. The purple color development is proportional to the concentration of dissolved oxygen. Test results are measured at 535 nm.

* See Optional Reagents and Apparatus on page 4.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
High Range Dissolved Oxygen AccuVac® Ampuls with 2 reusable Ampul caps	1	25/pkg	25150-25

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Polypropylene Beaker, 50-mL	1	each	1080-41
Sample Cell, 10-mL, with cap	1	each	21228-00

Optional Reagents and Apparatus

Description	Cat. No.
HQ10 Portable LDO Dissolved Oxygen Meter	51815-00
HQ20 Portable LDO Dissolved Oxygen/pH Meter	51825-00
sens***ion***™6 Dissolved Oxygen Meter	51850-01

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Updated November 2005 Edition 1

Oxygen, Dissolved

Method 8333

AccuVac® Ampul

Ultra High Range Method

UHR (1.0 to 40.0 mg/L O_2)

Scope and Application: For aquaculture

Test Preparation

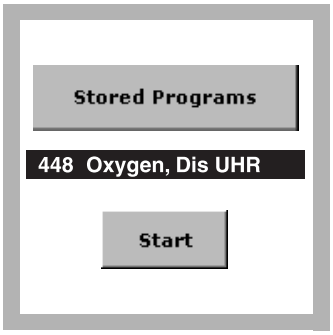
Before starting the test:

Analyze samples on-site. Do not store for later analysis.

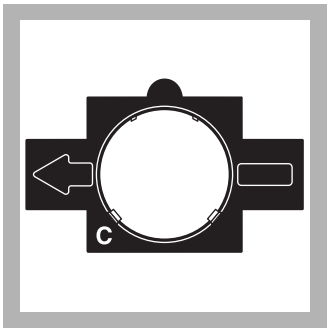
Collect the following items:	**Quantity**
High Range Dissolved Oxygen AccuVac®Ampuls, with reusable Ampul caps	1
Polypropylene Beaker, 50-mL	1
Sample Cell, 10-mL	1

Note: *Reorder information for consumables and replacement items is on page 3.*

AccuVac Ampul® Method 8333

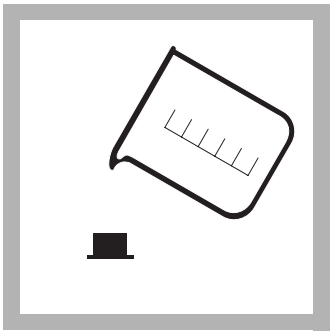


1. Select the test.

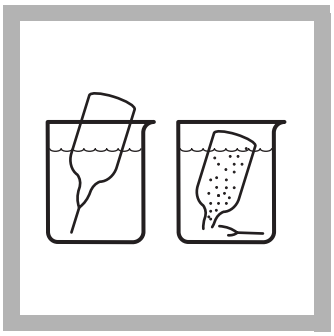


2. Insert Adapter C.

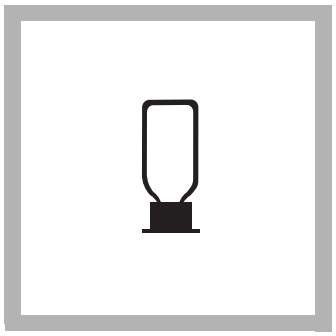
3. Blank Preparation: Fill a round sample cell with 10 mL of sample.



4. Fill a blue Ampul cap with sample.

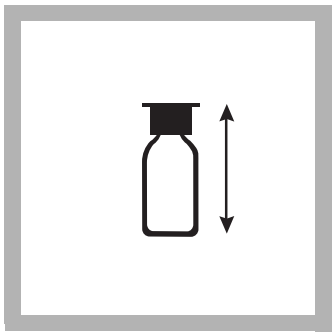


5. Prepared Sample: Fill a High Range Dissolved Oxygen AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.



6. Hold the Ampul with the tip pointing down and immediately insert the Ampul into the Ampul cap.

The cap prevents contamination from atmospheric oxygen.

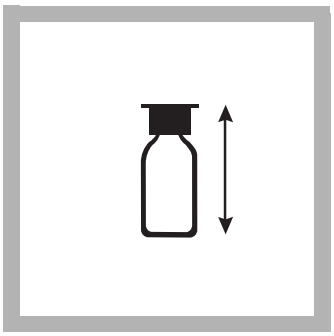


7. Shake the Ampul for 30 seconds.

A small amount of undissolved reagent will not affect results.

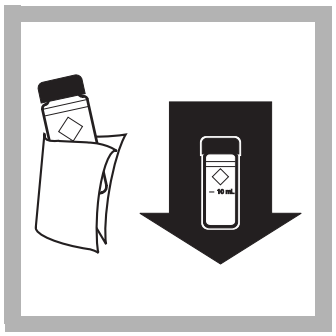
8. Touch **TIMER>OK**.

A two-minute reaction period will begin. This enables the oxygen that was degassed during aspiration to redissolve and react.

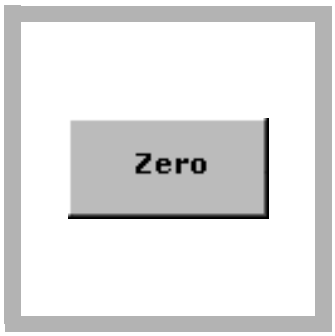


9. When the timer expires, shake the Ampul for 30 seconds.

Allow any bubbles to dissipate before proceeding.



10. Insert the blank into the cell holder.



11. Press **ZERO**.

The display will show: 0.0 mg/L O_2

12. Insert the prepared sample into the cell holder.

Press **READ**. Results are in mg/L O_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Cr^{3+}	Greater than 10 mg/L
Cu^{2+}	Greater than 10 mg/L
Fe^{2+}	Greater than 10 mg/L
Mg^{2+}	Magnesium is commonly present in seawater and interferes. If the sample contains more than 50% seawater, the oxygen concentration observed will be 25% lower than the true oxygen concentration. If the sample contains less than 50% seawater, the interference will be less than 5%.
Mn^{2+}	Greater than 10 mg/L
Ni^{2+}	Greater than 10 mg/L
NO^{2-}	Greater than 10 mg/L

Sample Collection, Preservation, and Storage

The main consideration in sampling with the High Range Dissolved Oxygen AccuVac Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen between breaking open the Ampul and reading the absorbance. This is accomplished by capping the Ampul with an Ampul cap. If the Ampul is securely capped, the Ampul should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards.

Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested may change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time, and other factors. A single dissolved oxygen test rarely reflects the accurate over-all condition of a body of water. Several samples taken at different times, locations, and depths are recommended for most reliable results. Samples must be tested immediately upon collection although only a small error results if the absorbance reading is taken several hours later.

Accuracy Check

The results of this procedure may be compared with the results of a titrimetric procedure (request Lit. Code 8042), or by using any of the following dissolved oxygen meters: sens***ion***™ 6 Dissolved Oxygen Meter*, HQ10 Portable LDO Dissolved Oxygen Meter*, or HQ20 Portable LDO Dissolved Oxygen/pH Meter*.

Summary of Method

The High Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum sealed in a 14-mL Ampul. When the AccuVac Ampul is broken open in a sample containing dissolved oxygen, it forms a yellow color which turns purple. The purple color development is proportional to the concentration of dissolved oxygen. Test results are measured at 680 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
High Range Dissolved Oxygen AccuVac® Ampuls, with 2 reusable Ampul caps	1	25/pkg	25150-25

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Polypropylene Beaker, 50-mL	1	each	1080-41
Sample Cell, 10-mL, with cap	1	each	21228-00

Optional Reagents and Apparatus

Description	Cat. No.
HQ10 Portable LDO Dissolved Oxygen Meter	51815-00
HQ20 Portable LDO Dissolved Oxygen/pH Meter	51825-00
sens***ion***™ 6 Dissolved Oxygen Meter	51850-01

*See Optional Reagents and Apparatus on page 3.

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Updated November 2005 Edition 1

Oxygen, Dissolved

Method 8316 **Indigo Carmine Method**

AccuVac® Ampuls **LR (6 to 800 µg/L O_2)**

Scope and Application: For boiler feedwater

Test Preparation

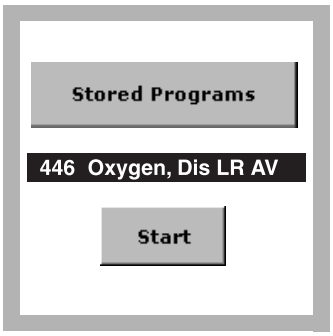
Before starting the test:

The Ampuls will contain a small piece of wire to maintain reagent quality. The solution will be yellow.

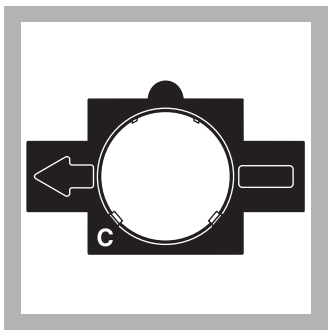
Collect the following items:	Quantity
Low Range Dissolved Oxygen AccuVac® Ampuls	1
Polypropylene Beaker, 50 mL	1
Sample Cell, 10-mL with cap	1

Note: *Reorder information for consumables and replacement items is on page 3.*

AccuVac® Ampul Method 8316



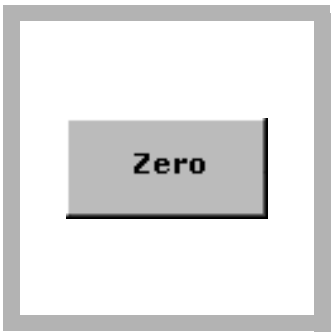
1. Select the test.



2. Insert Adapter C.

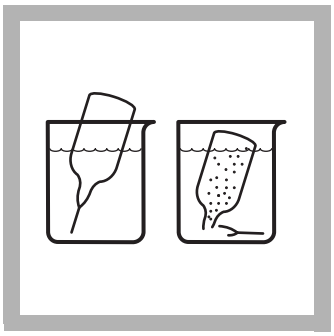
3. Blank Preparation: Fill a round sample cell with 10 mL of sample.

4. Insert the blank into the cell holder.



5. Press **ZERO**.

The display will show: 0 µg/L O_2



6. Fill a Low Range Dissolved Oxygen Ampul with sample. Keep the tip immersed while the Ampul fills completely.

7. Immediately insert the Ampul into the cell holder.

Press **READ**. Results are in µg/L O_2.

8. Use the initial reading. The reading is stable for 30 seconds. After 30 seconds the Ampul solution will absorb oxygen from the air.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Hydrazine	100,000 fold excess will begin to reduce the oxidized form of the indicator solution.
Sodium hydrosulfite	Reduces the oxidized form of the indicator solution and will cause a significant interference.

Excess amounts of thioglycolate, ascorbate, ascorbate + sulfite, ascorbate + cupric sulfate, nitrite, sulfite, thiosulfate, and hydroquinone will not reduce the oxidized form of the indicator and do not cause significant interference.

Sample Collection, Preservation, and Storage

The main consideration in this procedure is to prevent contaminating the sample with atmospheric oxygen. Sampling from a stream of water that is hard plumbed to the sample source is ideal. Use a funnel to maintain a continual flow of sample and yet collect enough sample to immerse the Ampul. It is important not to introduce air in place of the sample. Rubber tubing, if used, will introduce unacceptable amounts of oxygen into the sample unless the length of tubing is minimized and the flow rate is maximized. Flush the sampling system with sample for at least 5 minutes.

Accuracy Check

The results of this procedure may be compared using one of the following portable dissolved oxygen meters: HQ10*, HQ20*, or the sens***ion***™ 6*.

The reagent blank for this test can be checked by following these steps:

1. Fill a 50-mL beaker with sample and add approximately 50 mg sodium hydrosulfite.

2. Immerse the tip of a Low Range Dissolved Oxygen AccuVac Ampul in the sample into the tip. Aspirate the sample into the Ampul.

3. Determine the dissolved oxygen concentration according to the preceding procedure. The result should be 0 ± 6 µg/L.

* See Optional Apparatus on page 3.

Summary of Method

The Low Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum-sealed in a 14-mL Ampul. When the AccuVac Ampul is broken open in a sample containing dissolved oxygen, the yellow solution will turn blue. The blue color development is proportional to the concentration of dissolved oxygen. Test results are measured at 610 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Low Range Dissolved Oxygen AccuVac® Ampuls	1	25/pkg	25010-25

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, Polypropylene, 50 mL	1	each	1080-41
Sample Cells, 10 mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Sodium Hydrosulfite, technical grade	500 g	294-34

Optional Apparatus

Description	Cat. No.
HQ10 Portable LDO Meter	51815-00
HQ20 Portable LDO Meter	51825-00
sens***ion***™ 6	51850-01

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Updated November 2005 Edition 1

Oxygen Demand, Chemical

★Method 8000

Reactor Digestion Method[1]

TNTplus™

LR (TNT821, 3–150 COD); HR (TNT822, 20–1500mg/L COD)

Scope and Application: For water, wastewater; digestion is required;
3–150 mg/L and 20– 1500 mg/L COD ranges are USEPA approved for wastewater analyses[2]

[1] Jirka, A.M.; Carter, M.J., *Analytical Chemistry*, 1975, 47(8), 1397
[2] *Federal Register*, April 21, 1980, 45(78), 26811-26812

Test Preparation

Before starting the test:

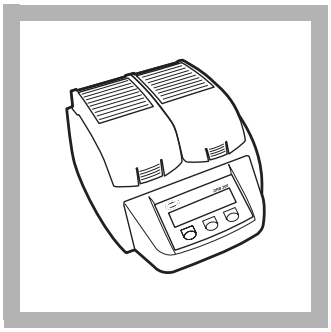
Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read *Safety Advice* and *Expiration Date* on package.
Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and associated MSDS sheets.
To run the optional blank for a set of samples, see Blanks for Colorimetric Determination on page 3.
Spilled reagent will affect test accuracy and is hazardous to skin and other materials.
Be prepared to wash spills with running water.
Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water. Review and follow instructions carefully.
Store unused (light-sensitive) vials in a closed box.

Collect the following items:	**Quantity**
Blender	1
DRB200 Reactor with 13-mm wells (use adapters with 16-mm holes)	1
COD TNTplus™ vials for the appropriate concentration range	varies
Light Shield	1
Pipettor for 2.0 mL Sample	1
Pipettor Tip	1
Test Tube Rack	2

Note: *Reorder information for consumables and replacement items is on page 5.*

TNT Plus Method 8000

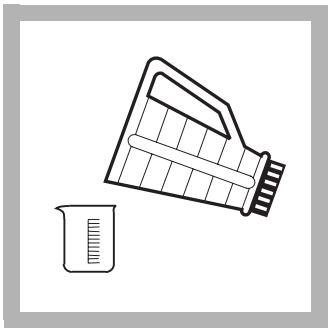
Important Note: *If the sample does not contain suspended solids, omit steps 1 and 3.*



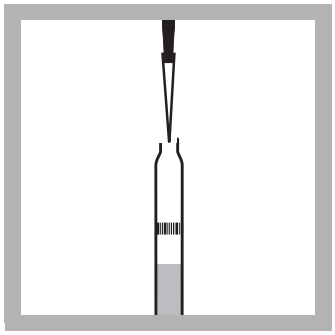
1. Turn on the DRB200 Reactor. Preheat to 150 °C.

Note: *For DRB200 Reactors with 16-mm wells, insert a 16-mm to 13-mm adapter sleeve into each well* ***before*** *turning on the reactor.*

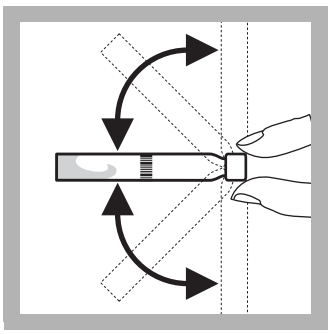
2. Homogenize 100 mL of sample for 30 seconds in a blender. For samples containing large amounts of solids, increase the homogenization time.



3. To help ensure that a representative portion of sample is analyzed, pour the homogenized sample into a 250-mL beaker and gently stir with a magnetic stir plate.

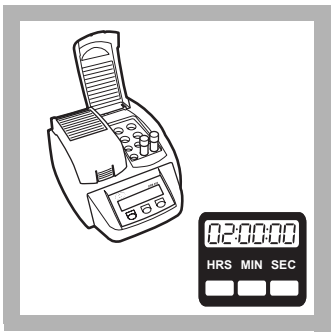


4. Carefully pipet 2.0 mL of sample into the vial. Cap and clean the outside of the vial.

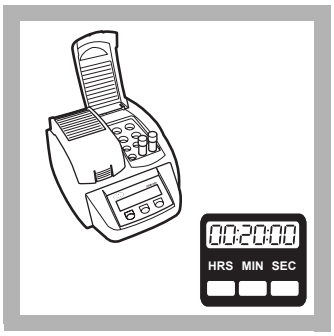


5. Hold the vial by the cap over a sink. Invert gently several times to mix. The sample vials will become very hot during mixing.

Place the vial in the preheated DRB200 Reactor. Close the protective lid

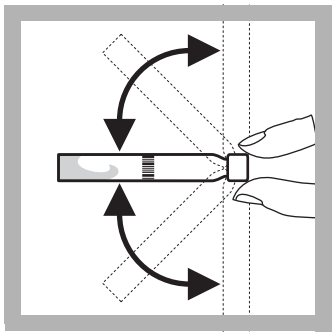


6. Heat for two hours.

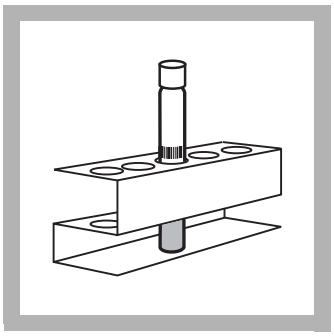


7. Turn the reactor off.

Wait about 20 minutes for the vial to cool to 120 °C or less.

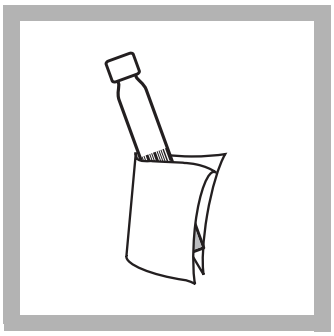


8. Invert the vial several times while still hot.

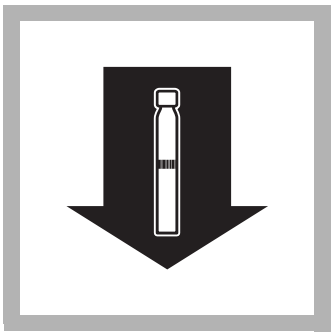


9. Place the vial into a rack to cool to room temperature.

Install the Light Shield in Cell Compartment #2.



10. Thoroughly clean the outside of the vial.



11. Insert the vial into the cell holder. Close the lid.

The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L COD.

Blanks for Colorimetric Determination

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark, and monitor decomposition by measuring its concentration periodically.

To subtract the value of the blank from a series of measurements, measure the blank per step 11. Press **OPTIONS>MORE>REAGENT BLANK**. Select **ON**.The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to 2000 mg/L Cl^-.

Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid* to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions.

* See Optional Reagents and Apparatus on page 5.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the 3 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2 mL as the sample volume. The result should be 100 mg/L COD. Or, dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to produce a 100-mg/L standard.

2. Check the accuracy of the 20 to 1,500 mg/L range by using either a 300 mg/L or 1000 mg/L COD Standard Solution. Use 2 mL of one of these solutions as the sample volume; the expected result will be 300 or 1000 mg/L COD respectively. Or, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with deionized water.

Summary of Method

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). When the 3–150 mg/L colorimetric method is used, the amount of Cr^{6+} remaining is determined. When the 20–1500 mg/L colorimetric method is used, the amount of Cr^{3+} produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences. Test results for the 3 to 150 mg/L range are measured at 420 nm. Test results for the 20 to 1,500 mg/L COD range are measured at 620 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Select the appropriate TNTplus™ COD Digestion Reagent Vial:			
Low Range, 3 to 150 mg/L COD	1–2 vials	25/pkg	TNT821
High Range, 20 to 1500 mg/L COD	1–2 vials	25/pkg	TNT822

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor,115 V, 9 x 13 mm + 2 x 20 mm (mono block)	1	each	DRB200-01
DRB200 Reactor, 230 V, 9 x 13 mm + 2 x 20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable volume, 1–5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	1	100/pkg	27952-00
Test Tube Rack, 13-mm	1–2	each	24979-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
COD Standard Solution, 300-mg/L	200 mL	12186-29
COD Standard Solution, 1000-mg/L	200 mL	22539-29
Potassium Acid Phthalate, ACS	500 g	315-34
Oxygen Demand Standard (BOD, COD, TOC), 10-mL ampules	16/pkg	28335-10
Wastewater Influent Standard, for mixed parameters (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49
Wastewater Effluent Standard, for mixed parameters (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Beaker, 250 mL	each	500-46H
Blender, 2-speed, 120 VAC	each	26161-00
Blender, 2-speed, 240 VAC	each	26161-02
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 15x13 mm +15x13 mm (dual block)	each	DRB200-03
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230V, 15x13 mm+ 15x13 mm (dual block)	each	DRB200-07
DRB200 Reactor, 230 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-08
Stir Plate, magnetic	each	28812-00
Stir Bar, octagonal	each	20953-52
Sulfuric Acid, ACS	500 mL	979-49
TNTplus™ Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05

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Updated November 2005 Edition 1

Oxygen Demand, Chemical

★Method 8000 — Reactor Digestion Method[1]

(3 to 150, 20 to 1500, and 200 to 15,000 mg/L COD)

Scope and Application: For water, wastewater, and seawater; digestion is required; 3–150 mg/L and 20– 1500 mg/L COD ranges are USEPA approved for wastewater analyses[2]; 200–15,000 mg/L COD range are not USEPA approved.

[1] Jirka, A.M.; Carter, M.J., *Analytical Chemistry*, 1975, 47(8), 1397

[2] *Federal Register*, April 21, 1980, 45(78), 26811-26812

Test Preparation

Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and associated MSDS sheets.
Run one blank with each set of samples. Run all tests (the samples and the blank) with the same lot of vials. The lot number appears on the container label. See Blanks for Colorimetric Determination on page 4.
Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Be prepared to wash spills with running water
Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water. Review and follow instructions carefully.

Collect the following items:	**Quantity**
Beaker, 250-mL	1
Blender	1
COD Digestion Reagent vials	varies
DRB200 Reactor	1
Light Shield	1
Magnetic stirrer and stir bar	1
Opaque shipping container for storage of unused, light-sensitive reagent vials	varies
Pipet, TenSette®, 0.1 to 1.0 mL, with tips (for 200–15,000 mg/L range)	1
Pipet, volumetric, 2.00 mL	2
Pipet Filler, safety bulb	1
Test Tube Rack	2

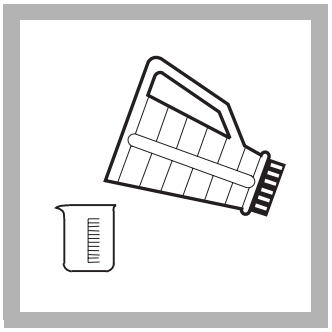
Note: *Reorder information for consumables and replacement items is on page 6.*

Note: *For greater accuracy, analyze a minimum of three replicates and average the results.*

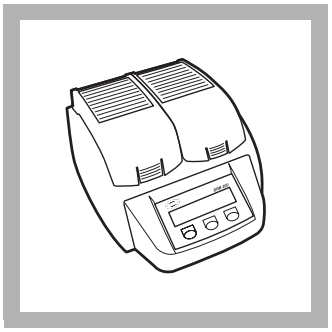
Digestion Procedure — Method 8000

1. Homogenize 100 mL of sample for 30 seconds in a blender. For samples containing large amounts of solids, increase the homogenization time.

If the sample does not contain suspended solids, omit steps 1 and 2.

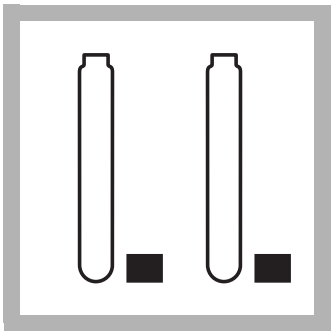


2. For the 200–15,000 mg/L range or to improve accuracy and reproducibility of the other ranges, pour the homogenized sample into a 250-mL beaker and gently stir with a magnetic stir plate.

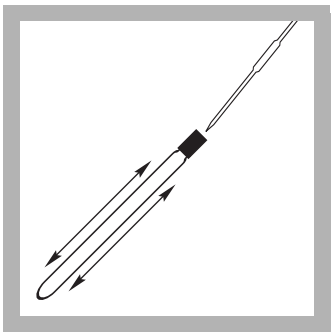


3. Turn on the DRB200 Reactor. Preheat to 150 °C.

See the DRB200 User Manual for selecting pre-programmed temperature applications.

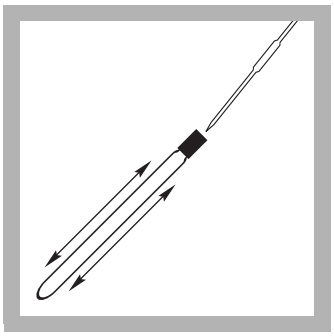


4. Remove the caps from two COD Digestion Reagent Vials. (Be sure to use vials for the appropriate range.)



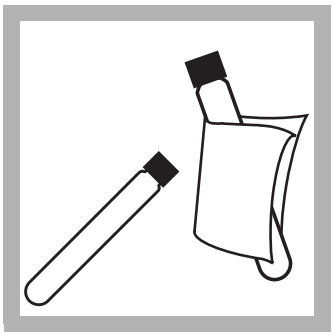
5. Prepared Sample: Hold one vial at a 45-degree angle. Use a clean volumetric pipet to add 2.00 mL of sample to the vial.

Use a TenSette® Pipet to add 0.20 mL for the 200–15,000 mg/L range.

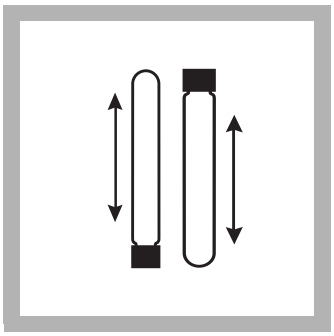


6. Blank Preparation: Hold a second vial at a 45-degree angle. Use a clean volumetric pipet to add 2.00 mL of deionized water to the vial.

Use a TenSette Pipet to add 0.20 mL for the 200–15,000 mg/L range.

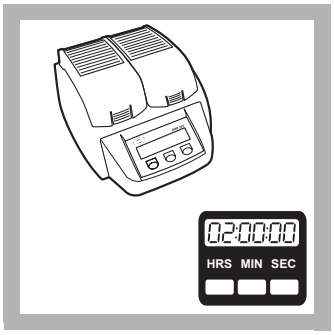


7. Cap the vials tightly. Rinse them with water and wipe with a clean paper towel.

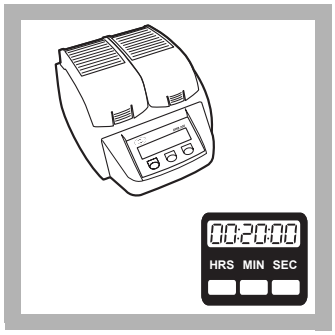


8. Hold the vials by the cap over a sink. Invert gently several times to mix. Insert the vials in the preheated DRB200 Reactor. Close the protective lid.

The sample vials will become very hot during mixing.

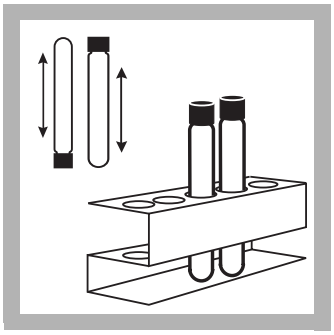


9. Heat the vials for two hours.



10. Turn the reactor off.

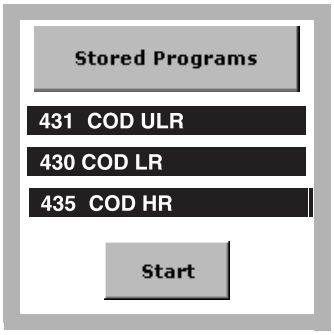
Wait about 20 minutes for the vials to cool to 120 °C or less.



11. Invert each vial several times while still warm. Place the vials into a rack and cool to room temperature.

Proceed to Colorimetric Determination Method 8000.

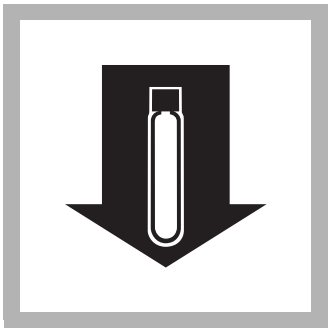
Colorimetric Determination Method 8000



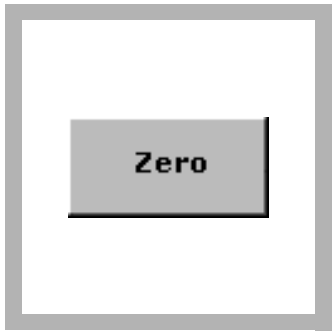
1. Select the ultra-low range, low range, or high range test.

Install the Light Shield in Cell Compartment #2.

2. Clean the outside of the vials with a damp towel followed by a dry one.



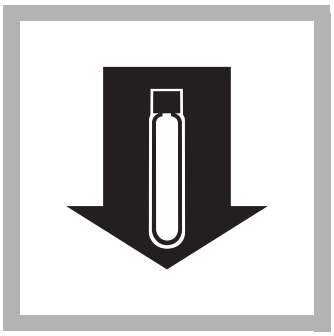
3. Insert the blank into the 16-mm cell holder.



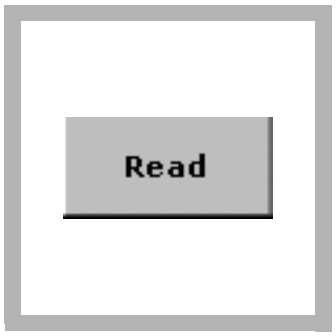
4. Press **ZERO**.

The display will show:

0.0 mg/L COD

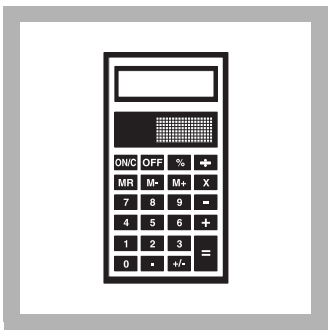


5. Insert the sample vial into the 16-mm cell holder.



6. Press **READ**.

Results are in mg/L COD.



7. If using High Range Plus COD Digestion Reagent Vials, multiply the result by 10.

For most accurate results with samples near 1500 or 15,000 mg/L COD, repeat the analysis with a diluted sample.

Blanks for Colorimetric Determination

The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark. Monitor decomposition by measuring the absorbance at the appropriate wavelength (356, 420, or 620 nm). Zero the instrument in the absorbance mode, using a vial containing 5 mL of deionized water and measure the absorbance of the blank. Record the value. Prepare a new blank when the absorbance has changed by about 0.01 absorbance units.

Interferences

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to the level specified in column 1 in Table 1. Samples with higher chloride concentrations should be diluted. Dilute the sample enough to reduce the chloride concentration to the level given in column 3.

If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.50 g of mercuric sulfate ($HgSO_4$) (Cat. No. 1915-20) to each COD vial before the sample is added. The additional mercuric sulfate will raise the maximum chloride concentration allowable to the level given in column 4 of Table 1.

Table 1 Interferences and Levels

Vial Type Used	Maximum Cl^- concentration in sample (mg/L)	Suggested Cl^- concentration of diluted samples (mg/L)	Maximum Cl^- concentration in sample when 0.50 $HgSO_4$ added
Ultra Low Range (0.7–40.0 mg/L)	2000	1000	N/A
Low Range (3–150 mg/L)	2000	1000	8000
High Range (20–1500 mg/L)	2000	1000	4000
High Range Plus (200–15,000 mg/L)	20,000	10,000	40,000

Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid* to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the 0.7 to 40.0 mg/L range with a 30 mg/L COD standard solution. Using class A glassware, prepare a 1000 mg/L solution by diluting 850 mg dried (120 °C, overnight) potassium acid phthalate (KHP) in 1000 mL of organic–free deionized water. Prepare a 30 mg/L dilution by diluting 3.00 mL of this solution into a 100.0 mL volumetric flask. Dilute to volume with deionized water, stopper, and invert 10 times to mix. Use 2 mL as the sample volume. The result should be 30 mg/L COD.

   - To adjust the calibration curve using the reading obtained with the 30 mg/L COD standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
   - Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST.**

2. Check the accuracy of the 3 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2 mL as the sample volume. The result should be 100 mg/L COD. Or dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to produce a 100-mg/L standard.

   - To adjust the calibration curve using the reading obtained with the 100 mg/L COD standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
   - Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST.**

3. Check the accuracy of the 20 to 1,500 mg/L range by using either a 300 mg/L or 1000 mg/L COD Standard Solution. Use 2 mL of one of these solutions as the sample volume; the expected result will be 300 or 1000 mg/L COD respectively. Or, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with deionized water.

   - To adjust the calibration curve using the reading obtained with the 300 mg/L or 1000 mg/L COD standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
   - Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST.**

* See Optional Reagents on page 7.

4. Check the accuracy of the 200 to 15,000 mg/L range by using a 10,000 mg/L COD standard solution. Prepare the 10,000 mg/L solution by dissolving 8.500 g of dried (120 °C, overnight) KHP in 1 liter of deionized water. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD.

   - To adjust the calibration curve using the reading obtained with the 10,000 mg/L COD standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
   - Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST.**

Alternate Reagents

Mercury-free COD2 Reagents can provide a mercury-free testing option for non-reporting purposes. For process control applications, COD2 Reagents will eliminate mercury waste and save on disposal costs. These reagents are fully compatible with test procedures and calibration curves programmed into the spectrophotometer. Determine chloride and ammonia for accurate results.

Note: *These reagents are not approved for USEPA reporting purposes. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications.*

Summary of Method

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). When the 0.7–40.0 or the 3–150 mg/L colorimetric method is used, the amount of Cr^{6+} remaining is determined. When the 20–1500 mg/L or 200–15,000 mg/L colorimetric method is used, the amount of Cr^{3+} produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences. Test results for the 0.7 to 40.0 mg/L range are measured at 350 nm. Test results for the 3 to 150 mg/L range are measured at 420 nm. Test results for the 20 to 1500 and the 2000 to 15,000 mg/L COD range are measured at 620 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
Ultra Low Range, 0.7 to 40 mg/L COD	1–2 vials	25/pkg	24158-25
Low Range, 3 to 150 mg/L COD	1–2 vials	25/pkg	21258-25
High Range, 20 to 1500 mg/L COD	1–2 vials	25/pkg	21259-25
High Range Plus, 200 to 15,000 mg/L COD	1–2 vials	25/pkg	24159-25
Water, deionized	varies	4 L	272-56

Alternate Reagents[1]

Description	Quantity/Test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
COD2, Low Range, 0 to 150 mg/L COD	1–2 vials	25/pkg	25650-25
COD2, High Range, 0 to 1500 mg/L COD	1–2 vials	25/pkg	25651-25
COD2, High Range, 0 to 1500 mg/L COD	1–2 vials	150/pkg	25651-15
COD2, High Range Plus, 0 to 15,000 mg/L COD	1–2 vials	25/pkg	28343-25

[1] These reagents are not approved for USEPA reporting purposes. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Blender, 2-speed, 120 VAC	1	each	26161-00
Blender, 2-speed, 240 VAC	1	each	26161-02
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Light Shield	1	each	LZV646

Recommended Standards

Description	Unit	Cat. No.
COD Standard Solution, 300-mg/L	200 mL	12186-29
COD Standard Solution, 1000-mg/L	200 mL	22539-29
Oxygen Demand Standard (BOD, COD, TOC)	16 10-mL ampules	28335-10
Pipet Filler, safety bulb	1	each
Pipet, TenSette®, 0.1 to 1.0 mL	1	each
Pipet Tips, for TenSette Pipet 19700-01	1	50/pkg
Pipet, Volumetric, Class A, 2.00 mL	1	each
Potassium Acid Phthalate, ACS	500 g	315-34
Stirrer, Electromagnetic, 120 VAC, with electrode stand	1	each
Stirrer, Electromagnetic, 230 VAC, with electrode stand	1	each
Test Tube Rack	1–2	each

Optional Reagents

Description	Unit	Cat. No.
COD Digestion Reagent Vials, 3 to 150 mg/L COD	150/pkg	21258-15
COD Digestion Reagent Vials, 200 to 1500 mg/L COD	150/pkg	21259-15
Mercuric Sulfate, 28 g	—	1915-20
Sulfuric Acid, 500 mL	—	979-49

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Updated November 2005 Edition 1

Oxygen Demand, Chemical

Method 10067

Manganese III Reactor Digestion Method (with optional chloride removal)[1]

(30 to 1000 mg/L COD Mn)

Scope and Application: For water and wastewater

[1] U.S. Patent 5,556,787

Test Preparation

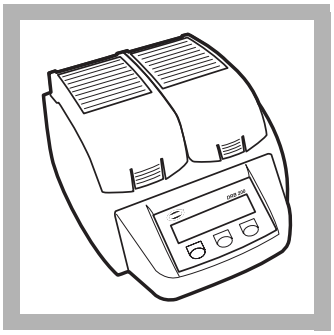
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
To determine if the sample contains chloride, use Quantab® Titrator Strips for low range chloride.
If the sample COD is expected to exceed 1000 mg/L, dilute the sample as described in Table 1 on page 3.
Homogenizing the sample promotes even distribution of solids and improves accuracy and reliability.
Run one blank with each lot of reagent. Run all samples and blanks with the same lot of vials. The lot number appears on the container label.
The stability of the reagent blank allows for reuse. Verify the reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range should be about 1.41–1.47.
If the sample boils during the digestion, the vial is not properly sealed. Test results will be invalid.
Spilled reagent will affect test accuracy and is hazardous. Do not run tests with spilled vials.
The maximum range of the VPD gauge is 40 inches of water; it will not indicate the full vacuum level obtained. Full vacuum is 20–25 inches of mercury; this can be measured at the vacuum pump with a gauge calibrated for inches of mercury.

Collect the following items	**Quantity**
Blender	1
DRB200 Reactor	1
Forceps, extra fine	1
Light Shield	1
Manganese III COD Reagent Vials, 20–1000 mg/L COD	1
Pipet, TenSette® (0.1 to 1.0 mL and 1.0 to 10.0 mL)	1 each
Pipet Tips for TenSette Pipet	2 of each
Sulfuric Acid, concentrated, ACS	1 mL
Test Tube Rack	1
Vacuum pretreatment device	1
Vacuum Pump	1
Vial, glass, for sample and acid	2
Water, deionized	varies

Note: *Reorder information for consumables and replacement items is on page 8.*

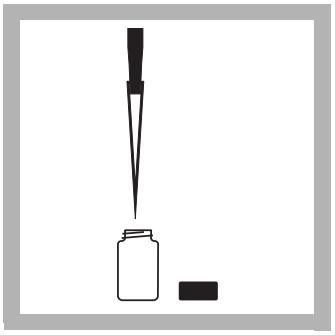
Preparing the Acidified Sample



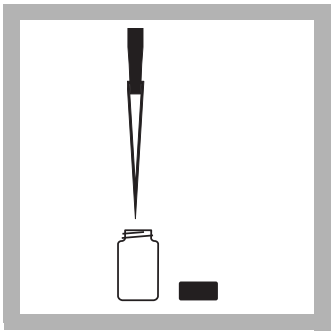
1. Turn on the DRB200 Reactor and heat to 150 °C.

2. Homogenize 100 mL of sample for 30 seconds in a blender.

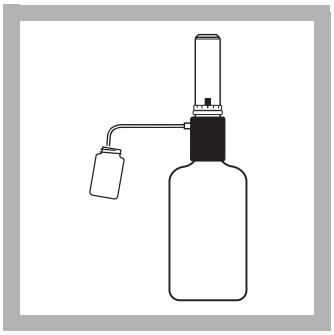
If suspended solids are present, continue to mix the sample while pipetting.



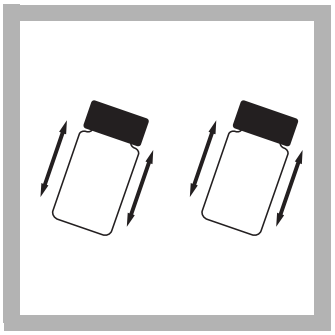
3. Blank Preparation: Pipet 9.0 mL of deionized water into an empty glass mixing cell.



4. Prepared Sample: Pipet 9.0 mL of homogenized sample into another empty glass mixing cell



5. Using an automatic dispenser or TenSette® Pipet, add 1.0 mL of concentrated sulfuric acid to both the sample cell and the blank.



6. Cap the cells tightly and invert several times.

The solution will become hot. Cool to room temperature before proceeding.

Acidified samples are stable for several months when refrigerated at 4 °C.

7. Proceed to Using the Vacuum Pretreatment Device on page 3.

Calculating the Multiplication Factor

All dilutions require that the ratio of sample to sulfuric acid remain at 9:1. For other dilutions that are not listed in Table 1, add the sample volume and the deionized water and divide by the sample volume to obtain the multiplication factor.

Mixing concentrated sulfuric acid and water is not aditive. Adding 1.0 mL of concentrated sulfuric acid to 9.0 mL of sample does not result in final volume of 10.0 mL. This factor is built into the calibration curve.

Note: *Mixing concentrated sulfuric acid and water is not additive. Adding 1.0 mL of concentrated sulfuric acid to 9.0 mL of sample does not result in a final volume of 10.0 mL. This factor is built into the calibration curve.*

Table 1 Multiplication Factors

Sample (mL)	Deionized Water (mL)	Range (mg/L COD)	Multiplication Factor
6.0	3.0	30–1500	1.5
3.0	6.0	60–3000	3
1.0	8.0	180–9000	9
0.5	8.5	360–18,000	18

For best results, use 0.5 mL or more of sample for diluting. If sample values exceed 18,000 mg/L COD, use a separate sample dilution before performing the sample chloride removal procedure.

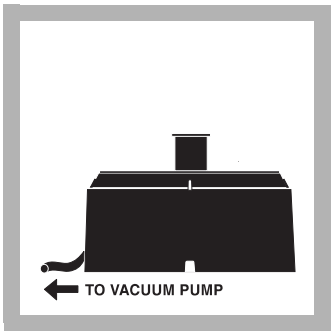
Example: Dilute the sample to a range of 90–4500 mg/L COD.

Sample Volume (2.0 mL) + Deionized water (7.0 mL) = Total Volume (9.0 mL)

$$\text{Multiplication Factor} = \frac{\text{Total Volume}}{\text{Sample Volume}} = \frac{9.0\ \text{mL}}{2.0\ \text{mL}} = 4.5$$

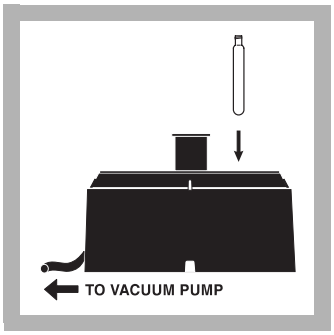
Standard test range is 50 to 1000 mg/L COD.
Example test range = 4.5(50) to 4.5(1000) = 225 to 4500 mg/L COD

Using the Vacuum Pretreatment Device

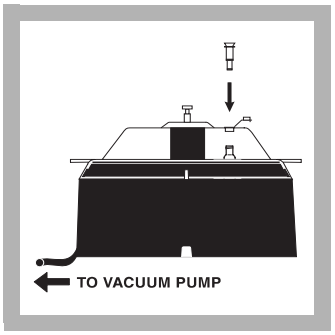


1. Attach the Vacuum Pretreatment Device[1] (VPD) to a vacuum pump (not an aspirator-type vacuum) that can create a vacuum of 20–25 inches of mercury.

[1] Patent Pending

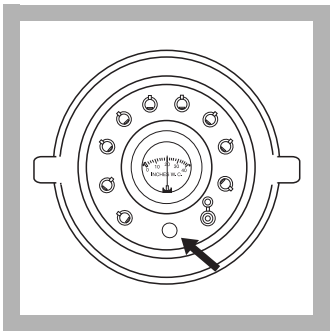


2. Label each Mn III COD vial and remove the cap. Insert the vials in one of the numbered holes in the VPD base.



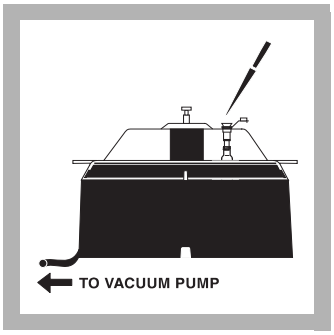
3. Place the VPD top on the base. Insert a fresh Chloride Removal Cartridge[2] (CRC) directly above each Mn III COD Reagent Vial. Plug any open holes in the VPD top using the stoppers provided.

[2] U.S. Patents 5,667,754; 5,683,914

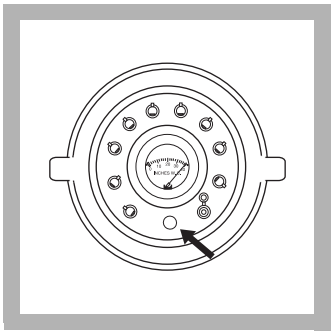


4. Turn on the vacuum pump and adjust the vacuum regulator valve on top of the VPD until the internal gauge reads 20 inches of water.

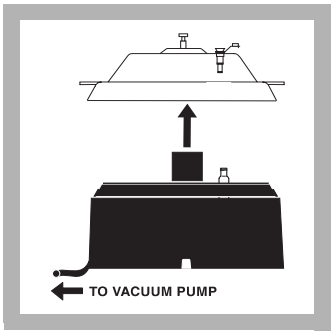
If the sample does not flow through the Chloride Removal Cartridge (CRC), increase the vacuum until flow starts, then reduce the vacuum down to 20 inches of water. Proceed as usual.



5. Pipet 0.60 mL of acidified sample (see Preparing the Acidified Sample on page 2) into the CRC. Pipet 0.6 mL of acidified blank into another CRC. It should take 30–45 seconds to draw the liquid through the CRC into each vial.



6. Close the vacuum regulator valve completely to achieve full vacuum. After one minute of full vacuum, slide the VPD back and forth several times to dislodge any drops clinging to the cartridge.

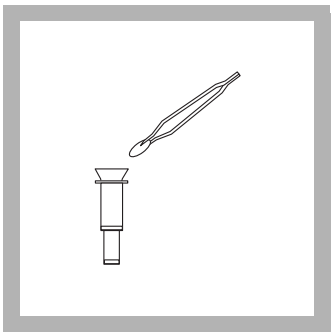


7. Open the VPD regulator valve to release the vacuum. Turn the pump off. Remove the VPD top and set it beside the base.

Dispose of the used Chloride Removal Cartridge. Do not reuse it.

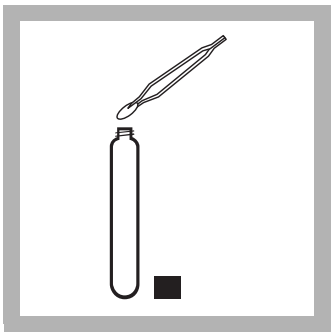
8. Proceed to Sample Preparation and Measurement on page 4.

Sample Preparation and Measurement



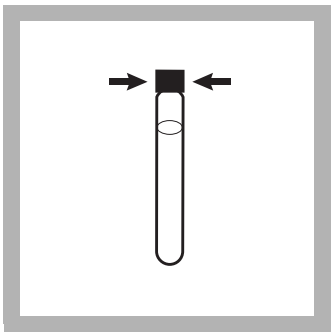
1. Use forceps to remove the filter from the top of each CRC.

If the sample does not contain suspended solids, it is not necessary to transfer the filter to the digestion vial.

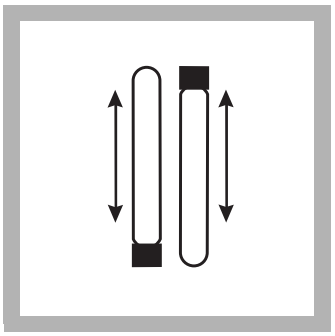


2. Insert each filter in the corresponding Mn III COD Vial. (Use numbers on the VPD as a guide.)

To avoid cross-contamination between samples, clean forcep tips between samples by wiping with a clean towel or rinsing with deionized water.



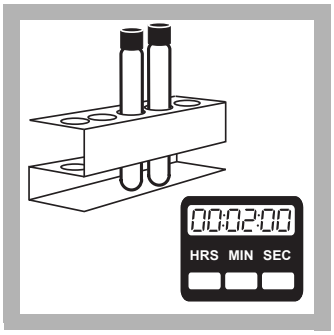
3. Remove the Mn III COD vial from the vacuum chamber and replace the original cap. Screw the cap on tightly.



4. Invert the vials several times to mix.

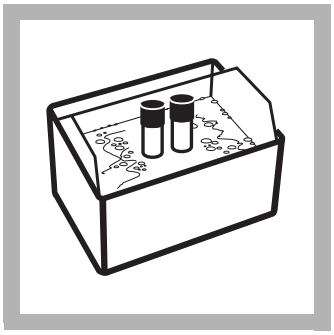
5. Insert the vials in the DRB200 Reactor at 150 °C. Close the protective cover. Digest for one hour.

To oxidize resistant organics, samples can be digested for up to four hours. Digest the blank for the same time period as the samples.

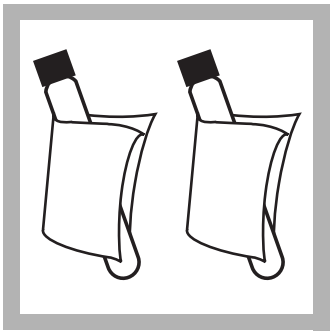


6. Insert the vials in a cooling rack for two minutes.

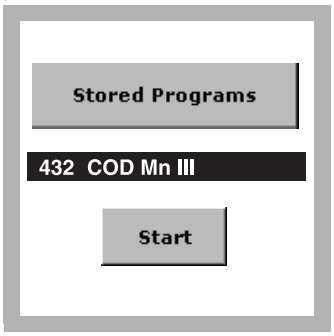
If the solution develops a colorless upper layer and a purple lower layer, invert the vial several times to mix and proceed to the next step.



7. Cool the vials to room temperature in a cool water bath or with running tap water for several minutes.



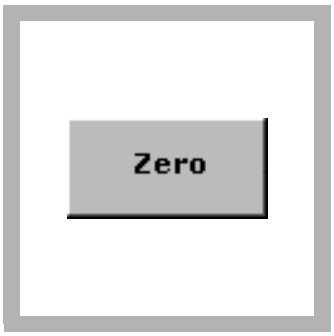
8. Remove the vials from the water and wipe with a clean, dry paper towel.



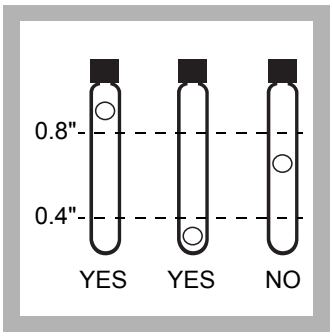
9. Select the test.

Install the Light Shield in Cell Compartment #2.

10. Insert the blank into the 16-mm cell holder.



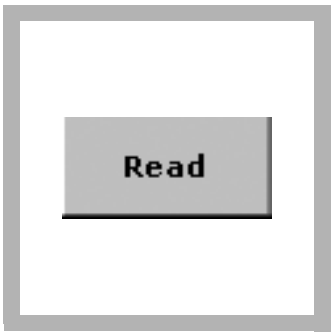
11. Press **ZERO**. The display will show:
0 mg/L COD Mn.



12. Make sure the filter disc is not suspended in the middle of the vial; it can interfere with the instrument reading.

The disc must be more than 20 mm (0.8"), or less than 10 mm (0.4"), from the bottom of the vial. Move it by gently swirling, or by lightly tapping the vial on the table top.

13. Wipe the prepared sample and insert it into the cell holder.



14. Press **READ**.

Results are in mg/L COD Mn.

Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted. A simple way to determine if chloride will affect test results is to run routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected after determining their concentrations with separate methods and adjusting the final COD test results accordingly.

Ammonia nitrogen is known to interfere in the presence of chloride; it does not interfere if chloride is absent.

Sample Collection, Preservation, and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions.

Accuracy Check

Standard Solution Method

1. Purchase a 800-mg/L COD Standard Solution or prepare an 800-mg/L COD standard solution by adding 0.6808 g of dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.60 mL of this solution as the sample volume. (Use 0.50 mL if performing the method version without chloride removal.)

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Chemical Oxygen Demand (COD) is defined as "... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant" (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, non-carcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to four hours for samples that are difficult to oxidize. Test results are measured at 510 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Manganese III COD Reagent Vials, 20–1000 mg/L COD	1	25/pkg	26234-25
Chloride Removal Cartridge (CRC)	1	25/pkg	26618-25
Sulfuric Acid, concentrated, ACS	1 mL	2.5 L	979-09
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Blender, 120 VAC	1	each	26747-00
Blender Container, 50–250 mL	1	2/pkg	26748-00
Cap, with inert Teflon liner, for mixing bottle	varies	12/pkg	24018-12
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Forceps, extra fine point	1	each	26696-00
Light Shield	1	each	LZV646
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips for TenSette Pipet19700-10	2	50/pkg	21997-96
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips for TenSette Pipet 19700-01	2	50/pkg	21856-96
Test Tube Rack	1	each	18641-00
Vacuum Pretreatment Device (VPD)	1	each	49000-00
Vacuum Pump, 1.2 CFM @ 60 Hz, 27.2 Hg Max, 1/8 HP, UL listed	1	each	28248-00
Vial, glass, for sample plus acid	2	each	24277-00

Recommended Standards

Description	Unit	Cat. No.
COD Standard Solution, 800-mg/L COD	200 mL	26726-29
Oxygen Demand Standard for BOD, COD, TOC, 10-mL ampules	16/pkg	28335-10
Potassium Acid Phthalate, ACS	500 g	315-34
Wastewater Standard, Influent Inorganics, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Cat. No.
Dispenser, automatic, 1.0–5.0 mL	25631-37
Titrator Strips, Quantab®, for low range chloride	27449-40

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WORLD HEADQUARTERS
Telephone: (970) 669-3050
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Updated November 2005 Edition 1

Oxygen Demand, Chemical

Method 10067

Manganese III Reactor Digestion Method (without chloride removal)[1]

(30 to 1000 mg/L COD Mn)

Scope and Application: For water and wastewater

[1] U.S. Patent 5,556,787

Test Preparation

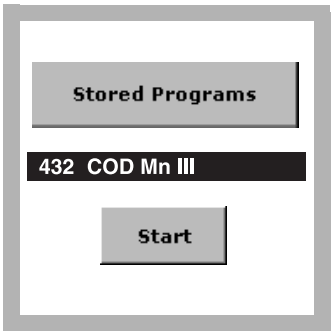
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
If the sample contains chloride, use the chloride removal method. To determine if the sample contains chloride, use Quantab® Titrator Strips for low range chloride.
If the sample COD value is not between 30 and 1000 mg/L, dilute the sample with deionized water to obtain this range. Multiply the final result by the dilution factor.
Homogenizing the sample promotes even distribution of solids and improves accuracy and reliability.
Stability of the reagent blank allows for reuse. Verify the reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range should be about 1.4–1.5.
If the sample boils during the digestion, the vial is not properly sealed. Test results will be invalid.
Use finger cots to handle hot sample cells.Spilled reagent will affect test accuracy and is hazardous. Do not run tests with spilled vials.
See the DRB200 User Manual for selecting pre-programmed temperature applications.

Collect the following items:	**Quantity**
Blender	1
DRB200 Reactor	1
Light Shield	1
Manganese III COD Reagent Vials, 20–1000 mg/L COD	1
Pipet, TenSette® (0.1 to 1.0 mL)	1
Pipet tips for TenSette Pipet	2
Test Tube Rack	1
Water, deionized	varies

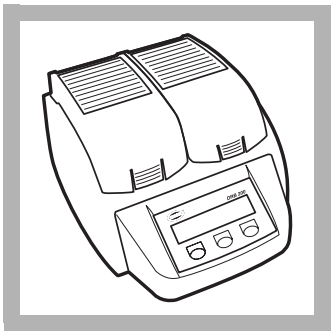
Note: *Reorder information for consumables and replacement items is on page 4.*

Without Chloride Removal — Method 10067



1. Select the test.

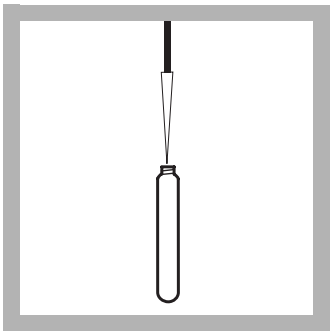
Install the Light Shield in Cell Compartment #2.



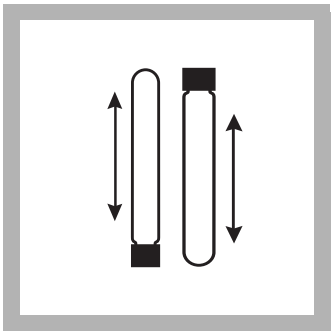
2. Turn on the DRB200 Reactor and heat to 150 °C.

3. Homogenize 100 mL of sample for 30 seconds in a blender.

If suspended solids are present, continue to mix the sample while pipetting.



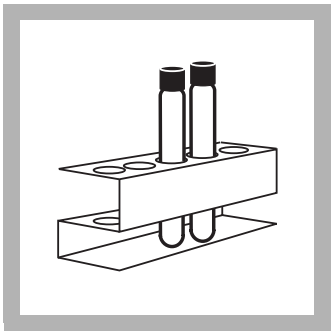
4. Pipet 0.5 mL of homogenized sample into one Mn III COD vial (the prepared sample) and 0.5 mL of deionized water into another Mn III COD vial (the blank).



5. Cap and invert several times to mix.

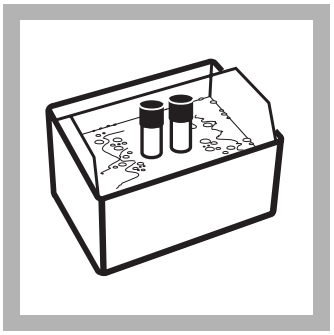
6. Insert the vials in the DRB200 Reactor at 150 °C. Close the protective cover. Digest for one hour.

Digest more resistant organics and the blank for up to four hours.

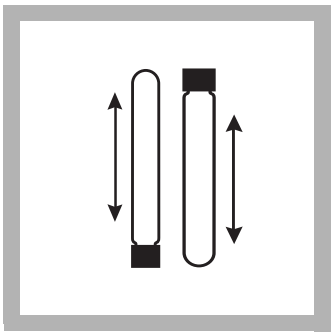


7. Remove the vials and place them in a cooling rack for two minutes.

If a vial develops a colorless upper layer and a purple lower layer, invert the vial several times to mix and proceed.

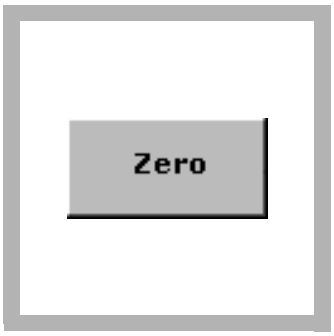


8. Cool the vials to room temperature in a cool water bath or with running tap water. This takes several minutes.

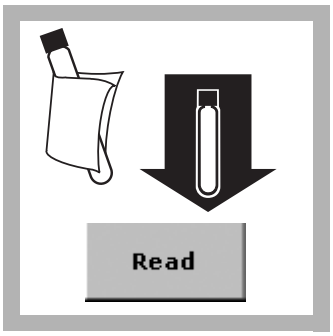


9. Invert the vials several times to mix.

10. Wipe the blank and insert it into the 16-mm round cell holder.



11. Press **ZERO**. The display will show:
0 mg/L COD Mn



12. Wipe the sample and insert it into the 16-mm round cell holder.

Press **READ**. Results are in mg/L COD Mn.

Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted.
A simple way to determine if chloride will affect test results is to run routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected after determining their concentrations with separate methods and adjusting the final COD test results accordingly.

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Accuracy Check

Standard Solution Method

1. Purchase a 800-mg/L COD Standard Solution or prepare an 800-mg/L COD standard solution by adding 0.6808 g of dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume.
2. To adjust the calibration curve using the reading obtained with the 800-mg/L COD standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

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Consumables and Replacement Items

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Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Blender, 120 VAC	1	each	26747-00
Blender Container, 50–250 mL	1	2/pkg	26748-00
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Light Shield	1	each	LZV646
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet19700-01	2	50/pkg	21856-96
Test Tube Rack	1	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
COD Standard Solution, 800-mg/L COD	200 mL	26726-29
Oxygen Demand Standard for BOD, COD, TOC, 10-mL ampules	16/pkg	28335-10
Potassium Acid Phthalate, ACS	500 g	315-34
Wastewater Standard, Influent Inorganics, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

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Updated November 2005 Edition 1

Oxygen Demand, Chemical

★Method 10211

Reactor Digestion Method

TNTplus™ 820

ULR (1–60 mg/L COD)

Scope and Application: For wastewater, process water, surface water, and cooling water; digestion is required

Test Preparation

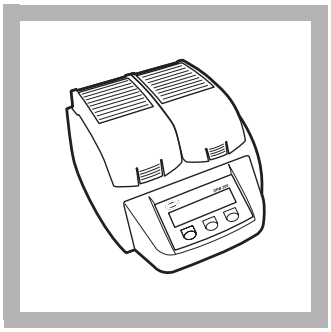
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F).
Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and associated MSDS sheets.
To run the optional blank for a set of samples, see Blanks for Colorimetric Determination on page 3.
TNTplus methods are activated from the Main Menu screen by inserting the sample vial into the sample cell holder.
Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Be prepared to wash spills with running water.
Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water. Review and follow instructions carefully.
Store unused (light-sensitive) vials in a closed box.

Collect the following items:	**Quantity**
Blender	1
DRB200 Reactor with 13-mm wells (use adapters with 16-mm holes)	1
COD TNT820 Reagent Set	varies
Light Shield	1
Pipettor for 2.0 mL Sample	1
Pipettor Tip	1
Test Tube Rack	1–3

Note: *Reorder information for consumables and replacement items is on page 5.*

TNTplus Method 10211

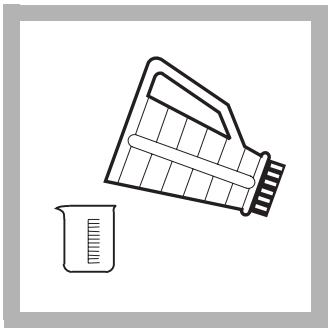


1. Turn on the DRB200 Reactor. Preheat to 150 °C.

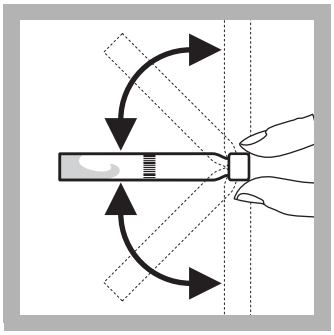
Note: *For DRB200 Reactors with 16-mm wells, insert a 16-mm to 13-mm adapter sleeve into each well* ***before*** *turning on the reactor.*

2. Homogenize 100 mL of sample for 30 seconds in a blender. For samples containing large amounts of solids, increase the homogenization time.

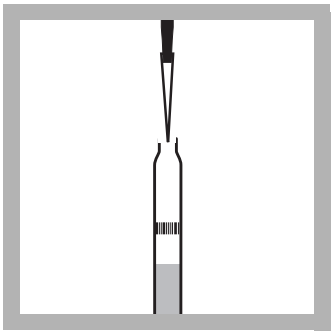
If the sample does not contain suspended solids, omit steps 2 and 3.



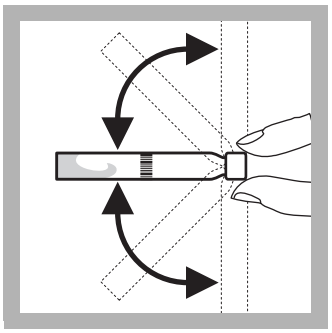
3. To help ensure that a representative portion of sample is analyzed, pour the homogenized sample into a 250-mL beaker and gently stir with a magnetic stir plate.



4. Invert a vial several times to bring the sediment in the bottom of the vial into suspension.

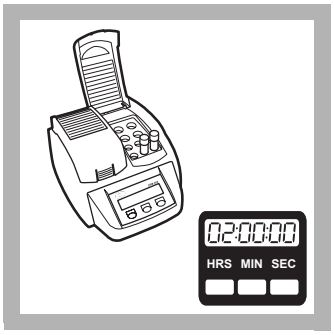


5. Carefully pipet 2.0 mL of sample into the vial. Cap and clean the outside of the vial.

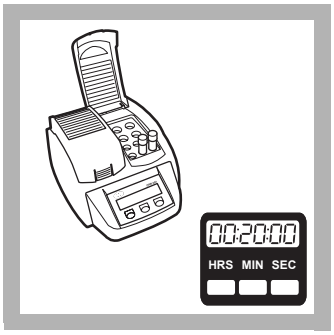


6. Hold the vial by the cap over a sink. Invert gently several times to mix. The sample vial will become very hot during mixing.

Place the vials in the preheated DRB200 Reactor. Close the protective lid

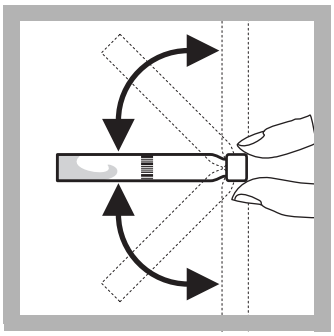


7. Heat for two hours.

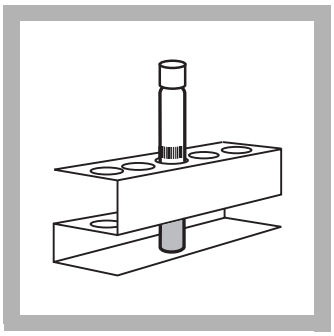


8. Turn the reactor off.

Wait about 20 minutes for the vial to cool to 120 °C or less.

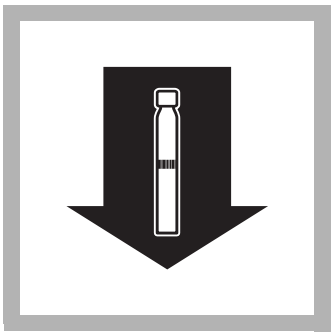


9. Invert the vial several times while still hot.



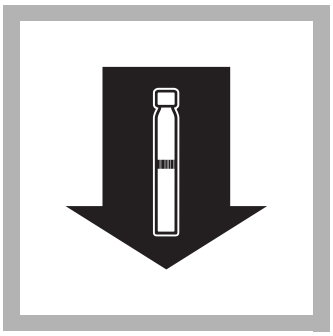
10. Place the vial into a rack to cool to room temperature.

Install the Light Shield in Cell Compartment #2.



11. Using the Zero vial from the sample vial lot, insert the Zero vial into the sample cell holder.

The instrument reads the barcode, then selects the method and set the instrument zero. The instrument displays L1 when the zeroing is complete.



12. Thoroughly clean the outside of the vial and insert it into the cell holder. Close the lid.

The instrument reads the barcode and reads the sample.

Results are in mg/L COD.

Blanks for Colorimetric Determination

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark, and monitor decomposition by measuring its concentration periodically.

To subtract the value of the blank from a series of measurements, measure the blank per step 12. Press **OPTIONS>MORE>REAGENT BLANK**. Select **ON**.The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to 1500 mg/L Cl^-. COD concentrations greatly in excess of the stated range will adversely affect color formation, resulting in a false reading that appears within the range of the method.

Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid* to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions.

* See Optional Reagents and Apparatus on page 5.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the 1 to 60 mg/L range with a 50 mg/L standard. Prepare by dissolving 42.5 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2 mL as the sample volume. The result should be 50 mg/L COD. Or, dilute 5 mL of 1000-mg/L COD Standard Solution to 100 mL to produce a 50-mg/L standard.

2. Alternatively, use 2.0 mL of a Wastewater Effluent Mixed Inorganics Standard Solution. This standard contains 25 mg/L COD in the presence of other ions such as phosphate, nitrate, ammonia, and sulfate.

Summary of Method

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). With this method, the amount of yellow Cr^{6+} remaining is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences. Test results are measured at 348 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Oxygen Demand, ULR TNT820 Reagent Set	1–2 vials	24/pkg	TNT820

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13 mm + 2x20mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13 mm + 2x20mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable volume, 1–5 mL	1	each	27951-00
Pipet Tips, for 27951-00 pipet	1	100/pkg	27952-00
Test Tube Rack	1–3	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
COD Standard Solution, 1000-mg/L	200 mL	22539-29
Potassium Acid Phthalate, ACS	500 g	315-34
Wastewater Effluent Standard, for mixed parameters (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Beaker, 250 mL	each	500-46H
Blender, 2-speed, 120 VAC	each	26161-00
Blender, 2-speed, 240 VAC	each	26161-02
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB200-03
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 15x13mm + 15x13 mm (dual block)	each	DRB200-07
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-08
Stir Plate, magnetic	each	28812-00
Stir Bar, octagonal	each	20953-52
Sulfuric Acid, ACS	500 mL	979-49
TNTplus™ Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05

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Updated November 2005 Edition 1

Oxygen Demand, Chemical

★Method 10212 **Reactor Digestion Method**

TNTplus™ 823 **UHR (250–15,000 mg/L COD)**

Scope and Application: For wastewater and process waters; digestion is required

Test Preparation

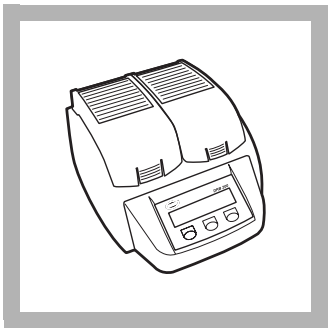
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F).
Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and associated MSDS sheets.
To run the optional blank for a set of samples, see Blanks for Colorimetric Determination on page 3.
Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Be prepared to wash spills with running water.
TNTplus methods are activated from the Main Menu screen by inserting the sample vial into the sample cell holder.
Wear appropriate eye protection and clothing for adequate user protection. If contact occurs, flush the affected area with running water. Review and follow instructions carefully.
Store unused (light-sensitive) vials in a closed box.

Collect the following items:	**Quantity**
Blender	1
DRB200 Reactor with 13-mm wells (use adapters with 16-mm holes)	1
COD, TNT823 Reagent Set	varies
Light Shield	1
Pipettor for 0.3 mL Sample	1
Pipettor Tip	1
Test Tube Rack	1–3

Note: *Reorder information for consumables and replacement items is on page 5.*

TNTplus Method 10212

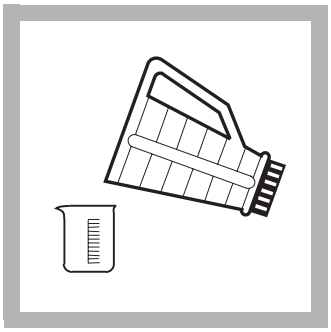


1. Turn on the DRB200 Reactor. Preheat to 150 °C.

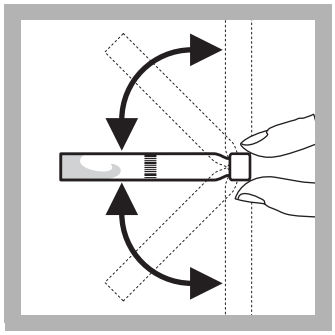
Note: *For DRB200 Reactors with 16-mm wells, insert a 16-mm to 13-mm adapter sleeve into each well* ***before*** *turning on the reactor.*

2. Homogenize 100 mL of sample for 30 seconds in a blender. For samples containing large amounts of solids, increase the homogenization time.

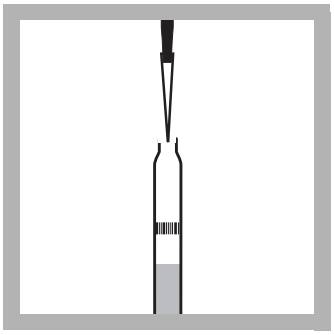
If the sample does not contain suspended solids, omit steps 2 and 3.



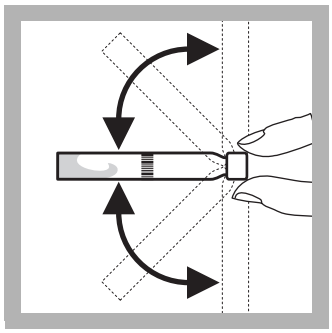
3. To help ensure that a representative portion of sample is analyzed, pour the homogenized sample into a 250-mL beaker and gently stir with a magnetic stir plate.



4. Invert a vial several times to bring the sediment in the bottom of the vial into suspension.

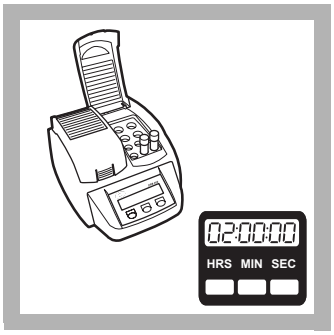


5. Carefully pipet 0.3 mL (300 µL) of sample into the vial. Cap and clean the outside of the vial.

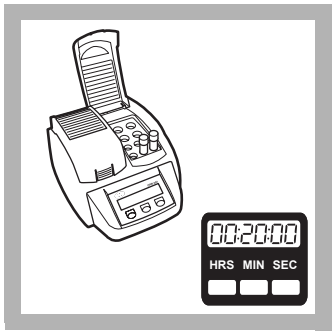


6. Hold the vial by the cap over a sink. Invert gently several times to mix. The sample vial will become very hot during mixing.

Place the vial in the preheated DRB200 Reactor. Close the protective lid

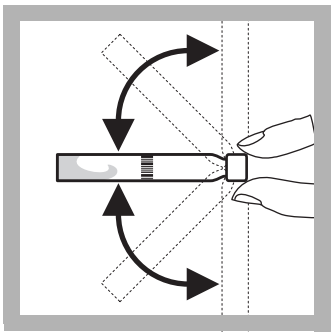


7. Heat for two hours.



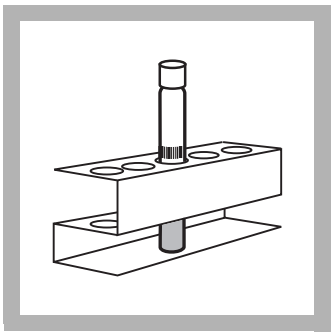
8. Turn the reactor off.

Wait about 20 minutes for the vial to cool to 120 °C or less.

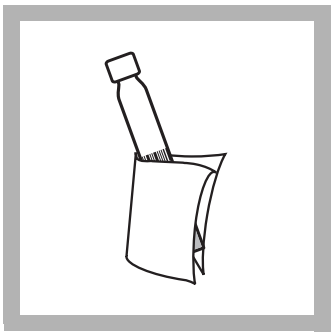


9. Invert the vial several times while still hot.

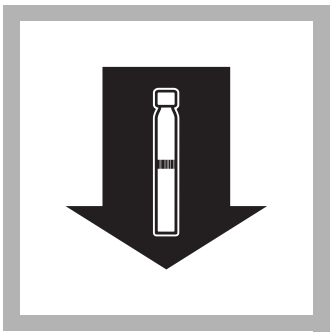
Install the Light Shield in Cell Compartment #2.



10. Place the vial into a rack to cool to room temperature.



11. Thoroughly clean the outside of the vial.



12. Insert the vial into the cell holder. Close the lid.

The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L COD.

Blanks for Colorimetric Determination

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark, and monitor decomposition by measuring its concentration periodically.

To subtract the value of the blank from a series of measurements, measure the blank per step 12. Press **OPTIONS>MORE>REAGENT BLANK**. Select **ON**.The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Interferences

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to 5000 mg/L Cl^-. COD concentrations greatly in excess of the stated range will adversely affect color formation, resulting in a false reading that appears within the range of the method.

Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid* to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions.

* See Optional Reagents and Apparatus on page 5.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the method range with a 1000 mg/L standard. Prepare by dissolving 0.8503 g of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 0.3 mL as the sample volume. Standard solutions of 1000mg/L COD and 617 mg/L COD are also available.

2. Alternatively, use 0.3 mL of a Wastewater Influent Mixed Inorganics Standard Solution. This standard contains 500 mg/L COD in the presence of other ions such as phosphate, nitrate, ammonia and sulfate.

Summary of Method

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). The amount of green color produced is directly proportional to amount of COD present. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex chloride interferences. Test results are measured at 620 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Oxygen Demand, UHR TNT823 Reagent Set	1–2 vials	25/pkg	TNT823

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13mm + 2x20mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable volume, 100–1000 µL	1	each	27949-00
Pipet Tips, for 27949-00 pipet	1	400/pkg	27950-00
Test Tube Rack	1–3	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
COD Standard Solution, 1000-mg/L	200 mL	22539-29
Potassium Acid Phthalate, ACS	500 g	315-34
Oxygen Demand Standard (BOD, COD, TOC), 10-mL ampules, 617 mg/L COD	16/pkg	28335-10
Wastewater Influent Standard, for mixed parameters (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Beaker, 250 mL	each	500-46H
Blender, 2-speed, 120 VAC	each	26161-00
Blender, 2-speed, 240 VAC	each	26161-02
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB200-03
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 15x13mm + 15x13 mm (dual block)	each	DRB200-07
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-08
Stir Plate, magnetic	each	28812-00
Stir Bar, octagonal	each	20953-52
Sulfuric Acid, ACS	500 mL	979-49
TNTplus™ Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05

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Updated November 2005 Edition 1

Oxygen Scavengers

Method 8140

Iron Reduction Method for Oxygen Scavengers

Powder Pillows

**5 to 600 µg/L carbohydrazide;
3 to 450 µg/L DEHA;
9 to 1000 µg/L hydroquinone;
13 to 1500 µg/L iso-ascorbic acid [ISA];
15 to 1000 µg/L methylethyl ketoxime [MEKO]**

Scope and Application: For testing residual corrosion inhibitors (oxygen scavengers) in boiler feed water or condensate

Test Preparation

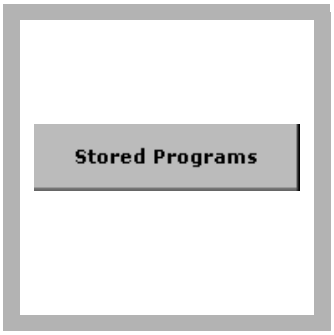
Before starting the test:

Analyze samples immediately. Do not preserve for later analysis.
The sample temperature should be 25 ± 3 °C (77 ± 5 °F).
Soak glassware with 1:1 hydrochloric acid solution. Rinse several times with deionized water. These two steps will remove iron deposits that can cause slightly high results.
To determine ferrous iron concentration, repeat the procedure, but do not add DEHA Reagent 2. Correct for the ferrous iron concentration: **OPTIONS>MORE>REAGENT BLANK>ON**. The reading attributed to the ferrous iron concentration will appear.

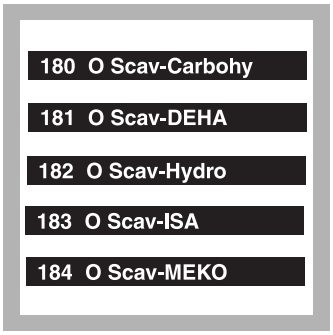
Collect the following items	Quantity
Bottle, glass mixing, with 25-mL mark	2
DEHA Reagent 1 Powder Pillow	2
DEHA Reagent 2 Solution	1 mL
Deionized Water	25 mL
Dropper, 0.5 and 1.0 mL marks	1
Hydrochloric Acid, 1:1, 6.0 N	varies
Sample Cells, 1-inch square, 10-mL	2

Note: *Reorder information for consumables and replacement items is on page 4.*

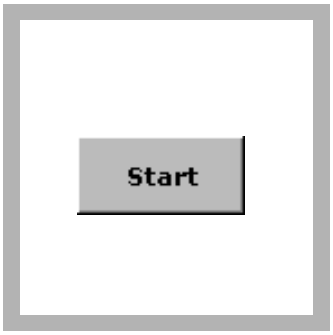
Powder Pillows Method 8140



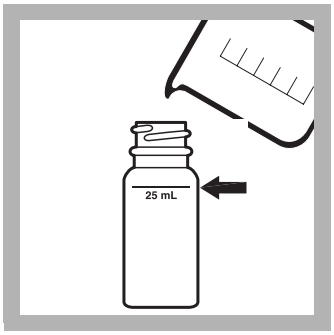
1. Press **STORED PROGRAMS**.



2. Select one of the tests.

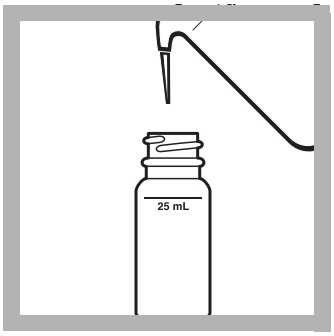


3. Press **START**.

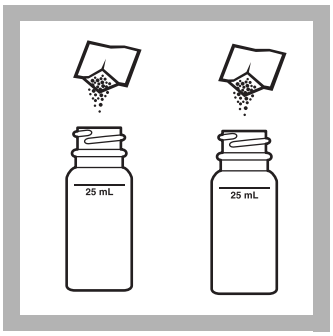


4. Prepared Sample: Fill a mixing bottle with 25 mL of sample.

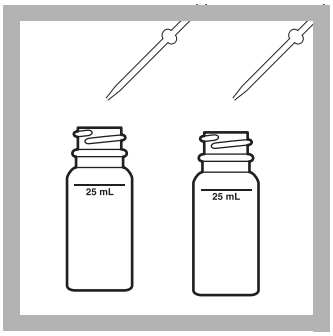
When determining oxygen scavengers that react quickly with oxygen at room temperature, cap the bottle.



5. Blank Preparation: Fill a second cell bottle 25 mL of deionized water.



6. Add the contents of one DEHA Reagent 1 Powder Pillow to each mixing bottle. Swirl to mix.



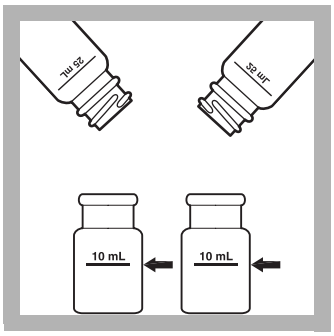
7. Add 0.5 mL of DEHA Reagent 2 Solution to each bottle. Mix. Place both sample cells in the dark.

A purple color will develop if an oxygen scavenger is present.

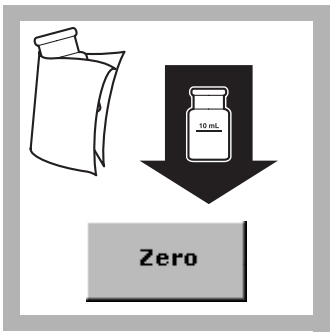
8. Press **TIMER>OK**.

A ten-minute reaction period (or a **two-minute** reaction period for **hydroquinone**) will begin.

Keep the sample cells in the dark during the reaction period.



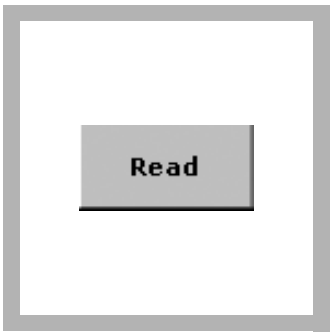
9. When the timer expires, transfer the blank and prepared samples into square sample cells.



10. Immediately after transferring to the 10-mL cell, wipe the blank and insert it into the cell holder with the fill line facing right. Close the cover. Press **ZERO**.

For greater accuracy, read the result immediately after the timer expires.

11. Immediately wipe the prepared sample and insert it into the cell holder with the fill line facing right.



12. Press **READ**.

Results are in µg/L.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Borate (as $Na_2B_4O_7$)	Greater than 500 mg/L
Cobalt	Greater than 0.025 mg/L
Copper	Greater than 8.0 mg/L
Ferrous Iron	All levels
Hardness (as $CaCO_3$)	Greater than 1000 mg/L
Light	Light may interfere. Keep sample cells in the dark during color development.
Lignosulfonates	Greater than 0.05 mg/L
Manganese	Greater than 0.8 mg/L
Molybdenum	Greater than 80 mg/L
Nickel	Greater than 0.8 mg/L
Phosphate	Greater than 10 mg/L
Phosphonates	Greater than 10 mg/L
Sulfate	Greater than 1000 mg/L
Temperature	Sample temperatures below 22 °C or above 28 °C (72 °F or 82 °F) may affect test accuracy.
Zinc	Greater than 50 mg/L

Substances which reduce ferric iron will interfere. Substances which complex iron strongly may also interfere.

Sample Collection, Preservation, and Storage

Collect samples in clean, dry, plastic or glass containers. Avoid excessive agitation or exposure to sunlight when sampling. Rinse the container several times with the sample. Allow the container to overflow and cap the container so that there is no headspace above the sample. Rinse the sample cell several times with sample, then carefully fill to the 25-mL mark. Perform the analysis immediately.

Summary of Method

Diethylhydroxylamine (DEHA) or other oxygen scavengers present in the sample react with ferric iron in DEHA Reagent 2 Solution to produce ferrous ion in an amount equivalent to the DEHA concentration. This solution then reacts with DEHA 1 Reagent, which forms a purple color with ferrous iron proportional to the concentration of oxygen scavenger. Test results are measured at 562 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Oxygen Scavenger Reagent Set, includes:	—	—	24466-00
(2) DEHA Reagent 1 Powder Pillows	2	100/pkg	21679-69
(1) DEHA Reagent 2 Solution	1 mL	100 mL	21680-42
Hydrochloric Acid, 1:1, 6.0 N	varies	500 mL	884-49
Water, deionized	25 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Bottle, glass mixing, with 25-mL mark	2	each	17042-00
Dropper, 0.5 and 1.0-mL marks	1	20/pkg	21247-20
Sample Cells, 10-20-25 mL, with caps	2	6/pkg	24019-06
Sample Cells, 1-inch square glass, 10 mL, matched pair	2	2/pkg	24954-02

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Updated November 2005 Edition 1

Ozone

Method 8311

Indigo Method

AccuVac® Ampul

LR (0.01 to 0.25 mg/L O_3),
MR (0.01 to 0.75 mg/L O_3),
HR (0.01 to 1.50 mg/L O_3)

Scope and Application: For water

Test Preparation

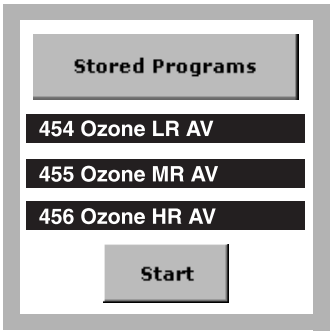
Before starting the test:

Analyze sample immediately. Do not preserve for later analysis.
Use tap water or deionized water for the blank (ozone-free water)
The sequence of measuring the blank and the sample is reversed in this procedure.

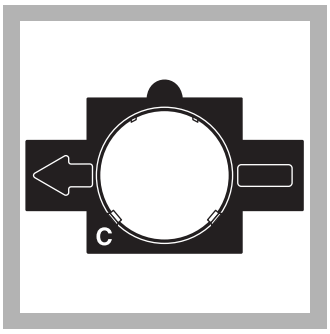
Collect the following items:	**Quantity**
Select Ozone AccuVac® Ampuls based on range:	
0–0.25 mg/L	2
0–0.75 mg/L	2
0–1.50 mg/L	2
Beaker, Polypropylene, 50-mL, low form with pour spout	1
Water, ozone-free	varies

Note: *Reorder information for consumables and replacement items is on page 3.*

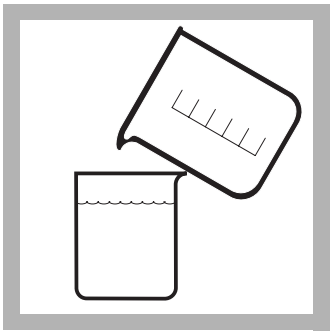
AccuVac Ampul — Method 8311



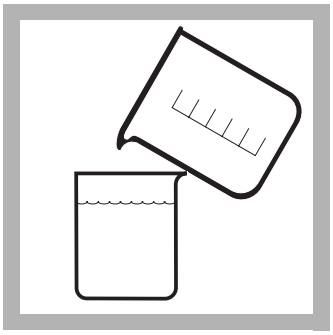
1. Select the test.



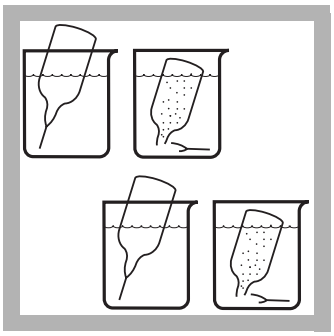
2. Insert Adapter C.



3. Blank Preparation: Collect at least 40 mL of ozone-free water in a 50-mL beaker.



4. Prepared Sample: Gently collect at least 40 mL of sample in another 50-mL beaker.



5. Fill one Indigo Ozone Reagent AccuVac Ampul with the sample and another with the blank.

Keep the tip immersed while the Ampul fills.

6. Quickly invert both Ampuls several times to mix.

Some of the blue color will be bleached if ozone is present.

7. Wipe the Ampuls with a cloth to remove fingerprints or other marks.

Insert the sample into the cell holder.

Press **ZERO**.

The display will show:

0.00 mg/L O_3

8. Insert the blank into the cell holder.

Press **READ**. Results are in mg/L O_3.

Sample Collection, Storage, and Preservation

The most important consideration when collecting a sample is to prevent the escape of ozone from the sample. The sample should be collected gently and analyzed immediately. Warming the sample, or disturbing the sample by stirring or shaking, will result in ozone loss. After collecting the sample, do not transfer it from one container to another unless absolutely necessary.

Stability of Indigo Reagent

Because indigo is light-sensitive, the AccuVac Ampuls should be kept in the dark at all times. The indigo solution, however, decomposes slowly under room light after filling with sample. The blank Ampul can be used for multiple measurements during the same day.

Summary of Method

The reagent formulation adjusts the sample pH to 2.5 after the Ampul has filled. The indigo reagent reacts immediately and quantitatively with ozone. The blue color of indigo is bleached in proportion to the amount of ozone present in the sample. Other reagents in the formulation prevent chlorine interference. No transfer of sample is needed in the procedure, therefore ozone loss due to sampling is eliminated. Test results are measured at 600 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Select one or more Ozone AccuVac® Ampuls based on range:			
0–0.25 mg/L	2	25/pkg	25160-25
0–0.75 mg/L	2	25/pkg	25170-25
0–1.5 mg/L	2	25/pkg	25180-25

Required Apparatus (AccuVac)

Description	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each
Polypropylene Beaker, 50-mL, Low Form, with pour spout	each	1080-41

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Snapper, AccuVac®	each	24052-00
Water, demineralized	4 L	272-56

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Updated November 2005 Edition 1

PCB (Polychlorinated Biphenyls)

Method 10050

Immunoassay Method[1]

Scope and Application: For soil

[1] This test is semi-quantitative. Results are expressed as greater or less than the threshold value used.

Test Preparation

This method analyzes for PCB that has been extracted from soil samples. Sample extracts, calibrators, and reagents are added to cuvettes coated with PCB-specific antibodies. The color that develops is then measured and compared with the color measurements of the calibrators. The test requires about 20 minutes for complete analysis. As many as 10 cuvettes can be run simultaneously.

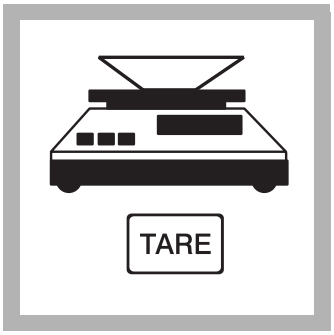
Before starting the test:

Read the entire procedure before starting. Identify and make ready all the necessary reagents, cuvettes, and other apparatus before beginning the analysis.
Timing is critical; follow instructions carefully.
A consistent technique when mixing the cuvettes is critical to this test. The best results come from using the cuvette rack and mixing as described in Using the 1-cm MicroCuvette Rack on page 6. Cuvettes can be mixed individually, but test results may not be as consistent.
Handle the cuvettes carefully. Scratches on the inside or outside may cause erroneous results. Carefully clean the outside of the cuvettes with a clean absorbent cloth or tissue before placing them into the instrument.
Antibody cuvettes and enzyme conjugate are made in matched lots. Do not mix reagent lots.
To avoid damaging the Color Developing Solution, do not expose it to direct sunlight.
There are two protocols in this procedure, one for levels of 1 ppm and 5 ppm, and another for 10 ppm and 50 ppm. Each uses a different quantity of calibrator and sample extract See PCB Protocols on page 5 for more information.
Store the reagents at 4 °C when they are not in use. Allow the reagents to reach room temperature before using them in an analysis. Actual testing may be done at temperatures ranging from 1– 38 °C.
The Soil Extractant contains methyl alcohol which is poisonous and flammable. Before using this and other reagents, read the Material Safety Data Sheet (MSDS) for proper use of protective equipment and other safety information.
Protective nitrile gloves are recommended for this procedure.

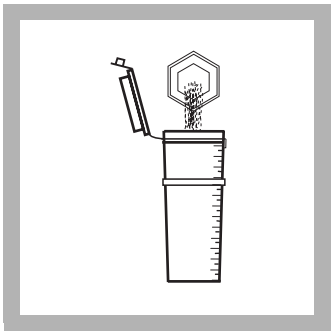
Collect the following items	Quantity
PCB Reagent Set	1
Water, deionized	varies
Caps, flip spout	1
Marker, laboratory	1
Rack, for 1-cm Micro Cuvettes	1
Wipes, disposable	1
Pipet, TenSette®, 0.1–1.0 mL and pipet tips	1
Soil Extraction Kit and Soil Scoop	1

Note: *Reorder information for consumables and replacement items is on page 9.*

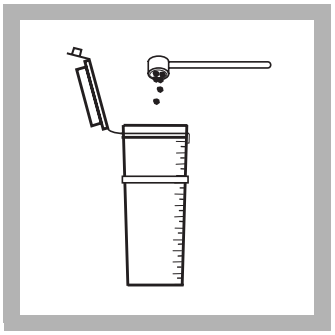
Soil Extraction Procedure



1. Weigh out 5 g of soil in the plastic weighing boat.

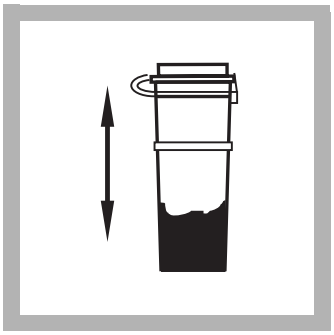


2. Carefully pour the soil into an extraction vial.



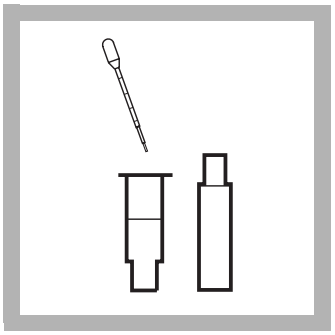
3. Use the 5-gram scoop to add one scoop of sodium sulfate to the extraction vial.

4. Use the graduated cylinder to transfer 10 mL of Soil Extractant into the extraction vial.



5. Cap the extraction vial tightly and shake vigorously for one minute.

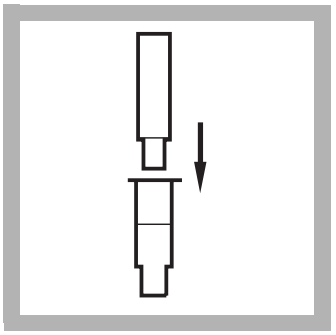
6. Allow to settle for at least one minute. Carefully open the extraction vial.



7. Using the disposable bulb pipet, withdraw 1.0–1.5 mL from the liquid layer at the top of the extraction vial.

Transfer it into the filtration barrel (the bottom part of the filtering assembly into which the plunger inserts).

Do not use more than 1.5 mL. The bulb is marked in 0.25-mL increments.

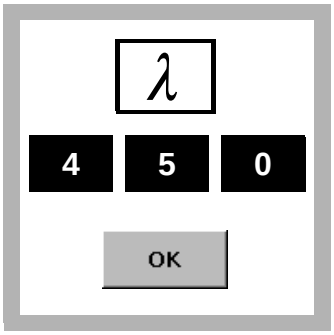


8. Insert the filtration plunger into the filtration barrel. Press firmly on the plunger until the sample extract is forced upward into the center of the plunger.

Use the resultant filtrate for the immunoassay in the Immunoassay for Soil Extracts on page 3.

It may be necessary to place the filtration assembly on a table and press down on the plunger.

Immunoassay for Soil Extracts

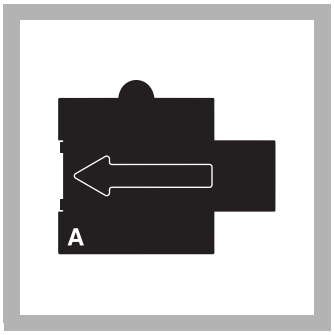


1. Press

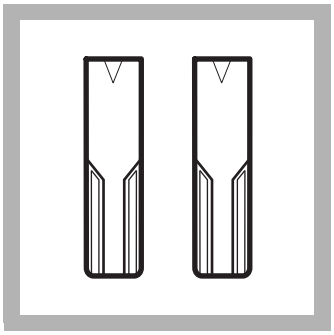
SINGLE WAVELENGTH

Press **OPTIONS** and press the λ button. Type in

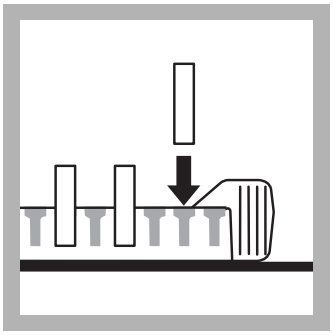
450 nm and press **OK.**



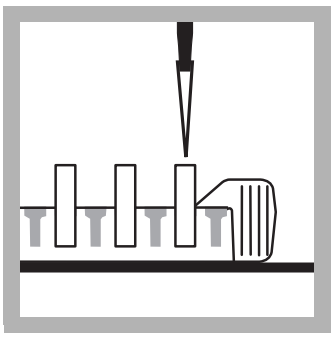
2. Insert Adapter A.



3. Label an Antibody Cuvette for each calibrator and each sample to be tested.

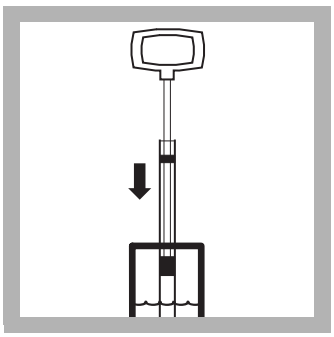


4. Insert the cuvettes into the rack snugly.



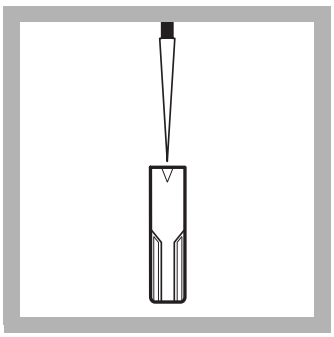
5. Pipet 0.5 mL of each Diluent Solution into the appropriately labeled cuvette.

Use a new pipette tip for each calibrator.
Important Note: *Have the necessary apparatus at hand for the next four steps; they must be done without delay.*

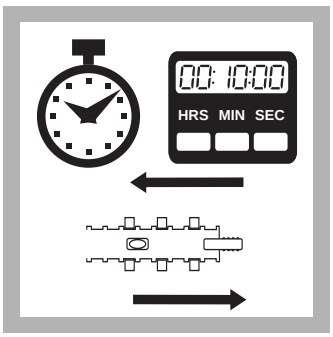


6. Use a Wiretrol® pipet to transfer the appropriate volume of calibrator or sample extract into each cuvette (see Table 1 on page 5).

Use a separate capillary tube for each solution.

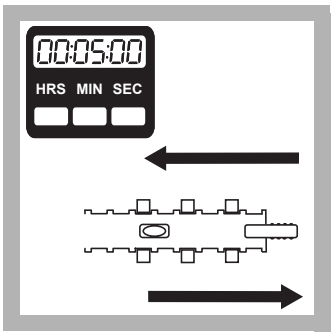


7. Immediately pipet 0.5 mL of PCB Enzyme Conjugate into each calibrator and sample cuvette. The same pipette tip can be used to add the enzyme conjugate to each cuvette.

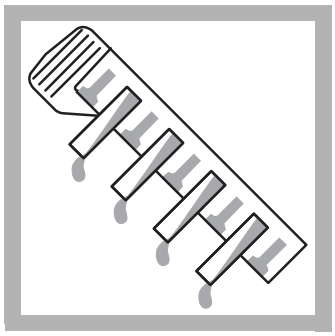


8. Press **OPTIONS**. Press **TIMER**. Enter 10:00 minutes and press **OK.**

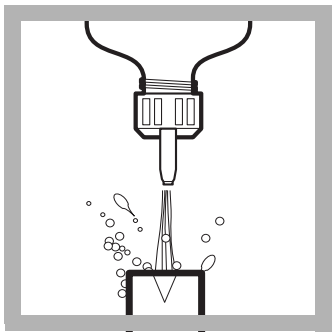
A 10-minute reaction time will begin. Immediately begin mixing the cuvettes for 30 seconds. See Using the 1-cm MicroCuvette Rack on page 6.



9. After 5 minutes mix the contents of the rack for 30 seconds (Using the 1-cm MicroCuvette Rack on page 6.)



10. At the end of the 10-minute period, discard the contents of all the cuvettes into an appropriate waste container.

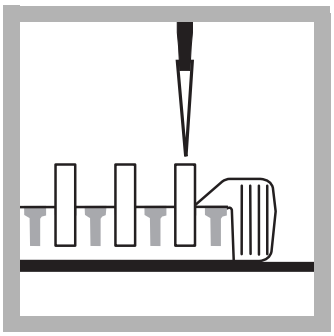


11. Wash each cuvette forcefully and thoroughly four times with deionized water. Empty the rinse water into the waste container.

Ensure that most of the water is drained from the cuvettes by turning the cuvettes upside down and tapping them lightly on a paper towel.

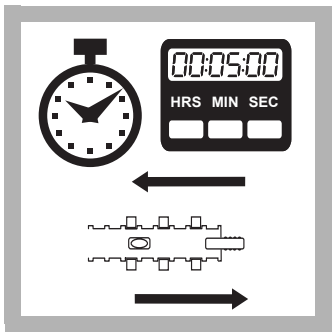
Color Development

Important Note: *Timing is critical. Follow instructions carefully.*



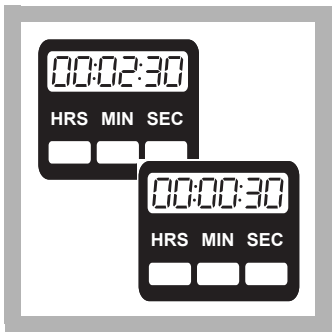
12. With the cuvettes still held snugly in the rack, pipet 0.5 mL of Color Developing Solution into each Antibody Cuvette.

Use a new pipette tip for each cuvette.

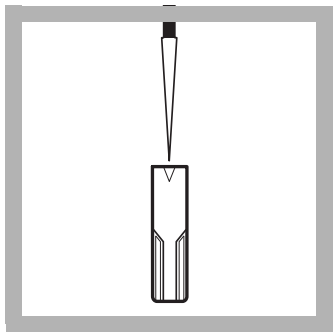


13. Press **OPTIONS**. Press **TIMER**. Enter 05:00 minutes and press **OK.**

A reaction period will begin. Mix, using the instructions in Using the 1-cm MicroCuvette Rack on page 6.



14. After 2.5 minutes, mix the contents of the rack a second time for a period of 30 seconds using the same technique.

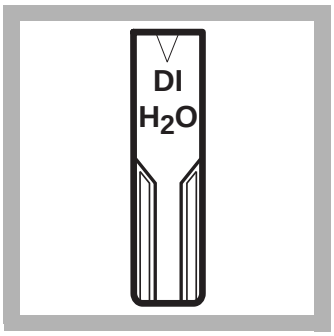


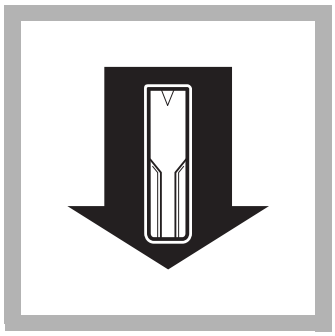
15. At the end of the 5-minute reaction period, pipette 0.5 mL of Stop Solution into each cuvette in the same order as the Color Developing Solution was added in step 12. Use the same pipette tip repeatedly for this step.

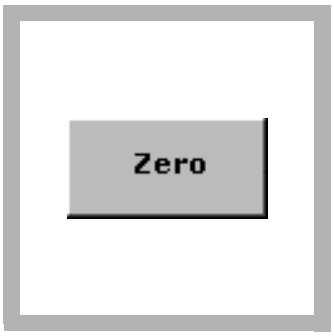
Slide the rack for 20 seconds (Using the 1-cm MicroCuvette Rack on page 6.)

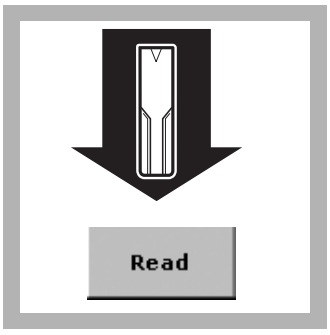
Blue solutions will turn yellow with the addition of the Stop Solution.

Measuring the Color









16. Label and fill a Zeroing Cuvette with deionized water. Wipe the outside of all the cuvettes with a tissue to remove water, smudges, and fingerprints.

17. Insert the filled zeroing cuvette into the cell holder with the arrow pointing to the right.

Orient the arrow in the same direction for all cuvettes.

18. Press **ZERO**.

The display will show:

0.000 Abs

19. Insert the first calibrator into the cell holder.

Press **READ**. The display will give an absorbance reading. Record the results for each calibrator and sample.

Repeat this step for all remaining calibrators and samples.

See Interpreting and Reporting Results on page 7 for help with interpretation of results.

PCB Protocols

There are two protocols in this procedure, one for levels of 1 ppm and 5 ppm, and another for 10 ppm and 50 ppm. Each uses a different quantity of calibrator and sample extract (Table 1).

Table 1 PCB Protocols

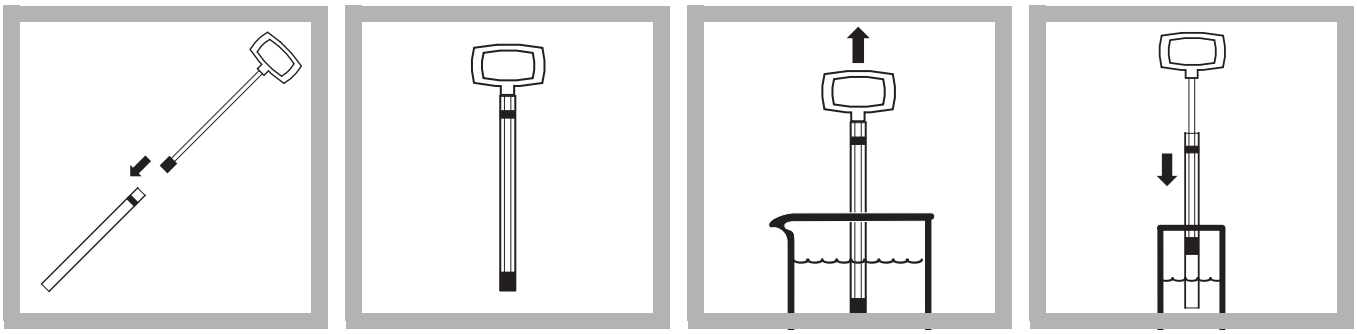
Range (as Arochlor 1248)	Volume of calibrator and sample extract used
1 ppm and 5 ppm	50 µL
10 ppm and 50 ppm	10 µL

To test across ranges, such as 1 and 50 ppm, test the lower concentration first. If the result is positive then test at the higher level. If the result of the test at the lower concentration is negative, the higher range test will be negative also, and need not be performed.

The same filtered extract can be used for both protocols if it is tightly capped between assays. The maximum time between assays cannot exceed one-half hour.

Using the Wiretrol®* Pipet

The Wiretrol Pipet can accurately measure small quantities of liquids. It consists of two parts: a Teflon®-tipped plunger and a calibrated capillary tube. The plunger can be reused; the capillary tubes must be discarded after one use.



1. Wet the orange Teflon® tip of the Wiretrol plunger in the sample and carefully insert it into the end of the capillary tube with the colored band.

2. Push the tip to the other end of the capillary tube until it barely extends beyond the end of the capillary tube.

3. Submerge the capillary tube below the surface of the liquid to be pipetted. Slowly and smoothly draw the Wiretrol plunger up until the bottom of the plunger tips reaches the appropriate volume line.

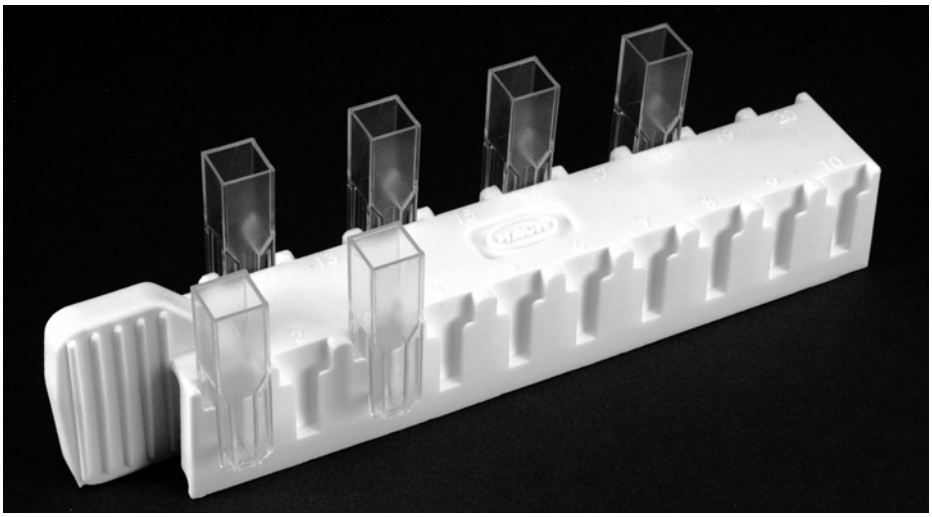
Touch the end of the tube to the side of the vessel to release remaining drops on the capillary tube tip.

4. To discharge the pipet, place the tip of the capillary tube below the surface of the solution and push the Wiretrol plunger down in one smooth motion. Change capillary tubes for each calibrator and sample.

Using the 1-cm MicroCuvette Rack

This rack (Figure 1) has been designed specifically to aid in achieving precise and accurate results when using the immunoassay technique to analyze several samples at the same time.

Figure 1 The 1-cm MicroCuvette Rack



* Wiretrol is a registered trademark of Drummond Scientific.

Loading the Rack—The cuvette rack is designed so that it may be inverted with the cuvettes in place. Identify each cuvette with a sample or calibrator number and insert all the cuvettes in the rack before beginning the procedure. Fit the cuvettes snugly into the rack, but do not force them or they may be difficult to remove and their contents may spill. The cuvettes should remain in place when the rack is inverted and tapped lightly.

Mixing—Set the rack on a hard, flat surface that is at least twice the length of the rack. Hold the rack by one end and vigorously slide it back and forth along its long axis for 30 seconds. The rack should move through a distance equal to its own length in each direction.

Interpreting and Reporting Results

There is an inverse relationship between the concentration of PCB and the reading. In other words, the higher the reading, the lower the concentration of PCB.

Table 2 Relative PCB Concentration

If the sample reading is...	the sample PCB Concentration is...
…less than calibrator reading	…greater than the calibrator concentration
…greater than calibrator reading	…less than the calibrator concentration

Example
Readings:

1 ppb PCB Calibrator: **0.775 Abs**

5 ppb PCB Calibrator: **0.430 Abs**

Sample #1: **0.200 Abs**

Sample #2: **0.600 Abs**

Sample #3: **0.900 Abs**

Interpretation for a Soil Sample

Sample #1—Sample reading is less than the readings for both calibrators. Therefore the sample concentration of PCB is greater than both 1 ppm and 5 ppm as Aroclor 1248.

Sample #2—Sample reading is between the readings for the 1 ppm and 5 ppm PCB calibrators. Therefore the sample concentration of PCB is between 1 ppm and 5 ppm as Aroclor 1248.

Sample #3—Sample reading is greater than the readings for both calibrators. Therefore the sample concentration of PCB is less than both 5 ppm and 1 ppm as Aroclor 1248.

Storing and Handling Reagents

- Wear protective gloves and eyewear.
- When storing reagent sets for extended periods of time, keep them out of direct sunlight. Store reagents at a temperature of 4 °C when not in use.
- Keep the foil pouch containing the Antibody Cuvettes sealed when not in use.
- If Stop Solution comes in contact with eyes, wash thoroughly for 15 minutes with cold water and seek immediate medical help.

Sensitivity

The PCB immunoassay cannot differentiate between the various Aroclors, but it detects their presence in differing degrees.

Table 3 Various PCBs in Soil

Compound	Concentration (ppm) to give a positive result at			
	1 ppm	5 ppm	10 ppm	50 ppm
1248	1	5	10	50
1016	2	9	20	67
1242	1.2	6	14	50
1254	1.4	4.6	11	28
1260	1.1	4.9	11	38

Table 4 Compounds Not Detectable At 1000 ppm

Biphenyl	2,4,6-trichlorophenyl	1,3-dichlorobenzene
2,4-dicholorophenyl	pentachlorophenol	1,4-dichlorobenzene
2,4,5-trichlorphenyl	1,2-dichlorobenzene	1,2,4-trichlorobenzene

Sample Collection and Storage

Analyze the samples as soon as possible after collection. If the samples must be stored, collect them in glass or Teflon® containers that have been washed with soap and water and rinsed with methanol. The container should be capped with a Teflon-lined cap. If a Teflon cap is not available, aluminum foil rinsed in methanol may be used as a substitute cap liner.

Summary of Method

Immunoassay tests use antigen/antibody reactions to test for specific organic compounds in water and soil. Antibodies specific for PCB are attached to the walls of plastic cuvettes. They selectively bind and remove PCB from complex sample matrices. A prepared sample and a reagent containing enzyme-conjugate molecules (analyte molecules attached to molecules of an enzyme) are added to the Antibody Cuvettes. During incubation, enzyme-conjugate molecules and PCB compete for binding sites on the antibodies. Samples with higher levels of analyte will have more antibody sites occupied by PCB and fewer antibody sites occupied by the enzyme-conjugate molecules.

After incubation, the sample and unbound enzyme conjugate are washed from the cuvette and a color-development reagent is added. The enzyme in the conjugate catalyzes the development of color. Therefore, there is an inverse relationship between color intensity and the amount of PCB in the sample. The resulting color is then compared with a calibrator to determine whether the PCB concentration in the sample is greater or less than the threshold levels. The PCB concentration is inversely proportional to the color development: the lighter the color, the higher the PCB concentration. Test results are measured at 450 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
PCB Reagent Set[1]	20 cuvettes	27735-00
Deionized Water	500 mL	242-49

[1] Immunoassay components are manufactured by Beacon Analytical Systems, Inc.

Required Apparatus

Description	Unit	Cat. No.
Adapter, 1-cm square cell	each	
Caps, flip spout	2/pkg	25818-02
Marker, laboratory	each	20920-00
Rack, for 1-cm Micro Cuvettes	each	48799-00
Wipes, disposable	box	20970-00
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28

Soil Extraction Reagents and Apparatus

Description	Unit	Cat. No.
Soil Scoop, 5-g, 4.25-cc	each	26572-05
Soil Extraction Refill Kit, includes:	each	27752-00
Dropper, LDPE, 0.5 and 1.0-mL	20/pkg	21247-20
Filter and Barrel Assembly	20/pkg	25676-20
Sodium Sulfate, anhydrous	250 g	7099-29
Soil Extractant Solution	200 mL	25677-29
Soil Sample Container	20/pkg	25929-20
Weighing Boat, 8.9-cm square	20/pkg	21790-20
Spatula, disposable	2/pkg	25693-20

Optional Reagents and Apparatus

Description	Cat. No.
Gloves, disposable nitrile, medium[1]	25505-02
Pipet Tips, for TenSette Pipet 19700-01	21856-96

[1] Other sizes available.

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Updated November 2005 Edition 1

Phenols

★Method 8047

4-Aminoantipyrine Method[1]

(0.002 to 0.200 mg/L)

Scope and Application: For water and wastewater; USEPA accepted (distillation required)[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

[2] Procedure is equivalent to USEPA method 420.1 for wastewater

Test Preparation

Before starting the test:

Analyze samples within four hours to avoid oxidation.
Spilled reagent affects test results and is hazardous to skin and other materials.
Use chloroform only with proper ventilation.
Phenol 2 Reagent Powder Pillows contain potassium ferricyanide. Both chloroform (D022) and cyanide (D001) solutions are regulated as hazardous waste by the Federal RCRA. **Do not pour these materials down the drain.** Chloroform solutions and the cotton plug used in the delivery tube of the separatory funnel should be collected for disposal as a reactive waste. Be sure that cyanide solutions are stored in a caustic solution with a pH >11 to prevent release of hydrogen cyanide gas. Refer to a current MSDS for safe handling and disposal information.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

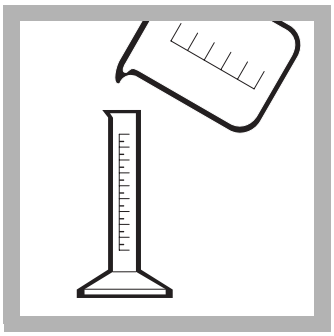
Collect the following items:	**Quantity**
Chloroform, ACS	60 mL
Clippers	1
Cotton Balls	1
Cylinder, graduated, 50-mL	1
Cylinder, graduated, 500-mL	1
Funnel, separatory, 500-mL	2
Hardness 1 Buffer Solution, pH 10.1	10 mL
Phenol 2 Reagent Powder Pillows	2
Phenol Reagent Powder Pillows	2
Pipet, volumetric, Class A, 5.00-mL	1
Ring, support, 4-inch	2
Sample Cells, 1-inch square, 25-mL, stoppered, matched pair	1
Support for Ring Stand, 5 x 8 inch base	1
Water, deionized	300 mL

Note: *Reorder information for consumables and replacement items is on page 5.*

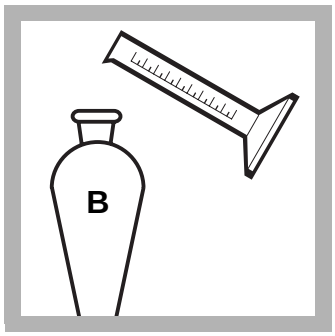
4-Aminoantipyrine Method 8047

1. Press **STORED PROGRAMS**.

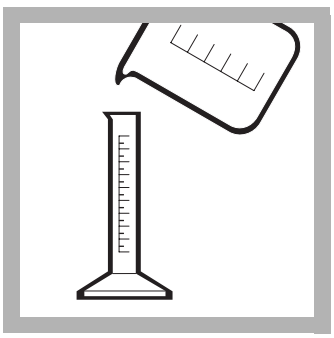
2. Select the test.



3. Measure 300 mL of deionized water in a 500-mL graduated cylinder.



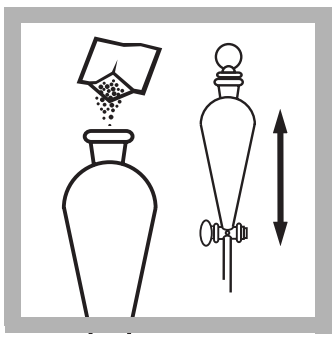
4. Blank Preparation: Pour the measured deionized water into a 500-mL separatory funnel.



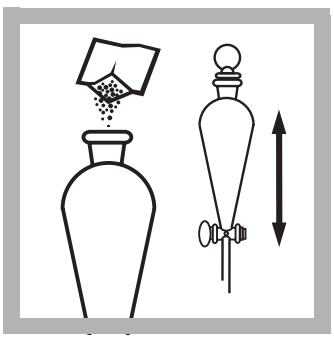
5. Measure 300 mL of sample in a 500-mL graduated cylinder.

6. Prepared Sample: Pour the measured sample into another 500-mL separatory funnel.

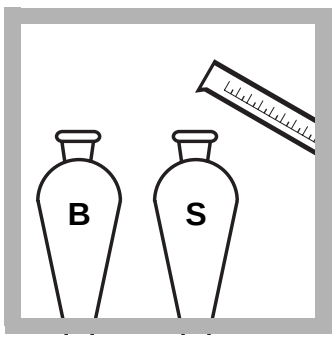
7. Add 5 mL of Hardness Buffer to each separatory funnel. Stopper and shake to mix.



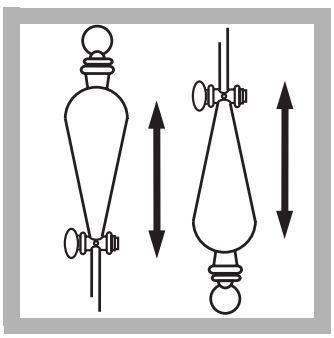
8. Add the contents of one Phenol Reagent Powder Pillow to each separatory funnel. Stopper and shake to dissolve.



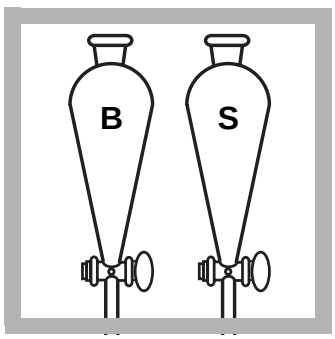
9. Add the contents of one Phenol 2 Reagent Powder Pillow to each separatory funnel. Stopper and shake to dissolve.



10. Add 30 mL of chloroform to each separatory funnel. Stopper each funnel.

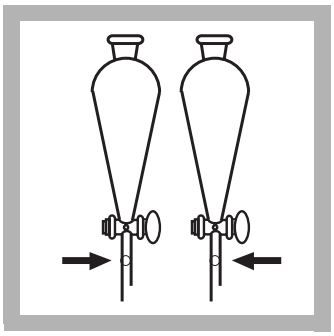


11. Invert each funnel and temporarily vent. Shake each funnel briefly and vent. Then vigorously shake each funnel for a total of 30 seconds (venting if necessary).



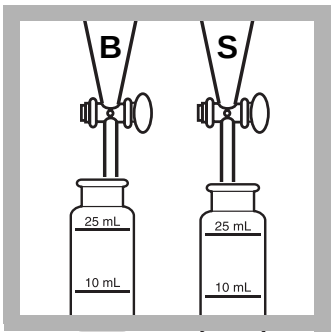
12. Remove the stoppers. Allow both funnels to stand until the chloroform settles to the bottom of the funnel.

The chloroform layer will be yellow to amber if phenol is present.



13. Insert a large, pea-sized cotton plug into the delivery tube of each funnel.

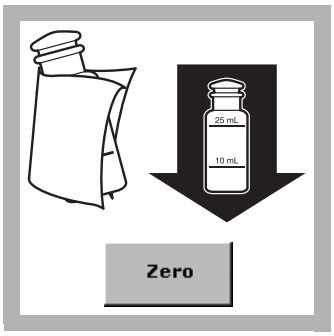
Filtering the chloroform layer through the cotton removes suspended water or particles. The volume of chloroform extract will be about 25 mL.



14. Drain the chloroform layers into separate sample cells (one for the blank and one for each sample).

Stopper the cells.

The water phase contains chloroform, which is hazardous. Dispose of properly.



15. Wipe the blank and insert it into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show: 0.000 mg/L Phenol

16. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Phenol.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
pH	The sample pH must be between 3 and 11.5 for best results.
Oxidizing or reducing agents	May interfere. Distill samples (see procedure below).
Sulfides or suspended matter	**1.** Distillation or the following pretreatment is necessary: **2.** Fill a clean 500-mL graduated cylinder with 350 mL of sample. Pour the sample into a clean 500-mL Erlenmeyer flask. **3.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow[1]. Swirl to mix. **4.** Filter 300 mL of the sample through a folded filter paper[1]. Use this solution in step 5.

[1] See Distillation Reagents and Apparatus on page 5.

Sample Collection, Storage, and Preservation

Most reliable results are obtained when samples are analyzed within four hours after collection. Use the following storage instructions only if prompt analysis is not possible. Collect 500 mL of sample in clean glass containers and add the contents of two Copper Sulfate Powder Pillows*. Adjust the pH to 4 or less with 10% Phosphoric Acid Solution*. Store at 4 °C (39 °F) or lower and analyze within 24 hours.

Accuracy Check

Standard Solution Method

For greater accuracy, analyze standard solutions when new lots of reagent are first used.

1. Weigh out 1.00 g of Phenol, ACS. Transfer to a 1000-mL volumetric flask. Dilute to the mark with freshly boiled and cooled deionized water. This is a 1000-mg/L stock solution.

* See Distillation Reagents and Apparatus on page 5.

2. Pipet 10.0 mL of the 1000-mg/L stock solution to a 1000-mL volumetric flask. Dilute to the mark with deionized water. This is a 10-mg/L working solution.
3. Prepare a 0.200-mg/L standard solution by pipetting 10.0 mL of the working solution into a 500-mL volumetric flask. Dilute to the mark with deionized water.
4. Perform the phenol procedure described above using the prepared standard.
5. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
6. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Distillation

Sample distillation as described in the following steps will eliminate interferences. The sample pH must be between 3 and 11.5 for the best results. See Interferences on page 3 for pretreatment guidelines.

1. Set up the Distillation Apparatus* by assembling the general purpose apparatus as shown in the Distillation Apparatus Manual. Use the 500-mL Erlenmeyer flask to collect the distillate. It may be necessary to use a laboratory jack to elevate the flask.
2. Place a stirring bar into the flask.
3. Measure 300 mL of water sample in a clean 500-mL graduated cylinder. Pour it into the distillation flask.
4. For proof of accuracy, use a 0.200-mg/L phenol standard (see Accuracy Check) in addition to the sample.
5. Using a serological pipet, add 1 mL of Methyl Orange Indicator* to the distillation flask.
6. Turn on the stirrer power switch. Set the stir control to 5.
7. Add 10% Phosphoric Acid Solution drop-wise until the indicator changes from yellow to orange.
8. Add the contents of one Copper Sulfate Powder Pillow* and allow to dissolve (omit this step if copper sulfate was used to preserve the sample). Cap the distillation flask.
9. Turn the water on and adjust it so a constant flow is maintained through the condenser. Set the heat control to 10.
10. Collect 275 mL of distillate in the Erlenmeyer flask, then turn the heat off.
11. Fill a 25-mL graduated cylinder to the 25-mL mark with deionized water. Add the water to the distillation flask.
12. Turn the still back on. Heat until another 25 mL of distillate is collected.
13. Using a clean graduated cylinder, re-measure the distillate to make sure 300 mL has been collected. The distillate is ready for analysis.

Summary of Method

The 4-aminoantipyrine method measures all ortho- and meta-substituted phenols. These phenols react with 4-aminoantipyrine in the presence of potassium ferricyanide to form a colored antipyrine dye. The dye is then extracted from the aqueous phase with chloroform and the color is measured at 460 nm. The sensitivity of the method varies with the type of phenolic compound. Because water samples may contain various types of phenolic compounds, the test results are expressed as the equivalent concentration of phenol.

* See Distillation Reagents and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Phenols Reagent Set (100 Tests), includes:	—	—	22439-00
(2) Chloroform, ACS	60 mL	4 L	14458-17
(3) Hardness 1 Buffer Solution, pH 10.1	10 mL	500 mL	424-49
(2) Phenol 2 Reagent Powder Pillows	2	100/pkg	1836-99
(2) Phenol Reagent Powder Pillows	2	100/pkg	872-99
Water, deionized	300 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers	1	each	968-00
Cotton Balls	1	100/pkg	2572-01
Cylinder, graduated, 50-mL	1	each	508-41
Cylinder, graduated, 500-mL	1	each	508-49
Funnel, separatory, 500-mL	2	each	520-49
Pipet Bulb, safety	1	each	14651-00
Pipet, volumetric, Class A, 5.00-mL	1	each	14515-37
Ring, support, 4-inch	2	each	580-01
Sample Cells, 1-inch square, 25-mL, matched pair	2	2/pkg	26126-02
Support for Ring Stand, 5 x 8 inch base	2	each	563-00

Distillation Reagents and Apparatus

Description	Unit	Cat. No.
Balance, lab, 300 g x 0.01 g, 100–240 V	each	28018-01
Copper Sulfate Powder Pillows	50/pkg	14818-66
Distillation Heater and Support Apparatus, 115 VAC	each	22744-00
Distillation Heater and Support Apparatus, 230 VAC	each	22744-02
Distillation Apparatus Set, general purpose	each	22653-00
Filter Paper	—	1894-57
Flask, Erlenmeyer, 500 mL	each	505-49
Funnel	each	1083-67
Methyl Orange Indicator Solution, 0.5-g/L	100 mL MDB	148-32
Phenol, ACS	113 g	758-14
Phosphoric Acid Solution, 10%	100 mL MDB	14769-32
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418-99

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Updated November 2005 Edition 1

Phosphonates

Method 8007 Persulfate UV Oxidation Method[1]

Powder Pillows (0.02 to 2.50 and 1.0 to 125.0 mg/L)

Scope and Application: For boiler and cooling water, wastewater, and seawater

[1] Adapted from Blystone, P., Larson, P., *A Rapid Method for Analysis of Phosphate Compounds*, International Water Conference, Pittsburgh, PA. (Oct 26-28, 1981)

Test Preparation

Before starting the test:

Clean glassware with 1:1 Hydrochloric Acid Solution[1], followed by a distilled water rinse. Do not clean glassware with commercial detergent.
Wear UV safety goggles while the UV lamp is on.
Do not handle the UV lamp surface. Fingerprints will etch the glass. Wipe the lamp with a soft, clean tissue between samples
The digestion in step 8 is normally completed in less than 10 minutes. However, contaminated samples or a weak lamp can cause incomplete phosphate conversion. Check conversion efficiency by running a longer digestion and seeing if the readings increase.

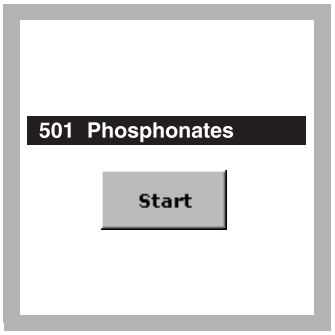
[1] See Optional Reagents and Apparatus on page 6.

Collect the following items:	Quantity
Bottle, square, with 25-mL mark	1
Cylinder, mixing, graduated, 50-mL	1
Goggles, UV safety	
Pipet, serological, 10-mL	1
PhosVer® 3 Phosphate Reagent Powder Pillows	2
Potassium Persulfate Powder Pillow for Phosphonate	1
Safety bulb	1
Sample Cells, 1-square, 10-mL	2
Water, deionized	varies
UV Lamp with Power Supply	1

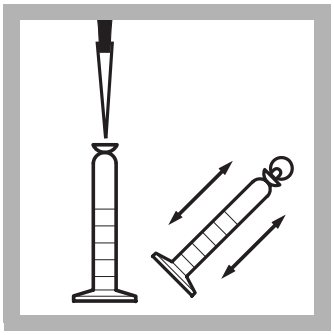
Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows Method 8007

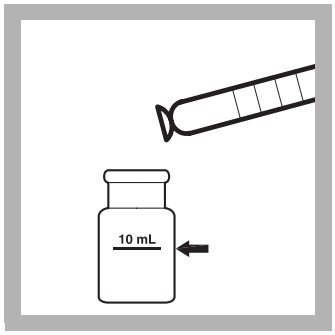
1. Press **STORED PROGRAMS**.



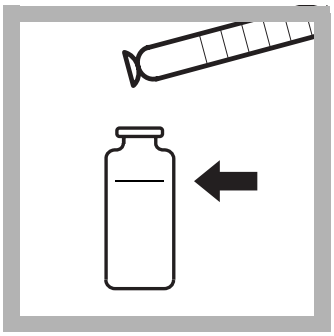
2. Select the test.



3. Choose the appropriate sample size from Table 1 on page 4. Pipet the chosen volume into a 50-mL graduated cylinder. If necessary, dilute the sample to 50-mL with deionized water and mix well.



4. Blank Preparation: Fill a square sample cell to the 10-mL mark with diluted sample from step 3.



5. Digested Sample: Fill a square sample bottle to the 25-mL mark with diluted sample from step 3.

6. Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to the bottle containing 25 mL of sample.

Swirl to dissolve the powder.

7. Insert the ultraviolet (UV) lamp into the sample bottle.

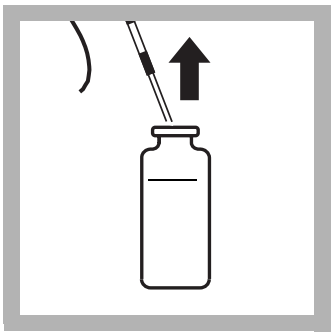
CAUTION
Wear UV safety goggles while the lamp is on.

8. Turn on the UV lamp.

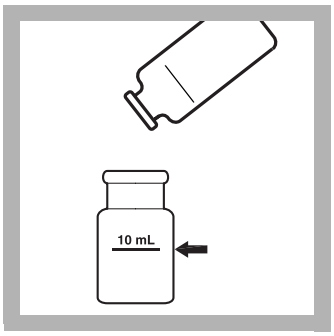
Press **TIMER>OK**.

A ten-minute reaction period will begin.

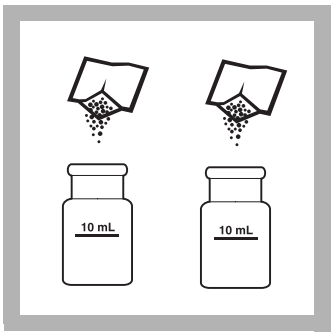
Phosphonates are converted to orthophosphate in this step.



9. When the timer expires, turn off the UV lamp and remove it from the sample.



10. Prepared Sample: Fill a second square sample cell to the 10-mL mark with the digested sample.



11. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the blank and prepared sample. Immediately swirl vigorously 20–30 seconds to mix. Some powder may not dissolve.

A blue color will develop if phosphate is present. Both sample and blank cells may develop color. The increase in sample color is proportional to the phosphonate concentration.

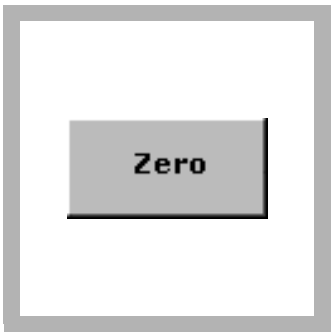
12. Press **TIMER>OK**.

A two-minute reaction period will begin.

If the sample is colder than 15 °C, allow four minutes for color development.

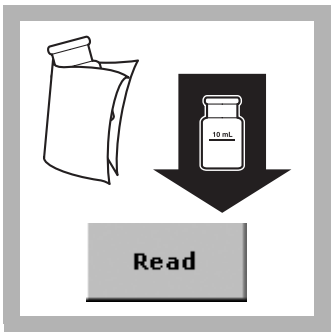
13. When the timer expires, insert the blank into the cell holder with the fill line facing right.

Complete steps 14–16 within three minutes after the timer expires.



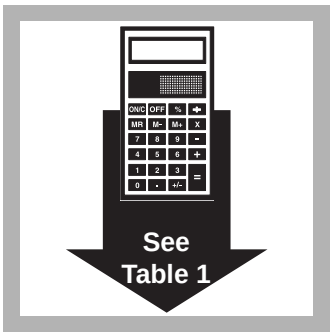
14. Press **ZERO**.

The display will show: 0.00 mg/L PO_4^{3-}



15. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L PO_4^{3-}.



16. Multiply the value in step 15 by the appropriate multiplier in Table 1 on page 4 to obtain the actual phosphonate concentration.

Table 1 Expected Ranges with Multipliers

Expected Range (mg/L phosphonate)	Sample Volume (mL)	Multiplier
0–2.5	50	0.1
0–5	25	0.2
0–12.5	10	0.5
0–25	5	1.0
0–125	1	5.0

To express results in terms of active phosphonate, multiply the final value in step 16 by the appropriate conversion factor in Table 2.

Table 2 Conversion Factors by Phosphonate Type

Phosphonate Type	Conversion Factor
PBTC	2.84
NTP	1.050
HEDPA	1.085
EDTMPA	1.148
HMDTMPA	1.295
DETPMPA	1.207
HPA	1.49
active phosphonate (mg/L) = phosphonate concentration from step 16 ↔ conversion factor	

Interferences

Table 3 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	100 mg/L
Arsenate	Interferes at all levels
Benzotriazole	10 mg/L
Bicarbonate	1000 mg/L
Bromide	100 mg/L
Calcium	5000 mg/L
CDTA	100 mg/L
Chloride	5000 mg/L
Chromate	100 mg/L
Copper	100 mg/L
Cyanide	100 mg/L (Increase the UV digestion to 30 minutes.)
Diethanoldithiocarbamate	50 mg/L
EDTA	100 mg/L
Iron	200 mg/L
Nitrate	200 mg/L
NTA	250 mg/L
Orthophosphate	15 mg/L
Phosphites and organophosphorus compounds	Reacts quantitatively. Meta- and polyphosphates do not interfere.

Table 3 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Silica	500 mg/L
Silicate	100 mg/L
Sulfate	2000 mg/L
Sulfide	Interferes at all levels
Sulfite	100 mg/L
Thiourea	10 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

The interference levels will decrease as the sample size increases. For example, copper does not interfere at or below 100 mg/L for a 5.00 mL sample. If the sample volume is increased to 10 mL, copper will begin to interfere above 50 mg/L.

Sample Collection, Storage, and Preservation

Collect samples in acid-cleaned (1:1 HCl*) plastic or glass bottles that have been rinsed with distilled water. Do not use a commercial detergent. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with Sulfuric Acid* (about 2 mL per liter). Store at 4 °C (39 °F). Preserved samples may be stored up to 24 hours. Correct the test result for volume additions.

Accuracy Check

Ideally, a solution containing the phosphonate product being used should be prepared. This will check the UV conversion of phosphonate to orthophosphate. Alternatively, a phosphate standard can be used to check the accuracy of the colorimetric part of the method.

Standard Solution

A 1-mg/L Phosphate Standard Solution can be used to check accuracy. Use 10 mL of this standard in place of the prepared sample in step 10 on page 3. Use deionized water for the blank. A multiplier value from Table 1 on page 4 is not needed. The result should be 10.0 mg/L phosphate, due to a factor of 10 in calibration.

Summary of Method

This method is directly applicable to boiler and cooling tower samples. The procedure is based on a UV-catalyzed oxidation of phosphonate to orthophosphate. The orthophosphate reacts with the molybdate in the PhosVer 3 reagent to form a mixed phosphate/molybdate complex. This complex is reduced by the ascorbic acid in the PhosVer 3, yielding a blue color that is proportional to the phosphonate present in the original sample. Test results are measured at 880 nm.

* See Optional Reagents and Apparatus on page 6.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Phosphonate Reagent Set for 10-mL sample (100 tests), includes:	—	—	24297-00
PhosVer® 3 Phosphate Reagent Powder Pillows, 10-mL	2	100/pkg	21060-69
Potassium Persulfate Powder Pillow for Phosphonate	1	100/pkg	20847-69
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Bottle, square, with 25-mL mark	1	each	17042-00
Polypropylene Beaker, 50-mL, low form, with pour spout	1	each	1080-41
Cylinder, mixing, graduated, 50-mL	1	each	1896-41
Goggles, UV safety	1	each	21134-00
Pipet, serological, graduated, 10-mL	1	each	532-38
Safety Bulb	1	each	14651-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
UV Lamp with Power Supply, 115 VAC	1	each	20828-00
OR			
UV Lamp with Power Supply, 230 VAC	1	each	20828-02

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, 1-mg/L	500 mL	2569-49

Optional Reagents and Apparatus

Description	Cat. No.
Hydrochloric Acid Solution, 1:1, 500 mL	884-49
Sulfuric Acid, 500 mL	979-49

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Updated November 2005 Edition 1

Phosphorus, Acid Hydrolyzable Digestion

★Method 8180 — Acid Digestion Method[1]

Scope and Application: For water, wastewater, and seawater; USEPA Accepted for wastewater analyses

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater* 4500-P B & E

Test Preparation

Before starting the test:

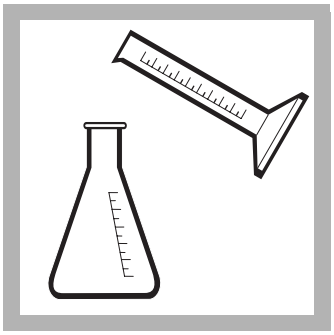
Rinse all glassware with 1:1 hydrochloric acid. Rinse again with deionized water.

The results of the reactive phosphorus test after the digestion will include the orthophosphate and the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the result of an orthophosphate test from this result. Make sure that both results are in the same units, either mg/L PO_4^{3-} or mg/L P before subtracting. The result from this test is subtracted from the result of a total phosphorus test to determine organic phosphorus.

Collect the following items:	Quantity
Sodium Hydroxide Solution, 5.0 N	2 mL
Sulfuric Acid Solution, 5.25 N	2 mL
Water, deionized	varies
Cylinder, graduated, 25-mL	1
Flask, Erlenmeyer, 125-mL	1
Hot Plate	1

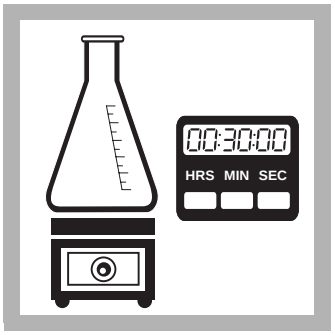
Note: *Reorder information for consumables and replacement items is on page 4.*

Acid Digestion — Method 8180



1. Use a graduated cylinder to measure 25 mL of sample. Pour the sample into a 125-mL Erlenmeyer flask.

2. Use a 1-mL calibrated dropper to add 2.0 mL of 5.25 N Sulfuric Acid Solution to the flask.

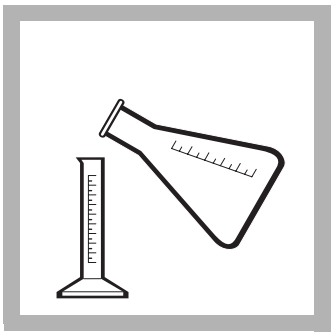


3. Place the flask on a hot plate. Boil gently for 30 minutes. Do not boil dry.

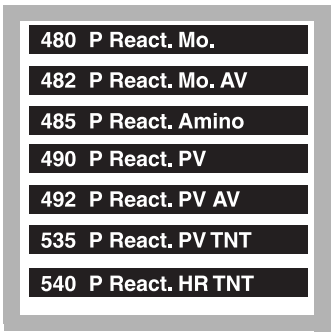
Concentrate the sample to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.

4. Cool the sample to room temperature.

5. Use a 1-mL calibrated dropper to add 2.0 mL of 5.0 N Sodium Hydroxide Solution to the flask. Swirl to mix.



6. Pour the sample into a 25-mL graduated cylinder. Adjust the volume to 25 mL with deionized water rinsings from the flask.



7. Proceed with a reactive phosphorus test of the expected acid hydrolyzable phosphorus concentration range.

Extend the color development time to 10 minutes for the PhosVer 3 method.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Alkaline or highly buffered samples	It may be necessary to add additional acid in step 2 to drop the pH of the solution below 1.
Turbidity	Use 50 mL of sample and double the reagent quantities. Use a portion of the reacted sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure.

Sample Collection, Storage, and Preservation

Analyze the samples immediately for the most reliable results. If prompt analysis is not possible, samples may be preserved up to 28 days by adjusting the pH to 2 or less with Concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N Sodium Hydroxide before analysis. Correct for volume additions.

Summary of Method

Phosphates present in condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat hydrolyzes the condensed inorganic forms to orthophosphate.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determining the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is USEPA accepted for NPDES reporting.

* See Optional Reagents on page 4.

The following reagents and apparatus are required in addition to those required for the active phosphorus test.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL MDB	2449-32
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 25-mL	1	each	508-40
Flask, Erlenmeyer, 125-mL	1	each	505-43
Hot Plate, 4 inch diameter, 120 VAC	1	each	12067-01
Hot Plate, 4 inch diameter, 240 VAC	1	each	12067-02

Required Apparatus (Field Applications)

Description	Unit	Cat. No.
Heatab Cookit, with 1 box heatabs	each	2206-00
Heatab Replacements	21/pkg	2207-00

Optional Reagents

Description	Cat. No.
Sodium, Hydroxide, 5.0 N, 1000 mL	2450-53
Sulfuric Acid, concentrated, 500 mL	979-49

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Updated November 2005 Edition 1

Phosphorus, Acid Hydrolyzable

Method 8180

Test 'N Tube™ Vials

PhosVer™ 3 with Acid Hydrolysis Method

(0.06 to 3.50 mg/L PO_4^{3-})

Scope and Application: For water, wastewater, and seawater

Test Preparation

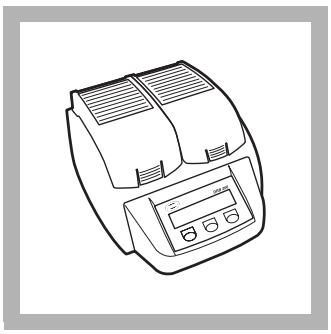
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Clean glassware with 1:1 Hydrochloric Acid Standard Solution. Rinse with deionized water. Do not use detergents that contain phosphate to clean glassware.
Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.

Collect the following items:	**Quantity**
Total and Acid Hydrolyzable Phosphorus Reagent Set	1
Deionized water	varies
DRB200 Reactor	1
Funnel, micro	1
Light Shield	1
Pipet, TenSette®, 1 to 10 mL, plus tips	
Test Tube Rack	1

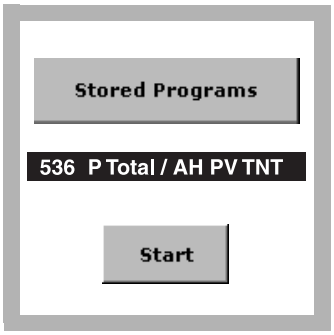
Note: *Reorder information for consumables and replacement items is on page 6.*

Test 'N Tube — Method 8180



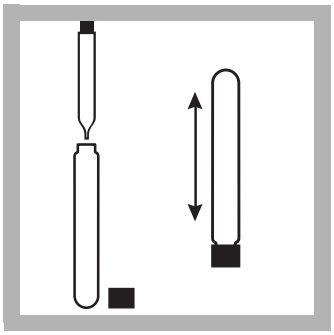
1. Turn on the DRB200 Reactor. Preheat to 150 °C.

See the DRB200 User Manual for selecting pre-programmed temperature applications.

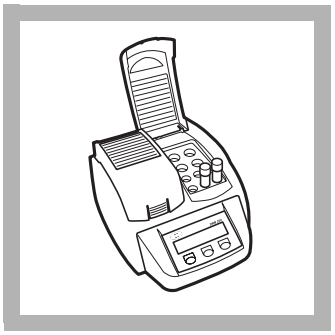


2. Select the test.

Install the Light Shield in Cell Compartment #2.



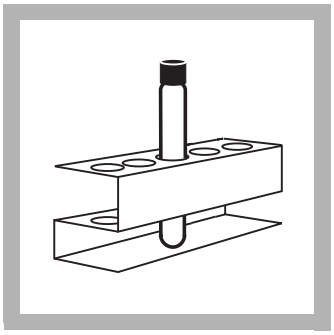
3. Use a TenSette Pipet to add 5 mL of sample to a Total and Acid Hydrolyzable Test Vial. Cap and mix.



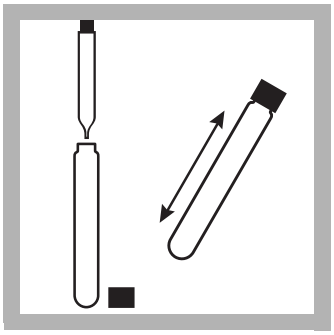
4. Insert the vial into the preheated DRB200 reactor. Close the protective cover.

5. Press **TIMER>OK**.

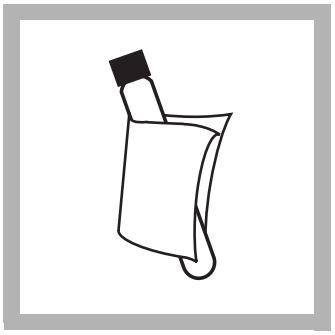
A 30-minute heating period will begin.



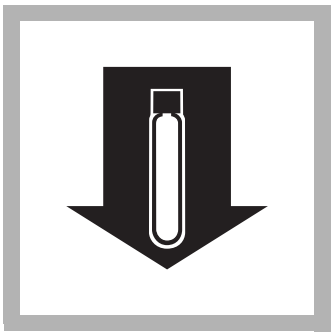
6. After the timer expires, carefully remove the vial from the reactor. Insert it in a test tube rack and cool to room temperature.



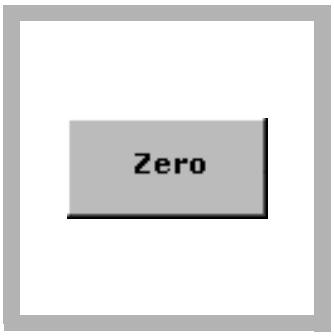
7. Using a TenSette Pipet, add 2 mL of 1.00 N sodium hydroxide to the vial. Cap tightly and shake to mix.



8. Clean the outside of the vial with a towel to remove fingerprints or other marks.



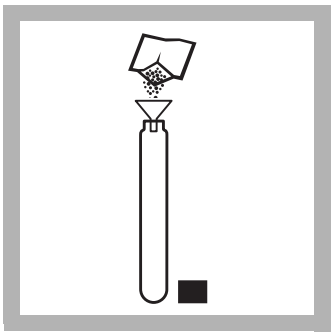
9. Insert the sample vial into the 16-mm round cell holder.



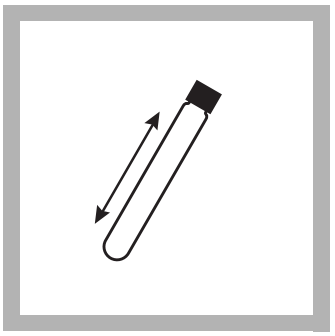
10. Press **ZERO**.

The display will show:

0.00 mg/L PO_4^{3-}



11. Using a funnel, add the contents of one PhosVer 3 Powder Pillow to the vial.

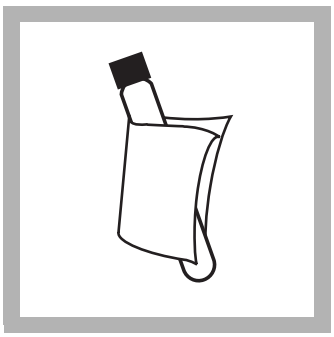


12. Immediately cap tightly and shake to mix for 10–15 seconds. The powder will not completely dissolve.

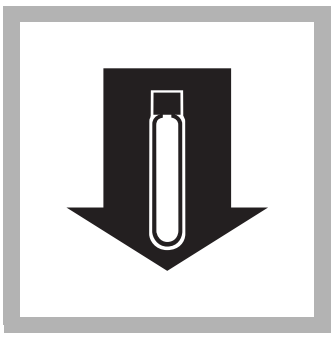
13. Press **TIMER>OK**.

A two-minute reaction period will begin.

Read results within two to eight minutes after adding the PhosVer 3 reagent.



14. Clean the outside of the vial with a towel to remove fingerprints or other marks.



15. Wipe the prepared sample and insert it into the 16-mm round cell holder.

16. Press **READ**.

Results are in mg/L PO_4^{3-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	All levels
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Sulfide	Greater than 9 mg/L. Remove sulfide interference as follows: **1.** Measure 25 mL of sample into a 50-mL beaker. **2.** Swirling constantly, add Bromine Water drop-wise until a permanent yellow color appears. **3.** Swirling constantly, add Phenol Solution drop-wise just until the yellow color disappears. Proceed with step 1.
Turbidity	Large amounts may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	Greater than 80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Sample Collection, Storage, and Preservation

Collect samples in plastic or glass bottles that have been acid washed with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing the sample at 4 °C (39 °F) for up to 48 hours.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Phosphate 2-mL Ampule Standard, 50-mg/L as PO_4^{3-}.

5. Prepare three sample spikes. Fill three Mixing Cylinders with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** and press **OK** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use a 3.0-mg/L Phosphate Standard Solution in place of the sample. Perform the procedure as described.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Phosphates present in condensed inorganic forms (meta-, pyro-, or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreating the sample with acid and heat hydrolyzes the condensed inorganic forms to orthophosphate.

Orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color. Test results are measured at 880 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Total and Acid Hydrolyzable Phosphorus Reagent Set, includes:		50 tests	27427-45
PhosVer 3 Phosphate Reagent Powder Pillows	1 pillow	50/pkg	21060-46
Potassium Persulfate Powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide, 1.54 N	varies	100 mL	27430-42
Sodium Hydroxide Standard Solution, 1.00 N	2 mL	100 mL	1045-42
Total and Acid Hydrolyzable Test Vials[1]	1 vial	50/pkg	—
Water, deionized	varies	100 mL	272-42

[1] Not sold separately.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Funnel, micro	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, volumetric, Class A, 2.00-mL	1	each	14515-36
Pipet, volumetric, Class A, 5.00-mL	1	each	14515-37
Pipet Filler, safety bulb	1	each	14651-00
Pipet, TenSette®, 1 to 10 mL	1	each	19700-10
Pipet Tips for TenSette Pipet 19700-10	1	250/pkg	21997-25
Test Tube Rack	1–3	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
Drinking Water Standard, Mixed Parameter, Inorganic for F^-, NO_3, PO_4, SO_4	500 mL	28330-49
Phosphate Standard Solution, 10-mL Voluette® Ampule, 50-mg/L as PO_4^{3-}	16/pkg	171-10
Phosphate Standard Solution, 1-mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, 3 mg/L as PO_4^{3-}	946 mL	20597-16
Wastewater Standard, Effluent Inorganics, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49

Optional Reagents

Description	Cat. No.
Bromine Water, 29 mL	2211-20
Cylinder, mixing, 25 mL	1896-40
Hydrochloric Acid Solution, 1:1, 500 mL	884-49
Phenol Solution, 29 mL	2112-20

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Updated November 2005 Edition 1

Phosphorus, Total, Digestion

★Method 8190 — Acid Persulfate Digestion Method[1]

Scope and Application: For water, wastewater, and seawater; USEPA Accepted for wastewater analyses when used with the ascorbic acid (PhosVer 3) method.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater* 4500-P B & E

Test Preparation

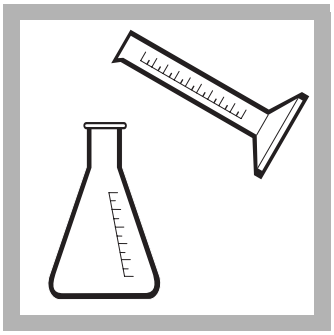
Before starting the test:

Rinse all glassware with 1:1 hydrochloric acid. Rinse again with deionized water.
The results of the reactive phosphorus test after the digestion will include the orthophosphate and the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the result of an orthophosphate test from this result. Make sure that both results are in the same units, either mg/L PO_4^{3-} or mg/L P before subtracting.

Collect the following items:	**Quantity**
Potassium Persulfate Powder Pillows	1
Sodium Hydroxide Solution, 5.0 N	2 mL
Sulfuric Acid Solution, 5.25 N	2 mL
Water, deionized	varies
Cylinder, graduated, 25-mL	1
Flask, Erlenmeyer, 125-mL	1
Hot Plate	1

Note: *Reorder information for consumables and replacement items is on page 4.*

Acid Digestion — Method 8190

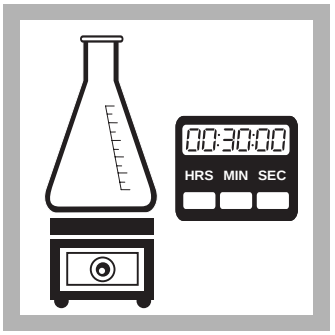


1. Use a graduated cylinder to measure 25 mL of sample. Pour the sample into a 125-mL Erlenmeyer flask.

2. Add the contents of one Potassium Persulfate Powder Pillow.

Swirl to mix.

3. Use a 1-mL calibrated dropper to add 2.0 mL of 5.25 N Sulfuric Acid Solution to the flask.

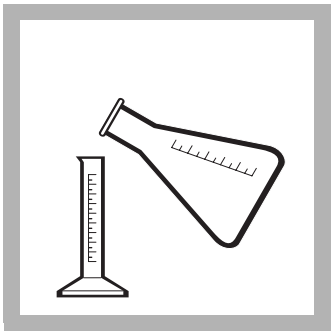


4. Place the flask on a hot plate. Boil gently for 30 minutes. Do not boil dry.

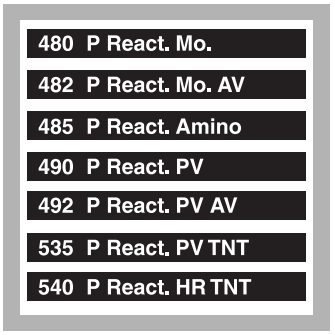
Concentrate the sample to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.

5. Cool the sample to room temperature.

6. Use a 1-mL calibrated dropper to add 2.0 mL of 5.0 N Sodium Hydroxide Solution to the flask. Swirl to mix.



7. Pour the sample into a 25-mL graduated cylinder. Adjust the volume to 25 mL with deionized water rinsings from the flask.



8. Proceed with a reactive phosphorus test of the expected total phosphorus concentration range.

Extend the color development time to 10 minutes for the PhosVer 3 (ascorbic acid) method.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Alkaline or highly buffered samples	It may be necessary to add additional acid in step 3 to drop the pH of the solution below 1.
Turbidity	Use 50 mL of sample and double the reagent quantities. Use a portion of the reacted sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure.

Sample Collection, Storage, and Preservation

Analyze the samples immediately for the most reliable results. If prompt analysis is not possible, samples may be preserved up to 28 days by adjusting the pH to 2 or less with Concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N Sodium Hydroxide before analysis. Correct for volume additions.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determining the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is USEPA accepted for NPDES reporting.

The following reagents and apparatus are required in addition to those required for the active phosphorus test.

* See Optional Reagents on page 4.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Potassium Persulfate Powder Pillows	1	100/pkg	2451-99
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL MDB	2449-32
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 25-mL	1	each	508-40
Flask, Erlenmeyer, 125-mL	1	each	505-43
Hot Plate, 4 inch diameter, 120 VAC	1	each	12067-01
Hot Plate, 4 inch diameter, 240 VAC	1	each	12067-02

Optional Reagents

Description	Unit	Cat. No.
Sodium, Hydroxide, 5.0 N	1000 mL	2450-53
Sulfuric Acid, concentrated	500 mL	979-49

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate)

Method 8178

Amino Acid Method[1]

(0.23 to 30.00 mg/L PO_4^{3-})

Scope and Application: For water, wastewater, and seawater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

Test Preparation

Before starting the test:

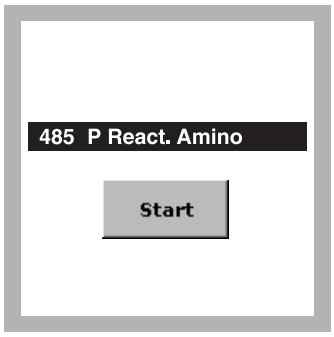
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust
The contents of one Amino Acid Reagent Powder Pillow may be substituted for 1 mL of amino acid reagent solution in step 5.

Collect the following items:	**Quantity**
Amino Acid Reagent	1 mL
Cylinder, 25-mL, graduated, mixing	1
Molybdate Reagent	1 mL
Sample Cells, 1-inch square, 10 mL, matched pair	2

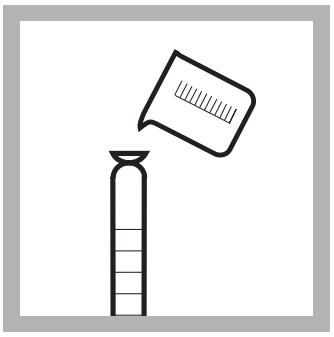
Note: *Reorder information for consumables and replacement items is on page 4.*

Amino Acid Method — Method 8178

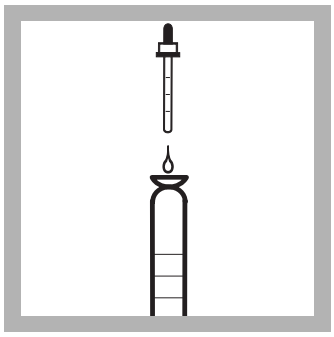
1. Press **STORED PROGRAMS**.



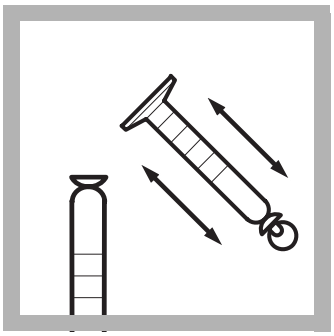
2. Select the test.



3. Fill a 25-mL mixing cylinder with 25 mL of sample.



4. Add 1 mL of Molybdate Reagent using a 1-mL calibrated dropper.

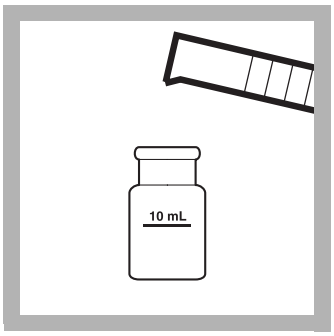


5. Prepared Sample: Add 1 mL of Amino Acid Reagent Solution. Stopper and invert several times to mix.

A blue color will form if phosphate is present.

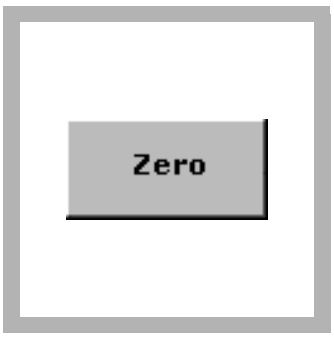
6. Press **TIMER>OK**.

A 10-minute reaction period will begin. Continue with step 7 while the timer is running.



7. Blank Preparation: Fill a square sample cell with untreated sample.

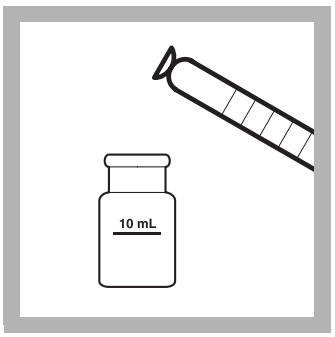
8. When the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.



9. Press **ZERO**.

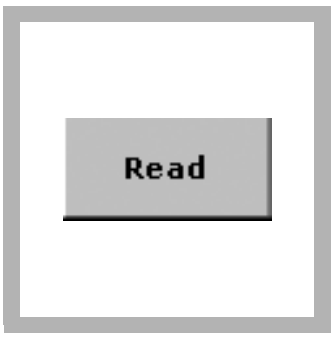
The display will show:

0.00 mg/L PO_4^{3-}



10. Fill a second square cell with prepared sample.

11. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.



12. Press **READ**.

Results are in mg/L PO_4^{3-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium (Ca^{2+})	Greater than 10,000 mg/L as $CaCO_3$
Chloride	Greater than 150,000 mg/L Cl^-
Colored samples	Add 1 mL of 10 N Sulfuric Acid Standard Solution[1] to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
High salt levels (Na^+)	May cause low results. To eliminate this interference, dilute the sample until two successive dilutions yield about the same result.
Magnesium	Greater than 40,000 mg/L as $CaCO_3$
Nitrites (NO_2^-)	Bleach the blue color. Remove nitrite interference by adding 0.05 g of sulfamic acid[1] to the sample. Swirl to mix. Continue with step 5.
Phosphates, high levels (PO_4^{3-})	As the concentration of phosphate increases, the color changes from blue to green, then to yellow and finally to brown. The brown color may suggest a concentration as high as 100,000 mg/L PO_4^{3-}. If a color other than blue is formed, dilute the sample and retest.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Sulfide (S^{2-})	Sulfide interferes. For samples with sulfide concentration less than 5 mg/L sulfide interference may be removed by oxidation with Bromine Water as follows: **1.** Measure 50 mL of sample into an Erlenmeyer flask. **2.** Add Bromine Water[1] drop-wise with constant swirling until permanent yellow color develops. **3.** Add Phenol Solution[1] drop-wise until the yellow color just disappears. Use this solution in steps 3 and 7.
Temperature	For best results, sample temperature should be 21 ±3 °C (70 ±5 °F).
Turbidity	May give inconsistent results for two reasons. Some suspended particles may dissolve because of the acid used in the test. Also, desorption of orthophosphate from particles may occur. For highly turbid samples, add 1 mL of 10 N Sulfuric Acid Standard Solution[1] to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

[1] See Optional Reagents and Apparatus on page 4.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use a commercial phosphate-based detergent for cleaning glassware because the phosphate content will contaminate the sample.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and store at 4 °C (39 °F) for up to 48 hours. The sample should have a neutral pH (6–8) and be at room temperature before analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.
2. Press **OPTIONS**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row.
4. Snap the neck off a Phosphate 2-mL Ampule Standard, 500-mg/L PO_4^{3-}.
5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 4.

Standard Solution Method

1. Use a 10-mg/L Phosphate Standard Solution. Perform the amino acid procedure as described above.
2. To adjust the calibration curve using the reading obtained with the 10-mg/L PO_4^{3-} Phosphate Standard Solution, press **OPTIONS** on the current program menu. Press **STANDARD ADJUST**.
3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

In a highly acidic solution, ammonium molybdate reacts with orthophosphate to form molybdophosphoric acid. This complex is then reduced by the amino acid reagent to yield an intensely colored molybdenum blue compound. Test results are measured at 530 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
High Range Reactive Phosphorus Reagent Set, includes:	—	100 tests	22441-00
Amino Acid Reagent	1 mL	100 mL MDB	1934-32
Molybdate Reagent	1 mL	100 mL MDB	2236-32

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, 25-mL, graduated, mixing	1	each	1896-40
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, 10-mg/L	946 mL	14204-16
Phosphate Standard Solution, 2-mL Voluette® Ampule, 500-mg/L PO_4^{3-}	16/pkg	14242-20
Wastewater Effluent Standard, for mixed parameters NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49
Wastewater Influent Standard for mixed parameters NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49
Water, deionized	4 liters	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Amino Acid Reagent Powder Pillow	100/pkg	804-99
Bromine Water	29 mL	2211-20
Flask, Erlenmeyer	each	505-43
Hydrochloric Acid Solution, 1:1	500 mL	884-49
Phenol Solution	29 mL	2112-20
Sulfamic Acid	113 g	2344-14
Sulfuric Acid Standard Solution, 10 N	1000 mL	931-53

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Updated November 2005 Edition 1

Phosphorus, Reactive

Method 8114

Pour-Thru Cell

Molybdovanadate Rapid Liquid Method[1]

HR (0.3 to 45.0 mg/L PO_4^{3-})

Scope and Application: For treated and natural waters

[1] Adapted from Standard Methods for the Examination of Water and Wastewater.

Test Preparation

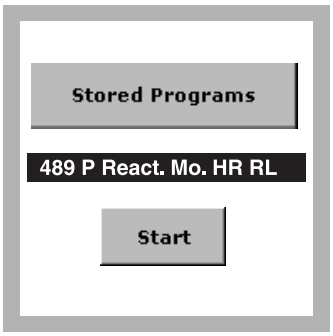
Before starting the test:

See the User Manual for Pour-Thru Module installation instructions.
Clean the Pour-Thru cell and all labware as specified in Treating Analysis Labware on page 4.
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel.
Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal information.

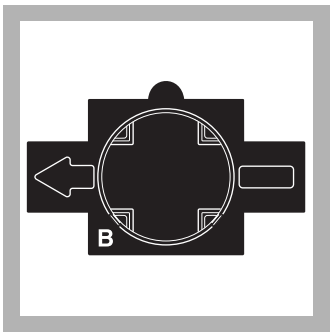
Collect the following items:	**Quantity**
Molybdovanadate Reagent	2
Water, deionized	25
Cylinder, graduated, 25-mL, poly	1
Dispenser, fixed volume, 1.0-mL, w/bottle	1
Flask, Erlenmeyer, 125-mL, PMP w/cap	2
Pour-Thru Cell Assembly Kit	1

Note: *Reorder information for consumables and replacement items is on page 5.*

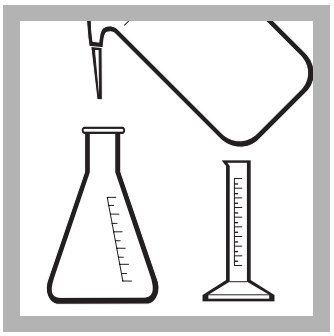
Pour-Thru Cell — Method 8114



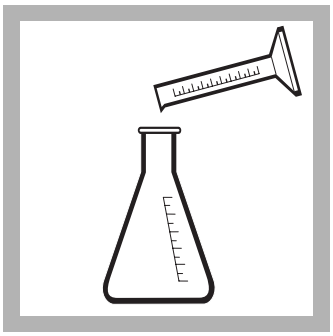
1. Select the test.



2. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch (round) path in line with the adapter arrow. Flush the Pour-Thru cell with 50 mL of deionized water.

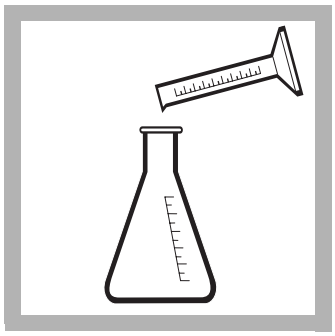


3. Rinse a clean plastic 125-mL Erlenmeyer flask and a 25-mL graduated cylinder with deionized water.



4. Blank Preparation: Measure 25 mL of deionized water in the graduated cylinder. Pour the water into the flask.

5. Rinse another clean plastic 125-mL Erlenmeyer flask with deionized water.



6. Prepared Sample: Measure 25 mL of sample in the graduated cylinder. Pour the water into the flask.

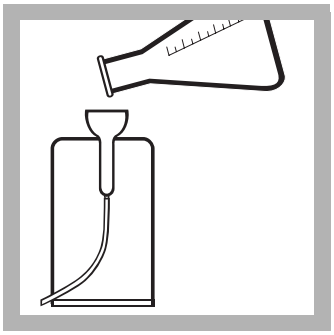
7. Add 1.0 mL of Molybdovanadate Reagent to each flask using a Repipet Jr. Dispenser. Swirl to mix.

A yellow color will develop in the sample if phosphate is present. A small amount of yellow may be present in the blank due to the reagent.

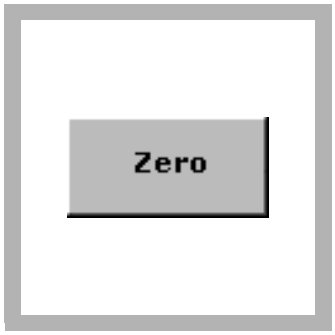
8. Press **TIMER>OK**.

A 7-minute reaction period will begin.

If the sample concentration is greater than 230 mg/L PO_4^{3-}, read at exactly seven minutes or make a 1:1 dilution of the sample and begin the test again.



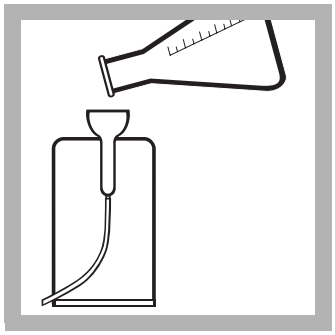
9. When the timer expires, pour the blank from the flask into the Pour-Thru Cell.



10. After the flow stops, press **ZERO**.

The display will show:

0.0 mg/L PO_4^{3-}



11. Pour the prepared sample from the flask into the Pour-Thru Cell.

Press **READ**. Results are in mg/L PO_4^{3-}.

Flush the Pour-Thru Cell with 50 mL of deionized water.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Arsenate	Negative interference. Positive interference if sample is heated.
Bismuth	Negative interference.
Fluoride	Negative interference.
Iron, Ferrous	Blue color is caused by ferrous iron but this does not affect results if the ferrous iron concentration is less than 100 mg/L.
Molybdate	Negative interference.
Silica	Positive interference if sample is heated.
Sulfide	Negative interference. Sulfide interference may be removed by oxidation with Bromine Water as follows: **1.** Measure 25 mL of sample into a flask. **2.** Add Bromine Water[1] drop-wise with constant swirling until permanent yellow color develops. **3.** Add Phenol Solution[1] drop-wise until the yellow color just disappears. Proceed with step 7.
Thiocyanate	Negative interference.
Thiosulfate	Negative interference.
Thorium	Negative interference.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

[1] See Optional Reagents and Apparatus on page 5.

Table 2 shows substances that do not interfere in concentrations less than 1000 mg/L.

Table 2 Noninterfering at Low Concentrations

Pyrophosphate	Tetraborate	Benzoate
Citrate	Lactate	Formate
Oxalate	Tartrate	Salicylate
Al^{3+}	Selenate	Mg^{2+}
Ca^{2+}	Ba^{2+}	Sr^{2+}
Li^{+}	Na^{+}	K^{+}
NH^{4+}	Cd^{2+}	Mn^{2+}
NO_3^{-}	NO_2^{-}	SO_4^{2-}
SO_3^{2-}	Pb^{2+}	Hg^{+}
Hg^{2+}	Sn^{2+}	Cu^{2+}
Ni^{2+}	Ag^{+}	U
Zr^{4+}	AsO_3^{-}	Br^{-}
CO_3^{2-}	ClO_4^{-}	CN^{-}
IO_3^{-}	Fe^{3+}	SiO_4^{4-}

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use detergents that contain phosphate for cleaning labware.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing the sample at 4 °C (39 °F) or below for up to 48 hours. Warm to room temperature before analyzing.

Treating Analysis Labware

Clean containers by normal means (do not use detergents containing phosphorus), then rinse with deionized water. Soak for several minutes in a 1:25 dilution of Molybdovanadate Reagent in deionized water. Rinse well with deionized water. Dedicate these containers for HR PO_4^{3-} analysis. Fill the Pour-Thru Cell with this same mixture of Molybdovanadate reagent and deionized water, and let stand for several minutes. Rinse with 50 mL of deionized water.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of ammonium hydroxide†, followed by several deionized water rinses. Invert a beaker over the glass funnel of the Pour-Thru Cell when not in use.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Phosphate Voluette® Ampule Standard Solution, 500-mg/L as PO_4^{3-}.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 5.
† Optional Reagents and Apparatus on page 5.

Standard Solution Method

1. Use a 10.0-mg/L Phosphate Standard in place of the sample.

2. To adjust the calibration curve using the reading obtained with the 10-mg/L PO_4^{3-} Phosphate Standard Solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration. Test results are measured at 430 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Molybdovanadate Reagent	2	500 mL	20760-49
Water, deionized	25	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 25-mL, poly	1	each	1081-40
Dispenser, fixed volume, 1.0-mL, w/bottle	1	each	21113-02
Flask, Erlenmeyer, 125-mL, PMP w/cap	2	each	20898-43
Pour-Thru Cell Module Assembly	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, 10-mg/L as PO_4^{3-}	946 mL	14204-16
Phosphate Standard Solution, Voluette® ampule, 10-mL, 500-mg/L as PO_4^{3-}	16/pkg	14242-10
Wastewater Influent Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Cat. No.
Ammonium Hydroxide, 500 mL	106-49
Bromine Water, 29 mL	2211-20
Cylinder, mixing, 25 mL	1896-40
Hydrochloric Acid Solution, 1:1, 500 mL	884-49
Phenol Solution, 29 mL	2112-20

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate)

Method 8114 **Molybdovanadate Method[1]**

Reagent Solution or AccuVac® Ampuls **(0.3 to 45.0 mg/L PO_4^{3-})**

Scope and Application: For water and wastewater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

Before starting the test:

For best results, sample temperature should be 20–25 °C (68–77 °F).
After adding reagent, a yellow color will form if phosphate is present. The blank will be slightly yellow because of the reagent.

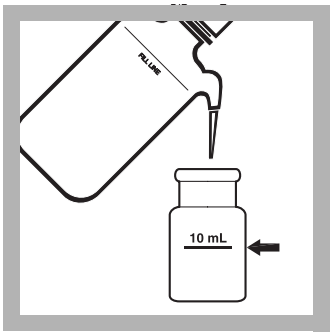
Collect the following items:	**Quantity**
Liquid Reagent Test:	
Cylinder, graduated, 25-mL	1
Molybdovanadate Reagent	2.0 mL
Sample Cells, 1-inch square, 10-mL	2
AccuVac Test:	
Molybdovanadate Reagent AccuVac® Ampuls	2
Beaker, 50-mL	2
Stopper for 18 mm Tube	2

Note: *Reorder information for consumables and replacement items is on page 6.*

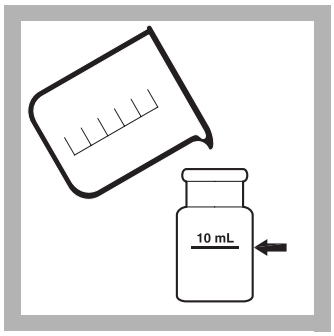
Reagent Solution Method 8114

1. Press **STORED PROGRAMS**.

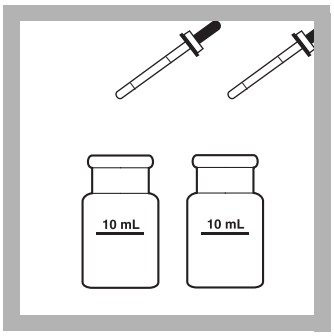
2. Select the test.



3. Blank Preparation: Fill a square sample cell with 10 mL of deionized water.



4. Prepared Sample: Fill a second square sample cell with 10 mL of sample.

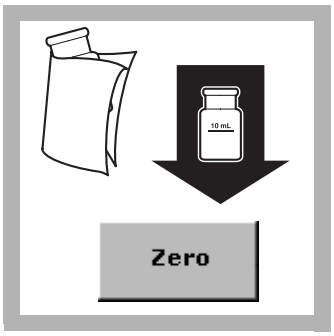


5. Add 0.5 mL of Molybdovanadate Reagent to each sample cell. Swirl to mix.

6. Press **TIMER>OK**.

A 7-minute reaction period will begin.

If the sample concentration is greater than 30 mg/L PO_4^{3-}, read at exactly seven minutes, or make a 1:1 dilution of the sample and repeat the test.



7. When the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

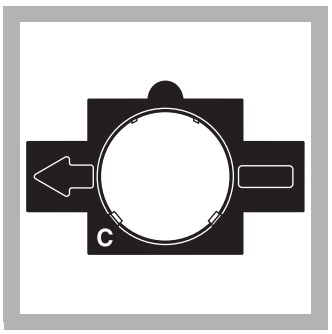
0.0 mg/L PO_4^{3-}

8. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

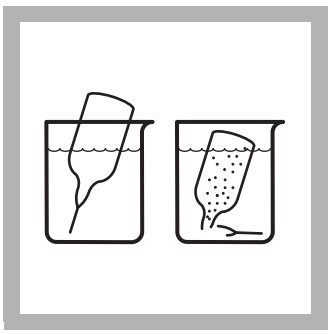
Press **READ**. Results are in mg/L PO_4^{3-}.

AccuVac Ampul — Method 8114

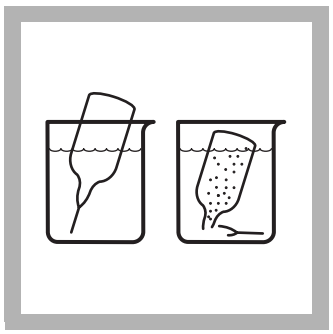
1. Select the test.



2. Insert Adapter C.



3. Prepared Sample: Collect 40 mL of sample in one 50-mL beaker. Fill a Molybdovanadate Reagent AccuVac Ampul with sample.



4. Blank Preparation: Collect 40 mL of deionized water in another 50-mL beaker.

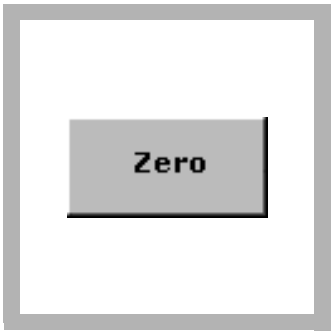
Fill another Ampul with deionized water. Keep the tips immersed while the Ampuls fill completely.

5. Press **TIMER>OK**.

A 7-minute reaction period will begin.

If the sample concentration is greater than 30 mg/L PO_4^{3-}, read at exactly seven minutes, or make a 1:1 dilution of the sample and repeat the test.

6. When the timer expires, wipe the blank and insert it into the cell holder.



7. Press **ZERO**.

The display will show:

0.0 mg/L PO_4^{3-}

8. Wipe the prepared sample and insert it into the cell holder.

Press **READ**. Results are in mg/L PO_4^{3-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Arsenate	Only interferes if sample is heated.
Iron, ferrous	Blue color caused by ferrous iron does not interfere if concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Only interferes if sample is heated.
Sulfide	Causes a negative interference. **1.** Measure 50 mL of sample into an Erlenmeyer flask. **2.** Add Bromine Water[1] drop-wise with constant swirling until a permanent yellow color develops. **3.** Add Phenol Solution[1] drop-wise until the yellow color just disappears. Proceed with step 4 (step 3 if using AccuVac procedure).
pH, extreme or highly buffered samples	May exceed buffering capacity of reagents. May require pretreatment. pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause negative interference.

[1] See Optional Reagents and Apparatus on page 6.

Table 2 shows substances that do not interfere in concentrations less than 1000 mg/L.

Table 2 Noninterfering at Low Concentrations

Pyrophosphate	Tetraborate	Benzoate
Citrate	Lactate	Formate
Oxalate	Tartrate	Salicylate
Al^{3+}	Fe^{3+}	Mg^{2+}
Ca^{2+}	Ba^{2+}	Sr^{2+}
Li^{+}	Na^{+}	K^{+}
NH^{4+}	Cd^{2+}	Mn^{2+}
NO_3^{-}	NO_2^{-}	SO_4^{2-}
SO_3^{2-}	Pb^{2+}	Hg^{+}
Hg^{2+}	Sn^{2+}	Cu^{2+}
Ni^{2+}	Ag^{+}	U^{4+}
Zr^{4+}	AsO_3^{-}	Br^{-}
CO_3^{2-}	ClO_4^{-}	CN^{-}
IO_3^{-}	SiO_4^{4-}	Selenate

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use a commercial detergent because the phosphate content will contaminate the sample.

Analyze samples as soon as possible for best results. If samples cannot be analyzed promptly, store the sample for up to 48 hours at 4 °C (39 °F) or below. Warm to room temperature before analyzing.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Phosphate 2-mL Ampule Standard, 500-mg/L PO_4^{3-}.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

* See Optional Reagents and Apparatus on page 6.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Standard Solution Method

1. Use a 10-mg/L phosphate standard solution in place of the sample. Perform the molybdovanadate procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. In the presence of vanadium, yellow molybdovanadophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration. Test results are measured at 430 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Molybdovanadate Reagent	1.0 mL	100 mL MDB	20760-32
OR			
Molybdovanadate Reagent AccuVac® Ampuls	2	25/pkg	25250-25
Water, deionized	25 mL	4 L	272-56

Required Apparatus (Liquid Reagent)

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, 25-mL	1	each	508-40
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper for 18 mm Tube	2	6/pkg	1731-06

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, 10-mg/L as PO_4^{3-}	946 mL	14204-16
Phosphate Standard Solution, 2-mL PourRite® Ampule, 500 mg/L as PO_4^{3-}	20/pkg	14242-20
Wastewater Influent Standard, for mixed parameters NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Cat. No.
Bromine Water, 29 mL	2211-20
Cylinder, mixing, 25 mL	1896-40
Hydrochloric Acid, 1:1, 500 mL	884-49
Phenol Solution, 30 g/L, 29 mL	2112-20
Stopper for 18 mm Tube, 25/pkg	1731-25

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate)

Method 8114 **Molybdovanadate Method[1]**

Test 'N Tube™ Vials **HR (1.0 to 100.0 mg/L PO_4^{3-})**

Scope and Application: For water and wastewater.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

Test Preparation

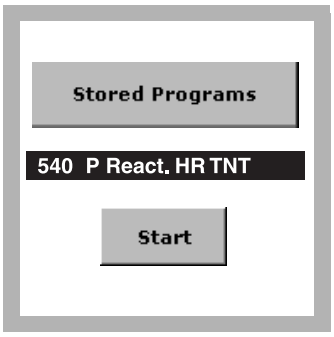
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Reagent blanks for each lot of reagents may be used more than once. At room temperature, the reagent blank is stable for up to three weeks.
The seven-minute reaction time in step 5 is for samples at 23 °C. For samples at 13 °C, wait 15 minutes. For samples at 33 °C, wait two minutes.
Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal information.

Collect the following items:	**Quantity**
High Range Reactive Phosphorus Test 'N Tube Vials	1
Water, deionized	5 mL
Dropper, LDPE, 0.5–1.0 mL	1
Light Shield	1
Pipet, TenSette®, 1 to 10 mL	1
Pipet Tips, for TenSette Pipet	1
Test Tube Rack	1–3

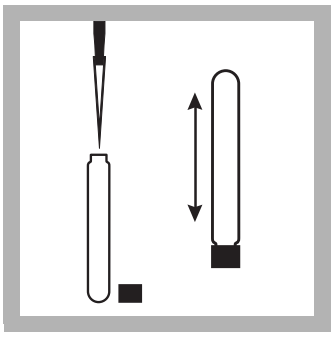
Note: *Reorder information for consumables and replacement items is on page 5.*

Test ‘N Tube Method 8114



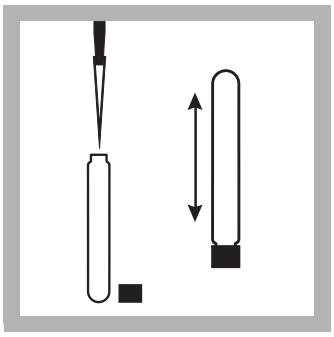
1. Select the test.

Install the Light Shield in Cell Compartment #2.



2. Blank Preparation: Use a TenSette® Pipet to add 5.0 mL of deionized water to a Reactive High Range Phosphorus Test ‘N Tube Vial.

Cap and invert to mix.



3. Prepared Sample: Use a TenSette Pipet to add 5.0 mL of sample to a Reactive High Range Phosphorus Test ‘N Tube Vial.

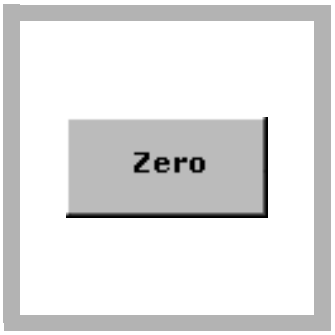
Cap and invert to mix.

4. Press **TIMER>OK**.

A seven-minute reaction period will begin.

Read the sample within two minutes after the timer expires.

5. Wipe the reagent blank and insert it into the 16-mm round cell holder.



6. Press **ZERO**.

The display will show:

0.0 mg/L PO_4^{3-}

7. Wipe the prepared sample vial and insert it into the 16-mm round cell holder.

8. Press **READ**.

Results are in mg/L PO_4^{3-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Arsenate	Only interferes if the sample is heated.[1]
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Only interferes if the sample is heated.[1]
Sulfide	Causes a negative interference. Remove interference as follows: **1.** Measure 25 mL of sample into a 50-mL beaker. **2.** Add Bromine Water[2] drop-wise with constant swirling until a permanent yellow color develops. **3.** Add Phenol Solution[2] drop-wise until the yellow color just disappears. Proceed with *step 1*.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, Cold (less than 20 °C)	Causes a negative interference.
Temperature, Hot (greater than 25 °C)	Causes a positive interference.

[1] Gentle warming of the sample to room temperature will not prevent this substance from interfering.
[2] See Optional Reagents and Apparatus on page 5.

Table 2 shows substances that do not interfere in concentrations less than 1000 mg/L.

Table 2 Noninterfering at Low Concentrations

Pyrophosphate	Tetraborate	Benzoate
Citrate	Lactate	Formate
Oxalate	Tartrate	Salicylate
Al^{3+}	Selenate	Mg^{2+}
Ca^{2+}	Ba^{2+}	Sr^{2+}
Li^{+}	Na^{+}	K^{+}
NH^{4+}	Cd^{2+}	Mn^{2+}
NO_3^{-}	NO_2^{-}	SO_4^{2-}
SO_3^{2-}	Pb^{2+}	Hg^{+}
Hg^{2+}	Sn^{2+}	Cu^{2+}
Ni^{2+}	Ag^{+}	U
Zr^{4+}	AsO_3^{-}	Br^{-}
CO_3^{2-}	ClO_4^{-}	CN^{-}
IO_3^{-}	Fe^{3+}	SiO_4^{4-}

Sample Collection, Storage, and Preservation

Collect samples in plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

For best results, analyze the samples immediately after collection. If prompt analysis is impossible, preserve the samples for up to 48 hours by filtering immediately and storing at 4 °C. The sample should be at room temperature before analysis.

* See Optional Reagents and Apparatus on page 5.

Accuracy Check

Standard Additions Method (Sample Spike)

1. Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphate to clean glassware.

2. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

3. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

4. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

5. Snap the neck off a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500-mg/L PO_4^{3-}.

6. Prepare three sample spikes. Fill three mixing cylinders with 10 mL of sample. Use the TenSette Pipet to add 0.1, 0.2 mL, and 0.3 mL of standard, respectively to each sample and mix thoroughly.

7. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **V IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use a 50-mg/L PO_4^{3-} standard in place of the sample. Perform this procedure as described.

2. To adjust the calibration curve using the reading obtained with the 50-mg/L PO_4^{3-} phosphate standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. In the presence of vanadium, yellow molybdovanadophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration. Test results are measured at 420 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
High Range Reactive Phosphorus Test 'N Tube™ Reagent Set, includes:	—	50 vials	27673-45
(1) Reactive High Range Phosphorus Test 'N Tube Vials[1]	1	50/pkg	—
(2) Water, deionized	5 mL	100 mL	272-42

[1] Not available separately.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Dropper, LDPE, 0.5–1.0 mL	1	20/pkg	21247-20
Light Shield	1	each	LZV646
Pipet, TenSette®, 1 to 10 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	1	50/pkg	21997-96
Test Tube Rack	1–3	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, 50-mg/L, as PO_4^{3-}	500 mL	171-49
Phosphate Standard Solution, Voluette® ampule, 500-mg/L as PO_4^{3-}, 10-mL	16/pkg	14242-10
Wastewater Influent Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Ammonium Hydroxide	500 mL	106-49
Bromine Water	29 mL	2211-20
Cylinder, mixing	25 mL	1896-40
Hydrochloric Acid Solution, 1:1	500 mL	884-49
Phenol Solution	29 mL	2112-20

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Updated November 2005 Edition 1

Phosphorus, Reactive

Method 10055

Pour-Thru Cell

Ascorbic Acid Rapid Liquid Method[1]

LR (19 to 3000 µg/L PO_4^{3-})

Scope and Application: For treated and natural waters

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

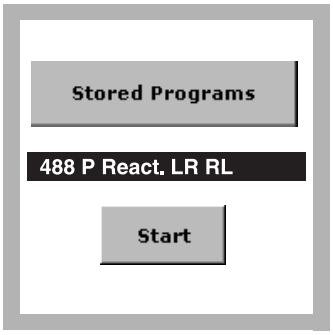
Test Preparation

Before starting the test:

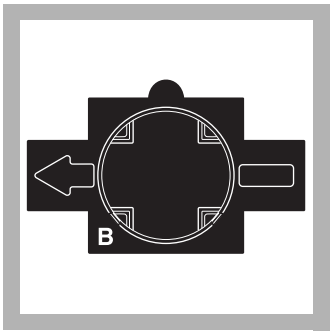
See the user manual for Pour-Thru Module installation instructions.
Clean the Pour-Thru cell and all labware as specified in Treating Analysis Labware on page 3.
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel.
See Reagent Preparation on page 3 for preparing the Ascorbic Acid reagent.
Reaction time depends on sample temperature. For most accurate results, samples should be at room temperature (about 20 °C).
Obtain a reagent blank for each lot of reagent when the normal sample phosphate concentration is less than 750 µg/L. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results.

Collect the following items:	**Quantity**
Ascorbic Acid Reagent Dilution Solution	1 mL
Ascorbic Acid Reagent Powder	varies
Cylinder, graduated, 25-mL, poly	2
Dispenser, fixed volume, 1.0-mL w/bottle	2
Flask, Erlenmeyer, 125-mL, PMP w/cap	2
Molybdate Reagent Solution	2 mL
Pour-Thru Cell Assembly	1
Water, deionized	varies

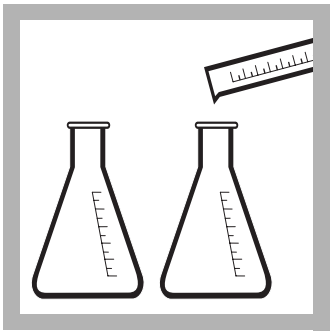
Note: *Reorder information for consumables and replacement items is on page 5.*



1. Select the test.



2. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch (round) path in line with the adapter arrow. Flush the Pour-Thru Cell with 50 mL of deionized water.

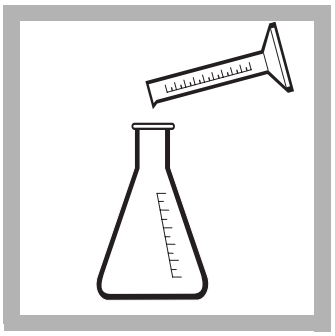


3. Rinse two clean Erlenmeyer flasks three times with the sample.

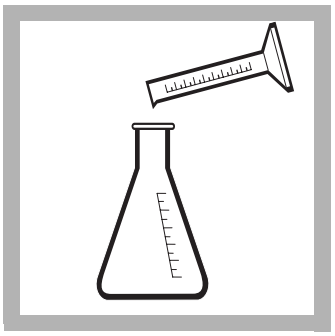
4. Rinse a clean 25-mL plastic graduated cylinder three times with the sample.

Pour-Thru Cell — Method 10055

5. Fill the rinsed cylinder to the 25-mL mark with sample.



6. Pour the contents of the 25-mL cylinder into one of the flasks.



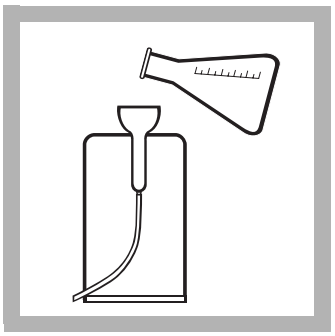
7. Measure a second 25-mL portion of sample into the graduated cylinder and pour the contents into the second flask.

8. Add 1.0 mL of Molybdate reagent to each flask using a Repipet Jr. Dispenser. Swirl to mix.

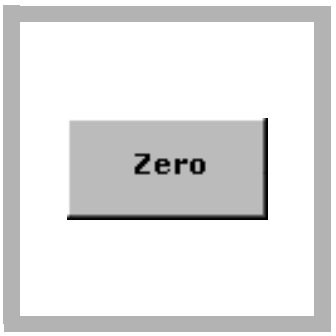
9. Prepared Sample: Add 1.0 mL of prepared Ascorbic Acid reagent to one of the flasks with a Repipet Jr. Dispenser. Swirl to mix. The remaining flask will be the blank.

10. Press **TIMER>OK**.

A five-minute reaction period will begin.



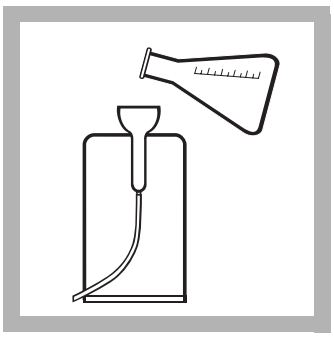
11. When the timer expires, pour the contents of the flask that contains the blank into the Pour-Thru Cell.



12. After the flow stops, press **ZERO**.

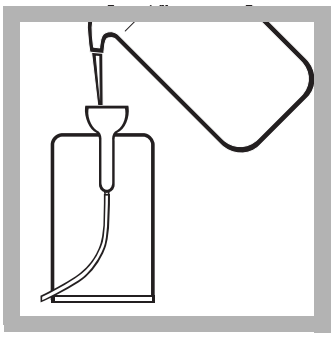
The display will show:

0 µg/L PO_4^{3-}



13. Pour the prepared sample into the Pour-Thru Cell.

Press **READ**. Results are in µg/L PO_4^{3-}.



14. Flush the Pour-Thru Cell with at least 50 mL of deionized water immediately after use.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	200 mg/L
Arsenate	Interferes
Chromium	100 mg/L
Copper	10 mg/L
Hydrogen sulfide	Interferes
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Turbidity	Samples with large amounts of turbidity may give inconsistent results because the acid present in the reagents may dissolve some of the suspended particles and variable desorption of orthophosphate from the particles may occur.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Treating Analysis Labware

All labware used in this test must be thoroughly cleaned to remove all traces of phosphate. Clean containers with a non-phosphate detergent followed by a rinse with deionized water. Fill and soak for 10 minutes with a 1:25 dilution of Molybdate Reagent in deionized water. Rinse well with deionized water. Keep containers tightly closed when not in use. Treat the Pour-Thru Cell with this same mixture of molybdate and water followed by thorough rinsing with deionized water.

Dedicate these containers for low-level phosphate analysis. If these containers are rinsed and capped after use, only occasional pre-treatment is necessary.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of Ammonium Hydroxide*, followed by several deionized water rinses. Invert a beaker over the glass funnel of the cell when not in use.

Reagent Preparation

The Ascorbic Acid reagent must be prepared before use. Using a powder funnel, add the contents of one 48 g bottle of Ascorbic Acid Reagent Powder* to one 450 mL bottle of Ascorbic Acid Reagent Dilution Solution*. Invert several times and swirl until the powder is completely dissolved. Attach Repipet Jr Dispensers to the top of this bottle and the Molybdate Reagent bottle.

This solution may develop a yellow color with time but will still give accurate results for up to one month after mixing if stored at 20–25 °C. Record the date of preparation on the bottle and discard any remaining solution after one month. Do not add fresh reagent to previously mixed reagent. Use of this reagent after one month may result in high reagent blanks and low values at high concentrations.

* See Optional Reagents and Apparatus on page 5.

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use detergents that contain phosphate for cleaning labware.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing at 4 °C (39 °F) for up to 48 hours.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Phosphate Standard Solution Ampule, either 15-mg/L (15,000 µg/L) as PO_4^{3-} or 50-mg/L (50,000 µg/L) as PO_4^{3-}. Use the 15-mg/L standard when the phosphate concentration of samples is less than 1000 µg/L. Be sure the correct standard concentration is set in step 3, above.

5. Prepare three sample spikes. Fill three Mixing Cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Run the test using a 1000-µg/L (1.000-mg/L) phosphate standard solution in place of the sample.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color. Reactive phosphorus includes existing orthophosphate in the sample plus a small fraction of condensed phosphate that may be hydrolyzed to orthophosphate during the test. Test results are measured at 880 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Rapid Liquid Low Range Phosphorus Reagent Set, includes:			26786-00
Ascorbic Acid Reagent Dilution Solution	1 mL	450 mL	25999-49
Ascorbic Acid Reagent Powder	varies	48 g	26512-55
Molybdate Reagent Solution	2 mL	500 mL	25998-49
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 25-mL, poly	2	each	1081-40
Dispenser, fixed volume, 1.0-mL w/bottle	2	each	21113-02
Flask, Erlenmeyer, 125-mL, PMP w/cap	2	each	20898-43
Pour-Thru Cell Assembly	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Drinking Water Standard, mixed Inorganics for NO_3, PO_4, SO_4	500 mL	28330-49
Phosphate Standard Solution, 1.00-mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, 3-mg/L as PO_4^{3-}	946 mL	20597-16
Phosphate Standard Solution, Voluette® ampule, 10-mL, 50-mg/L PO_4^{3-}	16/pkg	171-10
Phosphate Standard Solution, 15-mg/L PO_4^{3-}	100 mL	14243-42
Wastewater Effluent Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49

Optional Reagents and Apparatus

Description	Cat. No.
Ammonium Hydroxide, 500 mL	106-49
Cylinder, mixing, 25 mL	1896-40
Hydrochloric Acid Solution, 1:1, 500 mL	884-49

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate)

★Method 8048 **PhosVer 3 (Ascorbic Acid) Method[1]**

Powder Pillows or AccuVac® Ampuls **(0.02 to 2.50 mg/L PO_4^{3-})**

Scope and Application: For water, wastewater, and seawater; USEPA Accepted for reporting for wastewater analyses[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*

[2] Procedure is equivalent to USEPA method 365.2 and Standard Method 4500-P-E for wastewater.

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

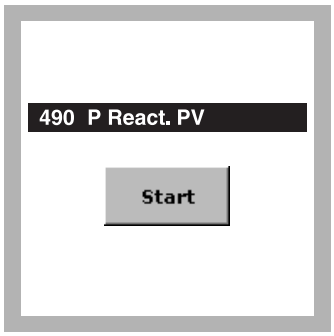
Collect the following items:	**Quantity**
Powder Pillow Test:	
PhosVer® 3 Phosphate Reagent powder pillow	1
Sample Cells, 1-in. square, 10-mL	2
Stopper for 18 mm Tube	1
AccuVac Test:	
Collect at least 40 mL of sample in a 50-mL beaker	40 mL
PhosVer® 3 Phosphate Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample Cell, 10-mL round	1
Stopper for 18-mm Tube (supplied with PhosVer AccuVacs)	1

Note: *Reorder information for consumables and replacement items is on page 6.*

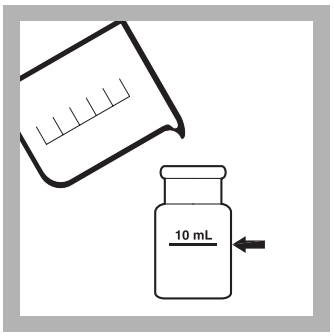
Note: *A blue color will develop if phosphorus is present.*

Powder Pillows — Method 8048

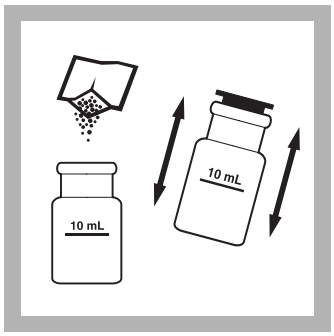
1. Press **STORED PROGRAMS**.



2. Select the test.



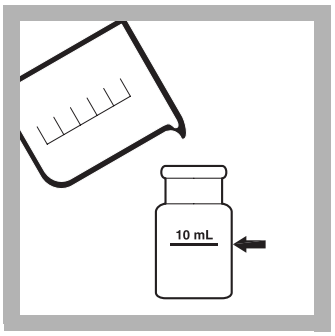
3. Fill a square sample cell with 10-mL of sample.



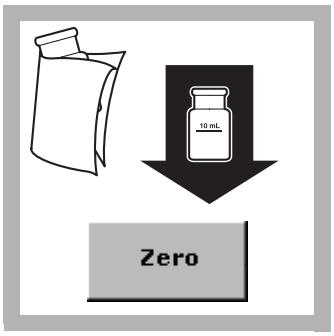
4. Prepared Sample: Add the contents of one PhosVer 3 phosphate Powder Pillow to the cell. Immediately stopper and shake vigorously for 30 seconds.

5. Press **TIMER>OK**.

A two-minute reaction period will begin. If the sample was digested using the Acid Persulfate digestion, a ten-minute reaction period is required.



6. Blank Preparation: Fill a second square sample cell with 10 mL of sample.



7. When the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.

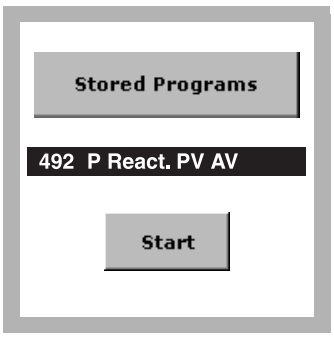
Press **ZERO**. The display will show:

0.00 mg/L PO_4^{3-}

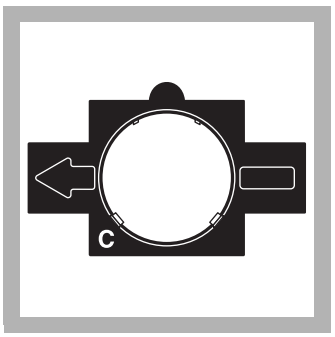
8. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L PO_4^{3-}.

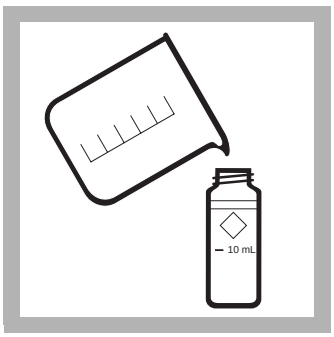
AccuVac® Ampul — Method 8048



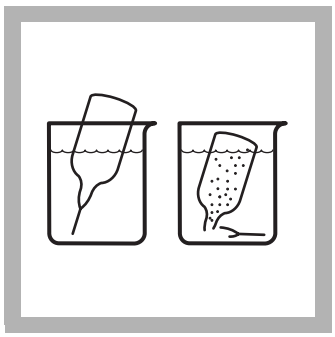
1. Select the test.



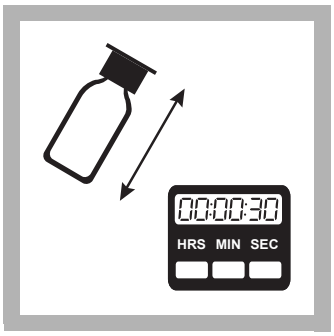
2. Insert Adapter C.



3. Blank Preparation: Fill a round sample cell with 10-mL of sample.



4. Prepared Sample: Fill a PhosVer 3 Phosphate AccuVac Ampul with sample. Keep the tip immersed while the Ampul fills completely.

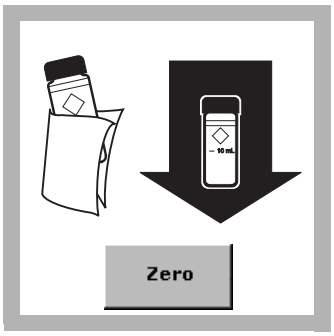


5. Secure an Ampul cap over the tip of the Ampul. Shake the Ampul for approximately 30 seconds.

Accuracy is unaffected by undissolved powder.

6. Press **TIMER>OK**.

A two-minute reaction period will begin. If the sample was digested using the Acid Persulfate digestion, a ten-minute reaction period is required.



7. When the timer expires, wipe the blank and insert it into the cell holder.

Press **ZERO**. The display will show:

0.00 mg/L PO_4^{3-}

8. Wipe the prepared sample and insert it into the cell holder.

Press **READ**. Results are in mg/L PO_4^{3-}.

Interference

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	Interferes at any level.
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Hydrogen Sulfide	Interferes at any level
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
pH, excess buffering	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment. pH 2–10 is recommended.
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Turbidity (large amounts) or color	May cause inconsistent results because the acid in the powder pillow may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles. For highly turbid or colored samples, add the contents of one Phosphate Pretreatment[1] Powder Pillow to 25 mL of sample. Mix well. Use this solution to zero the instrument.
Zinc	Greater than 80 mg/L

[1] See Optional Reagents and Apparatus on page 6.

Sample Collection, Storage, and Preservation

Collect sample in plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in phosphate analysis.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing at 4 °C (39 °F) for up to 48 hours. The sample should be at room temperature before analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a Phosphate 10-mL Ampule Standard, 50-mg/L PO_4^{3-}.

5. Prepare a 0.1-mL sample spike by adding 0.1 mL of standard to the unspiked sample. Press the timer icon. After the timer expires, read the result.

6. Prepare a 0.2-mL sample spike by adding 0.1 mL of standard to the 0.1-mL sample spike. Press the timer icon. After the timer expires, read the result.

7. Prepare a 0.3-mL sample spike by adding 0.1 mL of standard to the 0.2-mL sample spike. Press the timer icon. After the timer expires, read the result. Each addition should reflect approximately 100% recovery.

Note: *For AccuVac® Ampuls, fill three Mixing Cylinders† with 50 mL of sample and spike with 0.2 mL, 0.4 mL, and 0.6 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers*. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 6.
† See Optional Reagents and Apparatus on page 6.

Standard Solution Method

1. Prepare a 2.00 mg/L phosphate standard by pipetting 4.00 mL of 50 mg/L Phosphate Standard Solution into a 100 mL volumetric flask. Dilute to volume with demineralized water and mix. Use this solution in place of the sample, and perform the test as described above.

   (Alternately, use one of the mixed parameter standards listed in Recommended Standards on page 6. These contain 2.0 mg/L phosphate.)

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color. Test results are measured at 880 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
PhosVer® 3 Phosphate Reagent Powder Pillows, 10-mL	1	100/pkg	21060-69
OR			
PhosVer® 3 Phosphate Reagent AccuVac® Ampuls	1	25/pkg	25080-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper for 18 mm Tube	1	6/pkg	1731-06

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, 10-mL Voluette® Ampul, 50-mg/L as PO_4	16/pkg	171-10
Phosphate Standard Solution, 50-mg/L as PO_4	500 mL	171-49
Phosphate Standard Solution, 1-mg/L as PO_4	500 mL	2569-49
Standard, Drinking Water, Mixed Parameter, Inorganic: F-, NO_3, PO_4, SO_4	500 mL	28330-49
Wastewater Effluent Standard, for mixed parameters: NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL/L	28332-49
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Beaker, 50-mL	—	500-41H
Hydrochloric Acid Solution 1:1 500 mL	—	884-49
Mixing Cylinder 50 mL	—	1896-41
Phosphate Treatment Powder Pillow	—	14501-99
Stopper for 18 mm Tube	25/pkg	1731-06

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate)

★Method 8048

Test ‘N Tube™ Vials

PhosVer® 3 Method

(0.06 to 5.00 mg/L PO_4^{3-} or 0.02 to 1.60 mg/L P)

Scope and Application: For water, wastewater, and seawater; USEPA accepted for reporting wastewater analysis[1]

[1] Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P E for wastewater.

Tips and Techniques

Before starting the test:

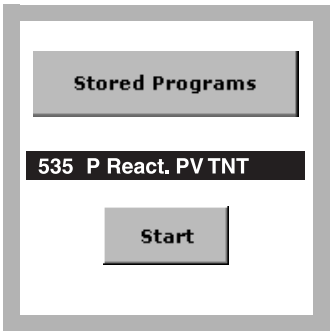
Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

Collect the following items:	Quantity
PhosVer® 3 Reagent Powder Pillow	1
Reactive Phosphorus Test ‘N Tube Vial	1
Light Shield	1
Micro funnel	1
Pipet, TenSette®, 1–10 mL	1
Pipet TIps for TenSette Pipet	1
Test Tube Rack	varies

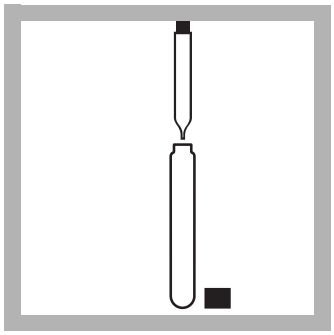
Note: *Reorder information for consumables and replacement items is on page 4.*

Test ‘N Tube — Method 8048



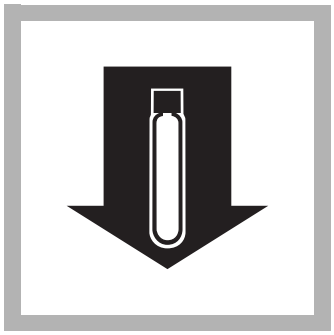
1. Select the test.

Install the Light Shield in Cell Compartment #2.

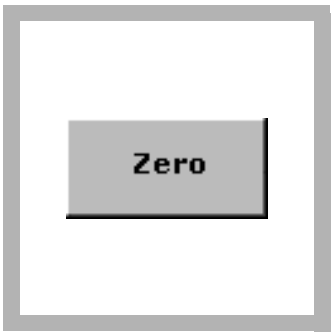


2. Use a TenSette® Pipet to add 5.0 mL of sample to a Reactive Phosphorus Test ‘N Tube Dilution Vial. Cap and mix.

3. Wipe the outside of the vial with a damp towel, followed by a dry one, to remove fingerprints or other marks.

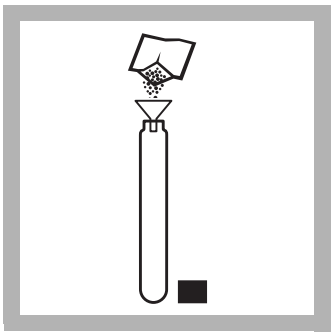


4. Insert the vial into the 16-mm round cell holder.

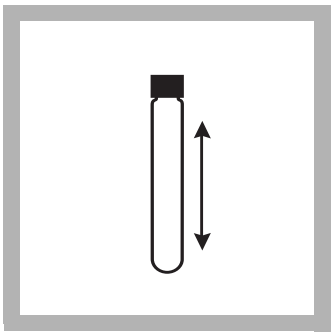


5. Press **Zero**.

The display will show: 0.00 mg/L PO_4^{3-}



6. Using a funnel, add the contents of one PhosVer 3 Phosphate Powder Pillow to the vial.

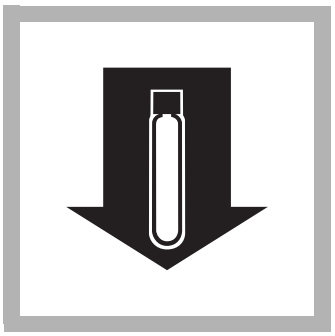


7. Immediately cap the vial tightly and shake for at least 20 seconds. The powder will not dissolve completely.

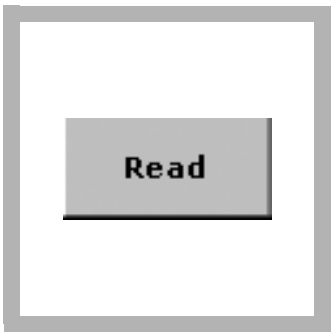
8. Press **TIMER>OK**.

A two-minute reaction period will begin. Read samples between two and eight minutes after adding the PhosVer 3 reagent.

9. Wipe the outside of the vial with a damp towel, followed by a dry one, to remove fingerprints or other marks.



10. When the timer expires, insert the vial into the 16 mm round cell holder.



11. Press **READ**.

Results are in mg/L PO_4^{3-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	All levels
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Sulfide	Greater than 6 mg/L. Remove sulfide interference as follows: **1.** Measure 25 mL of sample into a 50-mL beaker. **2.** Swirling constantly, add Bromine Water drop-wise until a permanent yellow color appears. **3.** Swirling constantly, add Phenol Solution drop-wise just until the yellow color disappears. Proceed with *step 1* of the phosphorus procedure.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments (continued)
Turbidity	Large amounts may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	Greater than 80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Sample Collection, Storage, and Preservation

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 48 hours by filtering immediately and storing at 4 °C. Warm samples to room temperature before analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. Clean glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. Do not use phosphate detergents to clean glassware.
2. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.
3. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
4. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
5. Snap the neck off a Phosphate 2-mL Ampule Standard, 50-mg/L as PO_4^{3-}.
6. Prepare three sample spikes. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to three 25-mL samples and mix each thoroughly.
7. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use a 3.0-mg/L phosphate standard solution instead of the sample. Perform the procedure as describe above.
2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

* See Optional Reagents and Apparatus on page 4.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color. Test results are measured at 880 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Reactive Phosphorus Test 'N Tube™ Reagent Set (50 tests), includes:	—	—	27425-45
PhosVer® 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Reactive Phosphorus Test 'N Tube Dilution Vials	1	50/pkg	NA[1]

[1] Not available separately

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Funnel, micro	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 1 to 10 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	1	50/pkg	21997-96
Test Tube Rack	1–3	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, Voluette™ Ampule, 50-mg/L as PO_4^{3-}, 2-mL	20/pkg	17120-H
Phosphate Standard Solution, 50-mg/L	500 mL	171-49
Phosphate Standard Solution, 1-mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, 3 mg/L as PO_4^{3-}	946 mL	20597-16
Standard, Drinking Water, Mixed Parameter, Inorganic for F^-, NO_3, PO_4, SO_4	500 mL	28330-49
Wastewater Effluent Standard, for mixed parameters: NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bromine Water 30 g/L	25 mL	2211-20
Hydrochloric Acid Solution 1:1	500 mL	884-49
Phenol Solution 30 g/L	29 mL	2112-20

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate) and Total

Method 10209 Reactive; Method 10210 Total

Ascorbic Acid Method

TNTplus 843

LR (0.15–4.50 mg/L PO_4^{3-} or 0.05–1.50 mg/L PO_4–P)

Scope and Application: For wastewater, drinking water, boiler water, surface water, and process water

Test Preparation

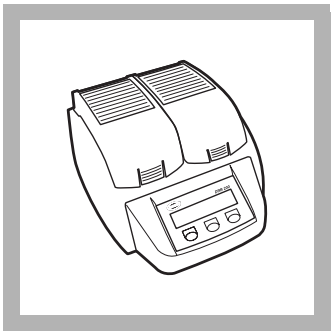
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 2–10.
The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.
TNT plus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

Collect the following items:	**Quantity**
Phosphorus, Reactive and Total LR TNT843 Reagent Set	1
DRB Reactor for use with 13 mm wells (use adapters with 16 mm holes)	1
Light Shield	1
Pipettor for 100–1000 µL Sample	1
Pipettor Tips for 100–1000 µL Pipettor	1
Pipettor for 5.0 mL Sample	1
Pipettor Tip	varies
Test Tube Tack	1

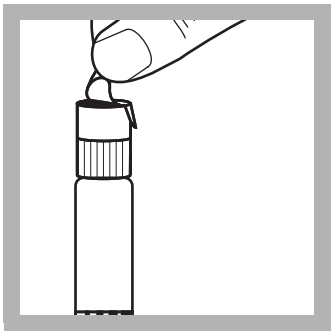
Note: *Reorder information for consumables and replacement items is on page 6.*

TNTplus—Phosphorus, Total Method 10210

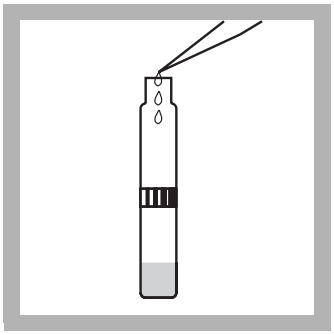


1. Turn on the DRB200 Reactor. Heat to 100 °C.

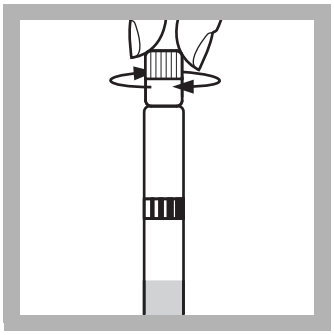
Note: *For DRB200 Reactors with 16-mm wells, insert a16-mm to 13-mm adapter sleeve into each well before turning on the reactor.*



2. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



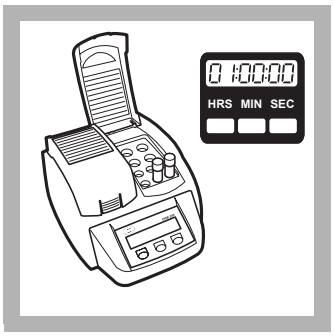
3. Carefully pipet 2.0 mL of sample into the vial.



4. Flip the DosiCap ***Zip*** over so the reagent side faces the vial. Screw the cap tightly onto the vial.

5. Shake the capped vial with 2–3 times to dissolve the reagent in the cap.

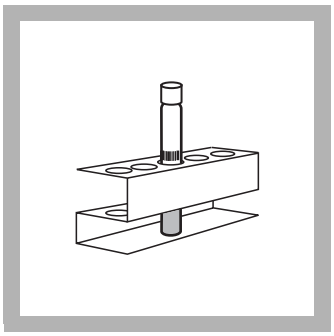
Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.



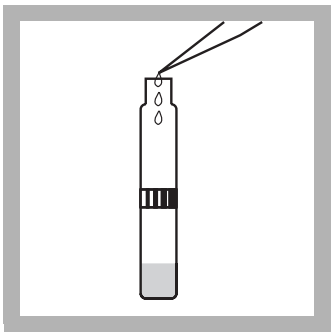
6. Insert the vial in the DRB200 Reactor.

Close the protective cover.

Heat for 1 hour at 100 °C.

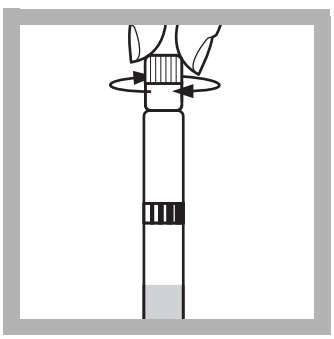


7. After the timer expires, carefully remove the hot vial from the reactor. Insert them in a test tube rack and allow to cool to room temperature (15–25 °C).

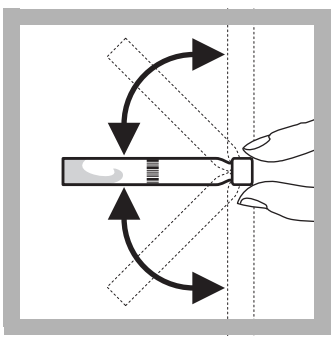


8. Pipet 0.2 mL (200 µL) of Reagent B into the cooled vial.

Immediately close the Reagent B container.

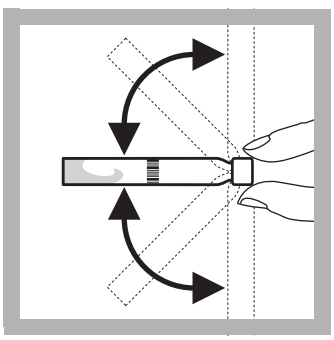


9. Screw a grey DosiCap C onto the vial.

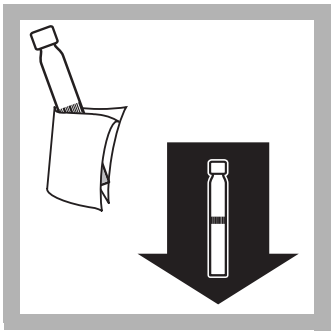


10. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

11. Wait 10 minutes. Install the Light Shield in Cell Compartment #2.



12. When the timer expires, invert the vial again 2–3 times.

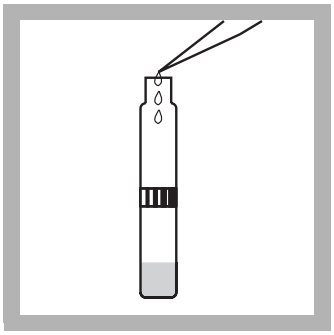


13. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

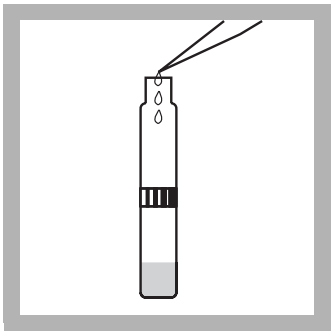
Results are in mg/L PO_4.

No instrument Zero is required.

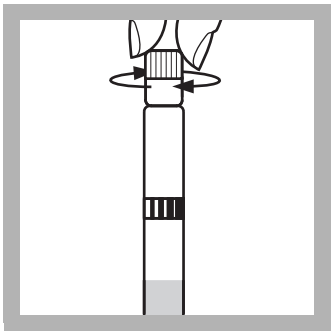
TNTplus—Phosphorus, Reactive (Orthophosphate Method 10209



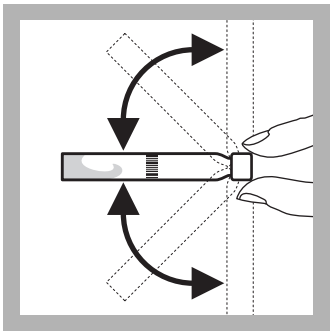
1. Carefully pipet 2.0 mL of sample into the vial.



2. Pipet 0.2 mL (200 µL) of Reagent B into the vial. ***Note:*** *Immediately close the Reagent B container.*



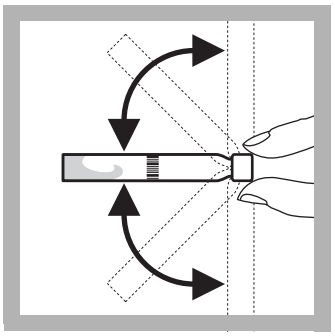
3. Screw a grey DosiCap C onto the vial.



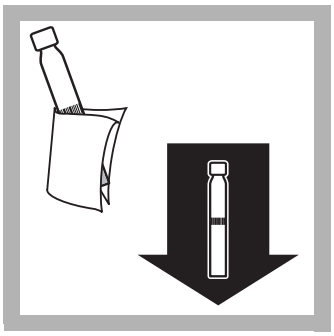
4. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

5. Wait 10 minutes.

Install the Light Shield in Cell Compartment #2.



6. When the timer expires, invert the vial again 2–3 times.



7. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4.

No instrument Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 13 of the total phosphorus procedure or step 7 of the reactive phosphorus procedure. Press **OPTIONS**>**MORE**>**REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Color or turbidity in samples can cause high results. The digestion in the total phosphate procedure usually destroys all color and turbidity and a sample blank is not required.

To compensate for color or turbidity in the reactive phosphate procedure the color forming reagent that is present in the DosiCap C is not added.

To determine the sample blank run the reactive procedure as given, but do not add the DosiCap C in step 3. Cap the vial with the original DosiCap ***Zip*** (do not remove the foil). Use the side of the cap without the reagent. The value obtained in step 7 is then subtracted from the value obtained on the original reactive phosphate sample to give the corrected sample concentration.

Alternatively, reactive phosphate samples that contain only turbidity may be first filtered through a membrane filter and then analyzed. Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined. Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
SO_4^{2-}	5000 mg/L
Cl^-	2000 mg/L
K^+, Na^+	1000 mg/L
NO_3^-	500 mg/L
Ca^{2+}	250 mg/L
Mg^{2+}	100 mg/L
CO_3^{2-}, Fe^{2+}, Fe^{3+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, NH_4^+. Mn^{2+}, Al^{3+}, CO_3^{2-}, SiO_2	50 mg/L
Sn^{4+}, Hg^{2+}	5 mg/L
Ag^+, Pb^{2+}	2.5 mg/L
Cr^{3+}	1 mg/L
Cr^{8+}	0.5 mg/L

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for total phosphorus up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C. Samples to be analyzed for reactive phosphorus should not be preserved with acid. These samples should be stored at 4 °C and analyzed within 48 hours. Warm samples to 15–25 °C and neutralize with 5.0 N Sodium Hydroxide* before analysis if acid has been added. Correct for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the reactive and total phosphorus methods with a 3 mg/L phosphate standard solution. Use 2.0 mL this 3 mg/L standard in place of the sample in step 3 of the total phosphorus procedure or step 2 of the reactive phosphorus procedure.
2. Alternately, use 2.0 mL of a Wastewater Effluent Mixed Parameters Inorganics Standard in place of the sample in step 2. This standard contains 2 mg/L phosphate in the presence of several other ions such as nitrate, sulfate and ammonia.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) are first converted to reactive orthophosphate in the total phosphorus procedure. Treatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are also converted to orthophosphates in the total phosphorus procedure by heating with acid and persulfate. The reactive phosphorus procedure measures only the reactive (ortho) phosphorus present in the sample.

The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue. Test results are measured at 890 nm.

* See Optional Reagents and Apparatus on page 6.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Phosphorus, Reactive and Total, LR TNT843 Reagent Set	1	25/pkg	TNT843

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27950-00
Pipettor, variable volume, 1–5 mL	1	each	27951-00
Pipettor Tips, for 27951-00 pipettor	1	400/pkg	27952-00
Test Tube Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Phosphate Standard Solution, 3-mg/L as PO_4^{3-}	946 mL	20597-16
Wastewater Effluent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB200-03
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 15x13mm + 15x13 mm (dual block)	each	DRB200-07
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-08
Filter Holder, glass for vacuum filtration (SUVA)	each	2340-00
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	each	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	each	546-53
Hydrochloric Acid 6N (1:1)	500 mL	884-49
Sodium Hydroxide, 5.0 N, 1000 mL	1000 mL	2450-53
Sulfuric Acid, concentrated, 500 mL	500 mL	979-49
Tubing, rubber	12 ft	560-19

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate) and Total

Method 10209 Reactive; Method 10210 Total **Ascorbic Acid Method**

TNTplus™ 844

HR (1.5 to 15.0 mg/L PO_4^{3-} or 0.5 to 5.0 mg/L PO_4–P)

Scope and Application: For wastewater, drinking water, boiler water, surface water, and process analysis

Test Preparation

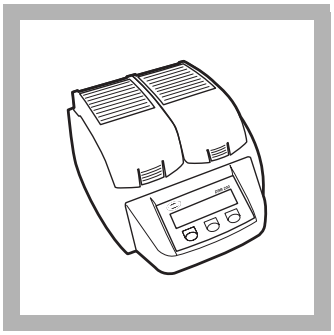
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 2–10.
The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.
TNT plus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.

Collect the following items:	**Quantity**
Phosphorus, Reactive Total HR TNT844 Reagent Set	1
DRB Reactor for use with 13-mm wells (use adapters with 16-mm holes)	1
Light Shield	1
Pipettor for 100–1000 µL Sample	1
Pipettor Tips for 100–1000 µL Pipettor	1
Test Tube Rack	2

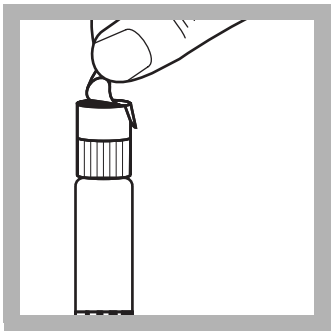
Note: *Reorder information for consumables and replacement items is on page 7.*

TNTplus—Phosphorus, Total Method 10210

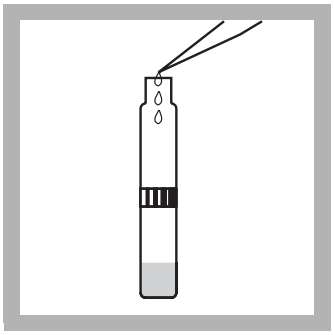


1. Turn on the DRB200 Reactor Heat to 100 °C.

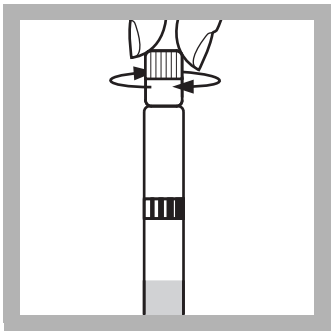
Note: *For DRB200 Reactors with 16-mm wells, insert a 16-mm to 13-mm adapter sleeve into each well before turning on the reactor.*



2. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



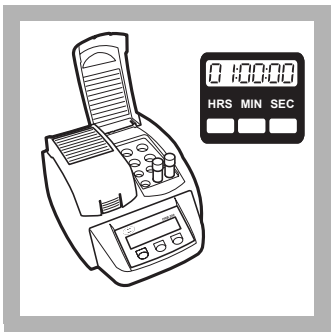
3. Carefully pipet 0.5 mL (500 µL) of sample into the vial.



4. Flip the DosiCap ***Zip*** over so the reagent side faces the vial. Screw the cap tightly onto the vial.

5. Shake the vial 2–3 times to dissolve the reagent in the cap.

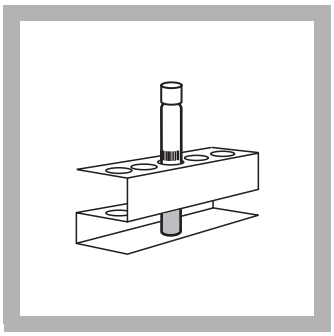
Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.



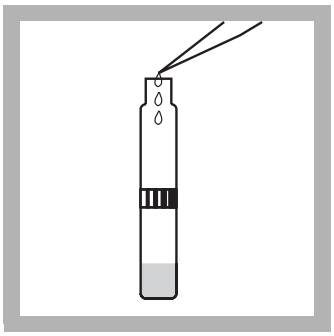
6. Insert the vial in the DRB200 Reactor.

Close the protective cover.

Heat for 1 hour at 100 °C.

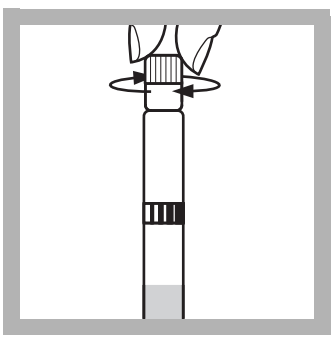


7. After the timer expires, carefully remove the hot vial from the reactor. Insert them in a test tube rack and allow to cool to room temperature (15–25 °C).

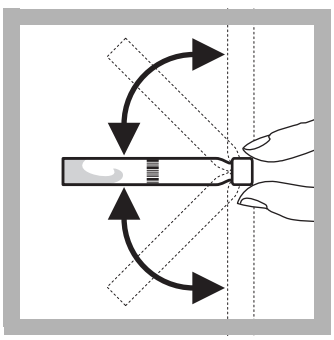


8. Pipet 0.2 mL (200 µL) of Reagent B into the cooled vial.

Immediately close the Reagent B container.

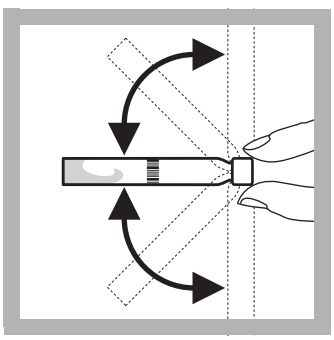


9. Screw a gray DosiCap C onto the vial.

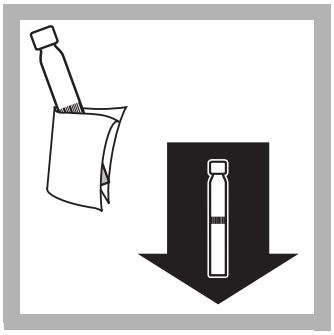


10. Invert the vial 2–3 times to dissolve the reagent in the DosiCap.

11. Wait 10 minutes. Install the Light Shield in Cell Compartment #2.



12. When the timer expires, invert the vial again 2–3 times.

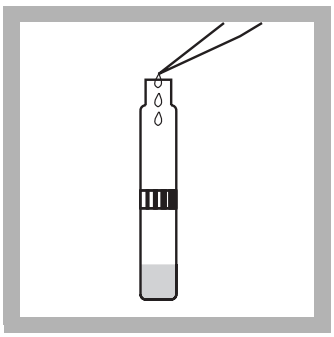


13. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

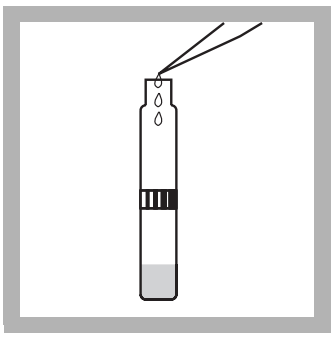
Results are in mg/L PO_4.

No instrument Zero is required.

TNTplus—Phosphorus, Reactive (Orthophosphate Method 10209

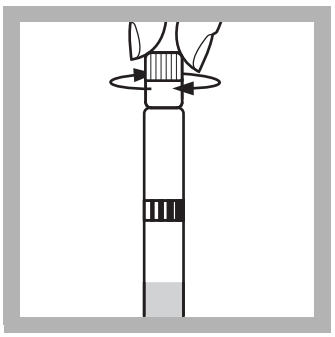


1. Carefully pipet 0.5 mL (500 µL) of sample into the vial.

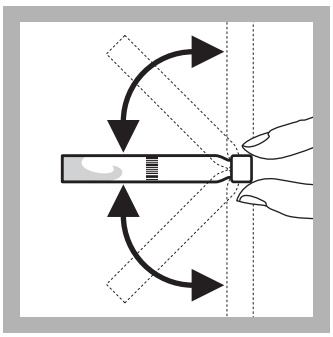


2. Pipet 0.2 mL (200 µL) of Reagent B into the vial.

Immediately close the Reagent B container.



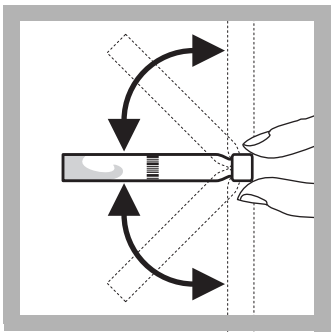
3. Screw a grey DosiCap C onto the vial.



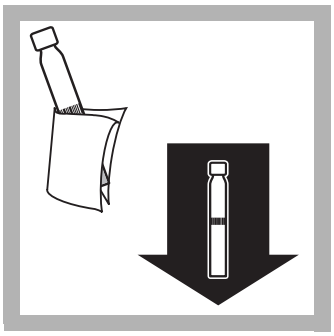
4. Invert the capped vial 2–3 times to dissolve the reagent in the DosiCap.

5. Wait 10 minutes.

Install the Light Shield in Cell Compartment #2.



6. When the timer expires, invert the vial again 2–3 times.



7. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4.

No instrument Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 13 of the total phosphorus procedure or step 7 of the reactive phosphorus procedure. Press **OPTIONS**>**MORE**>**REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Color or turbid samples can cause high results. The digestion in the total phosphate procedure usually destroys all color and turbidity and a sample blank is not required.

To compensate for color or turbidity in the reactive phosphate procedure the color forming reagent that is present in the DosiCap C is not added.

To determine the sample blank run the reactive procedure as given, but do not add the DosiCap C in step 3. Cap the vial with the original DosiCap ***Zip*** (do not remove the foil). Use the side of the cap without the reagent. The value obtained in step 7 is then subtracted from the value obtained on the original reactive phosphate sample to give the corrected sample concentration.

Alternatively, reactive phosphate samples that contain only turbidity may be first filtered through a membrane filter and then analyzed. Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined. Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
SO_4^{2-}	20 g/L
Cl^-	10 g/L
Ca^{2+}	1000 mg/L
K^+, Na^+	4000 mg/L
NO_3^-	500 mg/L
Mg^{2+}	400 mg/L
Co^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+},Cu^{2+}, Ni^{2+}, NO^{2-}, Cd^{2+}, NH^{4+}, Mn^{2+}, Al^{3+}, CO_3^{2-}	200 mg/L
I^-	100 mg/L
SiO_2	50 mg/L
Hg^{2+}	40 mg/L
Pb^{2+}	20 mg/L
Ag^+, Sn^{4+}	10 mg/L
Cr^{3+}	5 mg/L
Cr^{6+}	1 mg/L

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for total phosphorus up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid (about 2 mL per liter) and storing at 4 °C. Samples to be analyzed for reactive phosphorus should not be preserved with acid. These samples should be stored at 4 °C and analyzed within 48 hours. Warm samples to 15–25 °C and neutralize with 5.0 N Sodium Hydroxide before analysis if acid has been added. Correct for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the reactive and total phosphorus methods with a 10 mg/L phosphate standard solution. Use 5.0 mL of this standard in place of the sample in step 3 of the total phosphorus procedure or in step 2 of the reactive phosphorus procedure.
2. Alternately, use 0.5 mL of a Wastewater Effluent Mixed Parameters Inorganics Standard in place of the sample. This standard contains 10 mg/L phosphate in the presence of several other ions such as nitrate, sulfate and ammonia.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) are first converted to reactive orthophosphate in the total phosphorus procedure. Treatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are also converted to orthophosphates by heating with acid and persulfate in the total phosphorus procedure. The reactive phosphorus procedure measures only the reactive (ortho) phosphorus present in the sample.

The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue. Test results are measured at 890 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Phosphorus, Reactive and Total, HR TNT844 Reagent Set	1	25/pkg	TNT844

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-01
OR			
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipettor, variable volume, 100–1000 µL	1	each	27949-00
Pipettor Tips, for 27949-00 pipettor	1	400/pkg	27950-00
Test Tube Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Phosphate Standard Solution, 10-mg/L as PO_4	946 mL	14204-16
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB200-03
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 15x13mm + 15x13 mm (dual block)	each	DRB200-07
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-08
Filter Holder, glass for vacuum filtration (SUVA)	each	2340-00
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	each	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	each	546-53
Hydrochloric Acid 6N (1:1)	500 mL	884-49
Sodium Hydroxide, 5.0 N, 1000 mL	1000 mL	2450-53
Sulfuric Acid, concentrated, 500 mL	500 mL	979-49
Tubing, rubber	12 ft	560-19

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate) and Total

Method 10209 Reactive; Method 10210 Total

Ascorbic Acid Method

TNTplus™ 845

UHR (6 to 60 mg/L PO_4^{3-} or 2 to 20 mg/L PO_4–P)

Scope and Application: For wastewater, drinking water, boiler water, surface water, and process analysis

Test Preparation

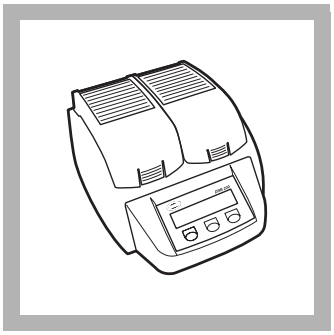
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). Recommended reagent storage temperature is 15–25 °C (59–77 °F).
Recommended sample pH is between 2–10.
TNT plus methods are activated from the Main Menu screen when the sample vial is inserted into the sample cell holder.
The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.

Collect the following items:	**Quantity**
Phosphorus, Reactive Total HR TNT845 Reagent Set	1
DRB200 Reactor with 13-mm wells (adapter available for DRB Reactors with 16-mm holes)	1
Light Shield	1
Pipettor for 100–1000 µL Sample	1
Pipettor Tips for 100–1000 µL Pipettor	2
Test Tube Rack	1–3

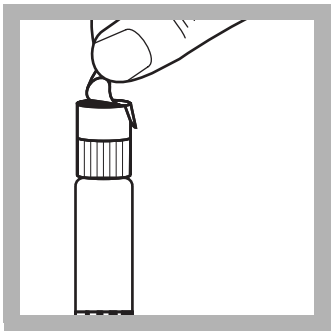
Note: *Reorder information for consumables and replacement items is on page 7.*

TNTplus—Phosphorus, Total Method 10210

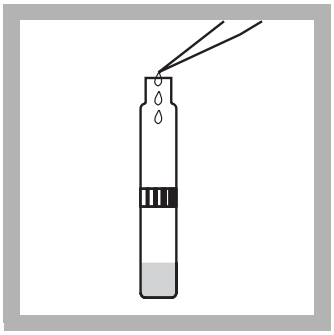


1. Turn on the DRB200 Reactor Heat to 100 °C.

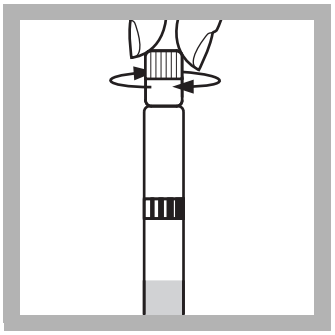
Note: *For DRB200 Reactors with 16-mm wells, insert a 16-mm to 13-mm adapter sleeve into each well before turning on the reactor.*



2. Carefully remove the protective foil lid from the DosiCap™ ***Zip***. Unscrew the cap from the vial.



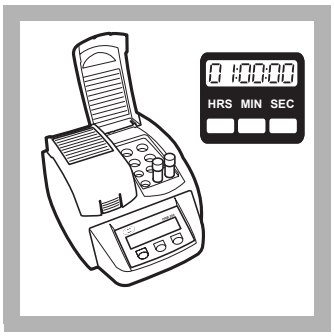
3. Carefully pipet 0.4 mL (400 µL) of sample into the vial.



4. Flip the DosiCap ***Zip*** over so the reagent side faces the vial. Screw the cap tightly onto the vial.

5. Shake the vial 2–3 times to dissolve the reagent in the cap.

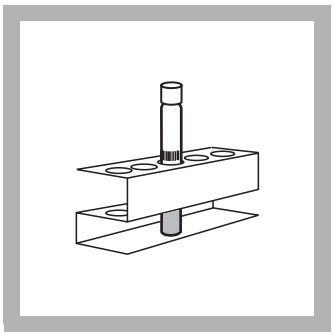
Verify that the reagent has dissolved by looking down through the open end of the DosiCap ***Zip***.



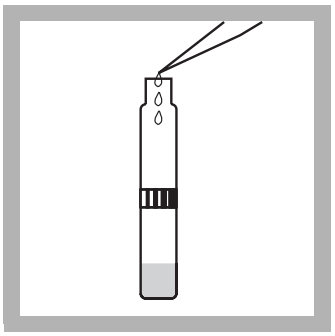
6. Insert the vial in the DRB200 Reactor.

Close the protective cover.

Heat for 1 hour at 100 °C.

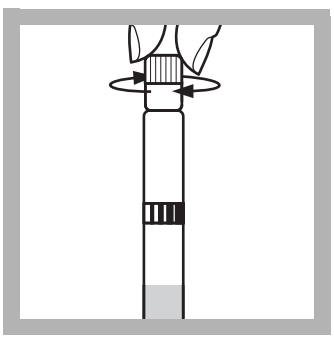


7. After the timer expires, carefully remove the hot vial from the reactor. Insert them in a test tube rack and allow to cool to room temperature (15–25 °C).

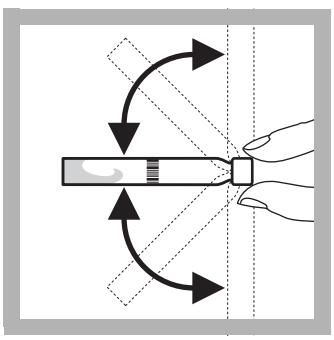


8. Pipet 0.5 mL (500 µL) of Reagent B into the cooled vial.

Immediately close the Reagent B container.

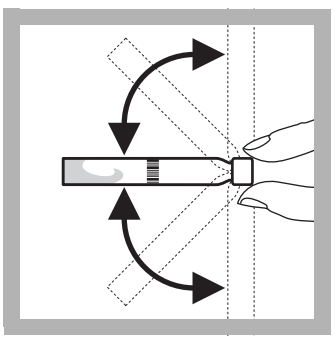


9. Screw a gray DosiCap C onto the vial.

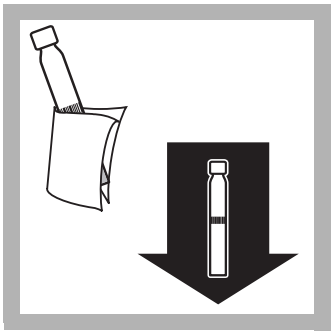


10. Invert the vial 2–3 times to dissolve the reagent in the DosiCap.

11. Wait 10 minutes. Install the Light Shield in Cell Compartment #2.



12. When the timer expires, invert the vial again 2–3 times.

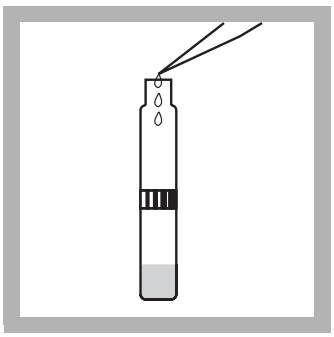


13. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

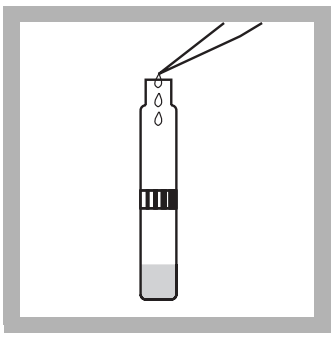
Results are in mg/L PO_4.

No instrument Zero is required.

TNTplus—Phosphorus, Reactive Method 10209

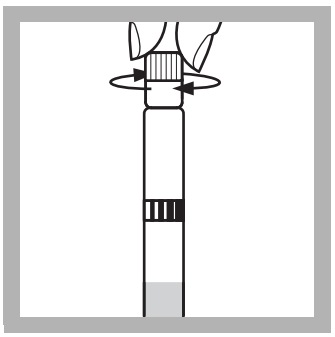


1. Carefully pipet 0.4 mL (400 µL) of sample into the vial.

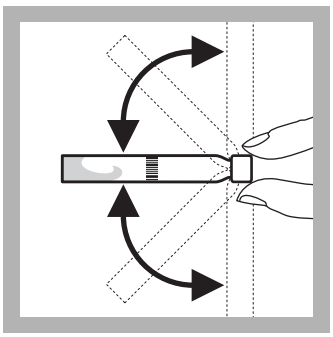


2. Pipet 0.5 mL (500 µL) of Reagent B into the vial.

Immediately close the Reagent B container.



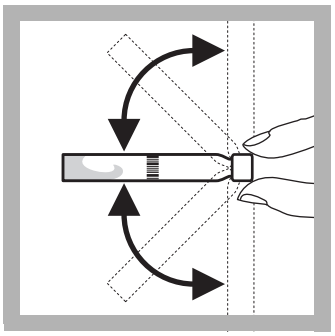
3. Screw a grey DosiCap C onto the vial.



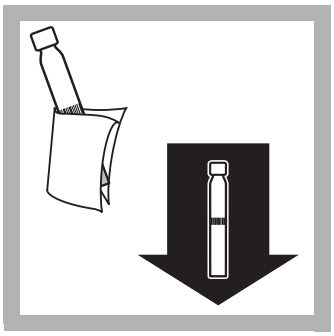
4. Invert the vial 2–3 times to dissolve the reagent in the DosiCap.

5. Wait 10 minutes.

Install the Light Shield in Cell Compartment #2.



6. When the timer expires, invert the vial again 2–3 times.



7. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4.

No instrument Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 13 of the total phosphorus procedure or step 7 of the reactive phosphorus procedure. Press **OPTIONS**>**MORE**>**REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Color or turbidity in samples can cause high results. The digestion in the total phosphate procedure usually destroys all color and turbidity and a sample blank is not required.

To compensate for color or turbidity in the reactive phosphate procedure the color forming reagent that is present in the DosiCap C is not added.

To determine the sample blank run the reactive procedure as given, but do not add the DosiCap C in step 3. Cap the vial with the original DosiCap ***Zip*** (do not remove the foil). Use the side of the cap without the reagent. The value obtained in step 7 is then subtracted from the value obtained on the original reactive phosphate sample to give the corrected sample concentration.

Alternatively, reactive phosphate samples that contain only turbidity may be first filtered through a membrane filter and then analyzed.Samples without color or turbidity do not require sample blanks.

Interferences

The ions listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. Cumulative effects and the influence of other ions have not been determined. Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
SO_4^{2-}	5000 mg/L
Cl^-	2000 mg/L
K^+, Na^+, Ca^{2+}	1000 mg/L
Mg^{2+}, NO_3^-	500 mg/L
Co^{2+}, Fe^{2+}, Fe^{3+}, Zn^{2+},Cu^{2+}, Ni^{2+}, I^-, NO_2^-, Cd^{2+}, Sn^{4+},NH_4^+, Mn^{2+}, Al^{3+}, Hg^{2+}, Pb^{2+}, SiO_2	50 mg/L
Ag^+	25 mg/L
Cr3+	10 mg/L
Cr6+	5 mg/L

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for total phosphorus up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C. Samples to be analyzed for reactive phosphorus should not be preserved with acid. These samples should be stored at 4 °C and analyzed within 48 hours. Warm samples to 15–25 °C and neutralize with 5.0 N Sodium Hydroxide* before analysis if acid has been added. Correct for volume additions.

Accuracy Check

Standard Solution Method

1. Check the accuracy of the reactive and total phosphorus methods with a 50 mg/L phosphate standard solution. Use 0.4 mL this 50 mg/L standard in place of the sample in step 3 of the total phosphorus procedure or in step 2 of the reactive phosphorus procedure.

2. Alternately, use 0.4 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample. This standard contains 10 mg/L phosphate in the presence of several other ions such as nitrate, sulfate and ammonia.

* See Optional Reagents and Apparatus on page 7.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) are first converted to reactive orthophosphate in the total phosphorus procedure. Treatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are also converted to orthophosphates by heating with acid and persulfate in the total phosphorus procedure. The reactive phosphorus procedure measures only the reactive (ortho) phosphorus present in the sample.

The reactive or orthophosphate ions react with molybdate and antimony ions in an acidic solution to form an antimonyl phosphomolybdate complex, which is reduced by ascorbic acid to phosphomolybdenum blue. Test results are measured at 890 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Phosphorus, Reactive and Total, UHR TNT845 Reagent Set	1	25/pkg	TNT845

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 115 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-01
DRB200 Reactor, 230 V, 9x13mm + 2x20 mm (mono block)	1	each	DRB200-05
Light Shield	1	each	LZV646
Pipet, variable volume, 100–1000 µL	1	each	27949-00
Pipet Tips, for 27949-00 pipet	1	400/pkg	27950-00
Test Tube Rack	1–3	each	18641-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Phosphate Standard Solution, 50-mg/L as PO_4^{3-}	500 mL	171-49
Wastewater Influent Inorganics Standard for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
TNTplus Reactor adapter sleeves, 16-mm to 13-mm diameter	5/pkg	28958-05
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
DRB200 Reactor, 115 V, 21x13 mm + 4x20 mm (dual block)	each	DRB200-02
DRB200 Reactor, 115 V, 15x13 mm + 15x13 mm (dual block)	each	DRB200-03
DRB200 Reactor, 115 V, 12x13 mm + 8x20 mm (dual block)	each	DRB200-04
DRB200 Reactor, 230 V, 21x13mm + 4x20 mm (dual block)	each	DRB200-06
DRB200 Reactor, 230 V, 15x13mm + 15x13 mm (dual block)	each	DRB200-07
DRB200 Reactor, 230 V, 12x13mm + 8x20 mm (dual block)	each	DRB200-08
Filter Holder, glass for vacuum filtration (SUVA)	each	2340-00
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	each	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	each	546-53
Hydrochloric Acid Solution, 1:1	500 mL	884-49
Sodium Hydroxide, 5.0 N	1000 mL	2450-53
Sulfuric Acid, concentrated	500 mL	979-49
Tubing, rubber	12 ft	560-19

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Updated November 2005 Edition 1

Phosphorus, Reactive (Orthophosphate)

Method 10214 **Molybdovanadate Method**

TNTplus™ 846

(5.0 to 90.0 mg/L PO_4^{3-} or 1.6 to 30 mg/L PO_4–P)

Scope and Application: For wastewater, drinking water, boiler water, surface water, and process water

Test Preparation

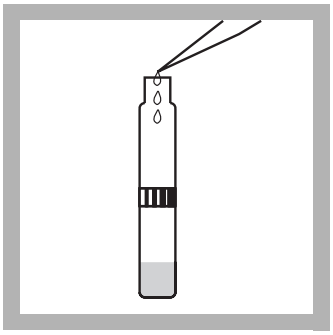
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Please read Safety Advice and Expiration Date on package.
Recommended sample and reagent temperature is 15–25 °C (59–77 °F). If the test is not performed at the recommended temperature an incorrect result may be obtained.
Recommended sample pH is between 3–10.
Recommended reagent storage is 15–25 °C (59–77 °F).
TNTplus methods are activated from the Main Menu when the same vial is inserted into the sample cell holder.

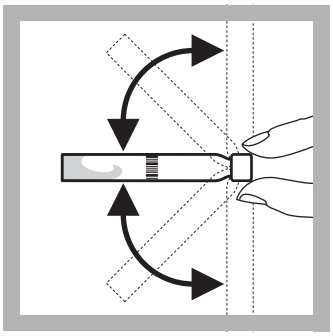
Collect the following items:	**Quantity**
Phosphorus, Reactive TNT846 Reagent Set	1
Light Shield	1
Pipettor for 5.0 mL Sample	1
Pipettor Tip	varies

Note: *Reorder information for consumables and replacement items is on page 4.*

TNTplus Method 10214



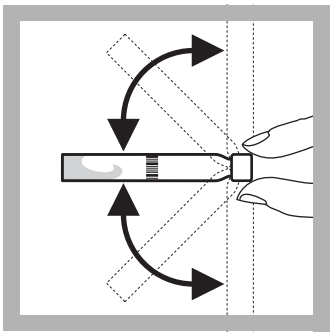
1. Pipet 5.0 mL of sample into the vial.



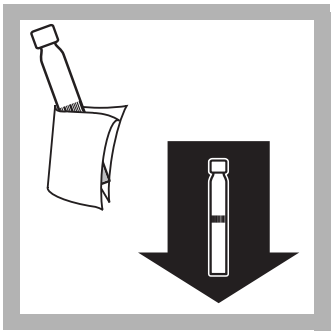
2. Cap and invert the vial gently 2–3 times.

3. Wait 10 minutes.

Install the Light Shield in Cell Compartment #2.



4. After the timer expires, invert the vial 2–3 times again.



5. Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects and performs the correct test.

Results are in mg/L PO_4^{3-}

No Zero is required.

Reagent Blanks

A reagent blank can be measured, and the value subtracted from the results of each test performed using the same reagent lot number. Use deionized water in place of sample and run the procedure as described.

To subtract the value of the blank from a series of measurements, measure the blank per step 5. Press **OPTIONS**>**MORE**>**REAGENT BLANK**. Press **ON**. The measured value of the blank should be displayed in the highlighted box. Press **OK** to accept this value. The reagent blank value will now be subtracted from all results until the function is turned off, or a different method is selected. Alternately, the blank can be recorded and entered at any later time by pressing the highlighted box and using the keypad to enter the value.

Sample Blanks

Color or turbidity in samples can cause high results. An optional Sample Blank Vial (TNT919) is available to correct for color or turbidity in these types of samples.

To use the Sample Blank Vial:

1. Insert the prepared sample vial for the TNT846 method into the photometer. This will launch the correct method and display the uncorrected sample result.

2. Remove the sample vial. Fill an TNT919 vial with 5.0 mL of sample and 1.0 mL of deionized water. Cap with the red stopper.

3. Insert the TNT919 Sample Blank Vial containing the untreated sample into the instrument. The barcode tells the instrument that this is the Sample Blank.

If the Sample Blank value falls within the allowable range, this value will be used to correct the result automatically. The instrument will subtract the Sample Blank from the uncorrected result. Alternatively, samples that contain only turbidity may be first filtered through a membrane filter and then analyzed. Samples without color or turbidity do not require sample blanks.

Interferences

The items listed in Table 1 have been individually checked up to the given concentrations and do not cause interference. The cumulative effects and influence of other ionshas not been detemined. Measurement results can be verified using sample dilutions or standard additions.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Level
SO_4^{2-}, Cl^-	1000 mg/L
K^+, Na^+, Ca^{2+}	500 mg/L
CO_3^{2-}, Fe^{2+}, Fe^{3+}, NO_3^-, Zn^{2+}, Cu^{2+}, Ni^{2+}, Cr^{3+}	50 mg/L
Pb^{2+}	5 mg/L

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples by storing at 4 °C. Samples to be analyzed for reactive phosphorus should not be preserved with acid. These samples should be analyzed within 48 hours. Warm samples to 15–25 °C and neutralize with 5.0 N Sodium Hydroxide* before analysis. Correct for volume additions.

Accuracy Check

Standard Solution Method

4. Check the accuracy of the reactive and total phosphorus methods with a 50 mg/L phosphate standard solution. Use 5.0 mL of this 50 mg/L standard in place of the sample in step 1.

5. Alternately, use 5.0 mL of a Wastewater Influent Mixed Parameters Inorganics Standard in place of the sample in step 1. This standard contains 10 mg/L phosphate in the presence of several other ions such as nitrate, sulfate and ammonia.

Summary of Method

Phosphate ions react with vanadate-molybdate reagent to form a yellow dye. Test results are measured at 435 nm.

* See Optional Reagents and Apparatus on page 4.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Phosphorus, Reactive TNT846 Reagent Set	1	25/pkg	TNT845

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Light Shield	1	each	LZV646
Pipettor, variable volume, 1–5 mL	1	each	27951-00
Pipettor Tips, for 27951-00 pipettor	1	100/pkg	27952-00

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Phosphate Standard Solution, 50-mg/L as PO_4^{3-}	500 mL	171-49
Wastewater Influent Inorganics Standard, inorganics for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bottle, sampling, low density poly, w/cap, 500 mL	12/pkg	20870-79
Filter Holder, glass for vacuum filtration (SUVA)	each	2340-00
Filter, membrane, 47-mm, 0.45-micron, hydrophilic, polyethersulfone for SUVA	each	28947-00
Flask, filtering, glass, 1000-mL (SUVA)	each	546-53
Hydrochloric Acid 6N (1:1)	500 mL	884-49
Sample Blank Vials	—	TNT919
Sodium Hydroxide, 5.0 N	1000 mL	2450-53
Sulfuric Acid, concentrated	500 mL	979-49
Test Tube Rack, for 13-mm vials	each	24979-00
Tubing, rubber	12 ft	560-19

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Updated November 2005 Edition 1

Phosphorus, Total

★Method 8190

PhosVer® 3 with Acid Persulfate Digestion Method

Test 'N Tube™ Vials

(0.06 to 3.50 mg/L PO_4^{3-} or 0.02 to 1.10 mg/L P)

Scope and Application: For water, wastewater, and seawater; USEPA Accepted for reporting wastewater analyses

Test Preparation

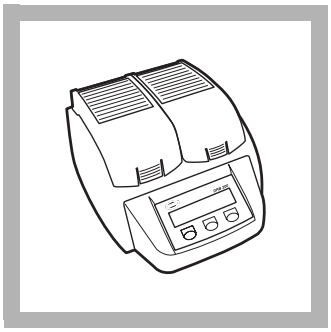
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
The test range for total phosphate is limited to 0.06 to 3.5 mg/L PO_4^{3-}. Values greater than 3.5 mg/L may be used to estimate dilution ratios, but should NOT be used for reporting purposes. If the value is greater than 3.5 mg/L, dilute the sample and repeat the digestion and the colorimetric test.
Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.

Collect the following items:	**Quantity**
Total Phosphorus Test 'N Tube™ Reagent Set	1
Deionized water	varies
DRB200 Reactor	1
Funnel, micro	1
Light Shield	1
Pipet, TenSette®, 1 to 10 mL, plus tips	
Test Tube Rack	1

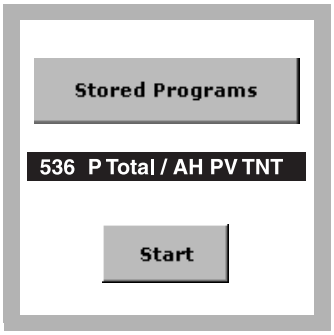
Note: *Reorder information for consumables and replacement items is on page 6.*

Test 'N Tube — Method 8190



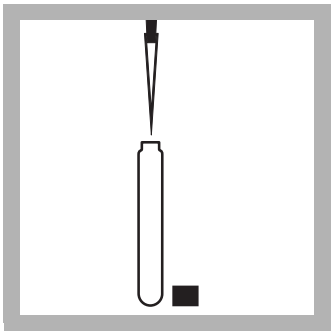
1. Turn on the DRB200 Reactor. Preheat to 150 °C.

See the DRB200 User Manual for selecting pre-programmed temperature applications.

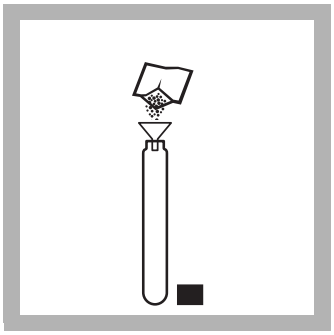


2. Select the test.

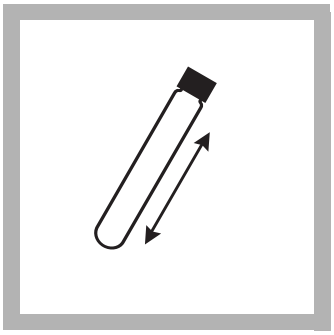
Install the Light Shield in Cell Compartment #2.



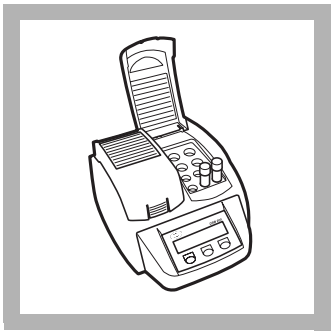
3. Use a TenSette® Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial.



4. Use a funnel to add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to the vial.



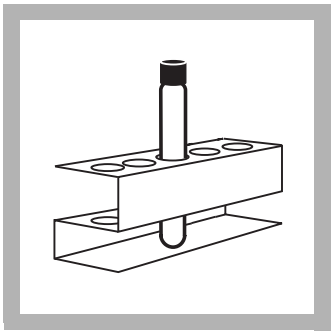
5. Cap tightly and shake to dissolve.



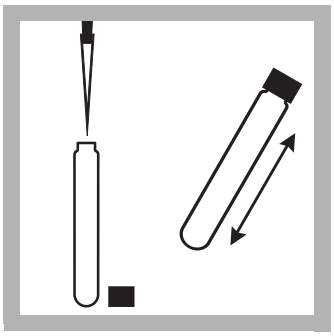
6. Insert the vial into the DRB200. Close the protective cover.

7. Press **TIMER>OK**.

A 30-minute heating period will begin.

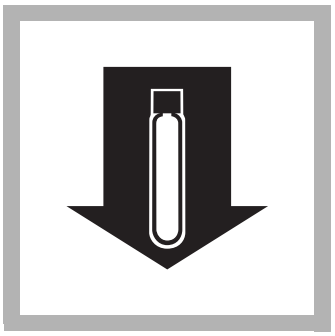


8. When the timer expires, carefully remove the hot vial from the reactor. Insert it in a test tube rack and cool to room temperature.

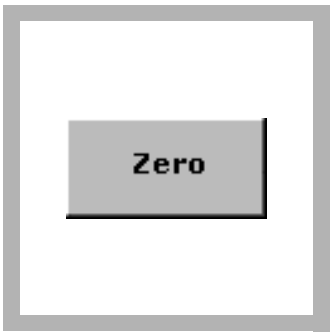


9. Use a TenSette Pipet to add 2 mL of 1.54 N Sodium Hydroxide Standard Solution to the vial. Cap and mix.

10. Wipe the outside of the vial with a damp cloth followed by a dry one.



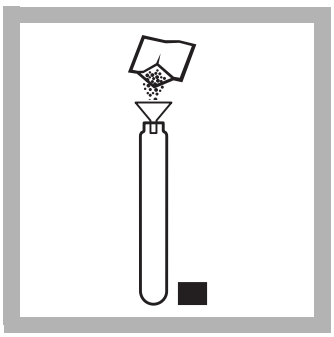
11. Insert the vial into the 16 mm cell holder.



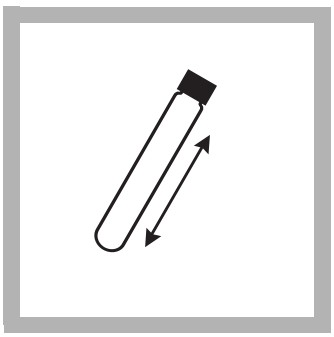
12. Press **ZERO**.

The display will show:

0.00 mg/L PO_4^{3-}



13. Use a funnel to add the contents of one PhosVer 3 Powder Pillow to the vial.



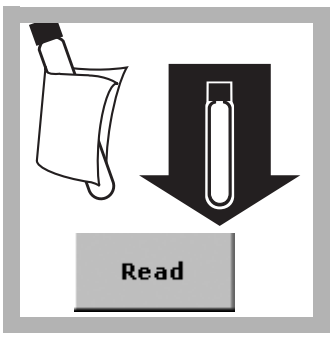
14. Immediately cap tightly and shake to mix for 20–30 seconds.

The powder will not dissolve completely.

15. Press **TIMER>OK**.

A two-minute reaction period will begin.

Read the sample within 2–8 minutes after the timer expires.



16. After the timer expires, wipe the outside of the vial with a wet towel, then a dry one. Insert the prepared sample vial into the 16 mm cell holder.

Press **READ**. Results are in mg/L PO_4^{3-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	Interferes at any level
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
pH, excess buffering	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Sulfide	Greater than 90 mg/L
Turbidity (large amounts) or color	May cause inconsistent results because the acid in the powder pillow may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	Greater than 80 mg/L

Sample Collection, Storage, and Preservation

Collect samples in plastic or glass bottles that have been acid washed with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze the samples immediately for the most reliable results. If prompt analysis is not possible, samples may be preserved up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N Sodium Hydroxide* before analysis. Correct for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. Clean glassware with 1:1 Hydrochloric Acid Standard Solution. Rinse again with deionized water. Do not use phosphate detergents to clean glassware.

2. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

3. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

4. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row.

5. Open a Phosphate 10-mL Ampule Standard, 50-mg/L as PO_4^{3-}.

* See Optional Reagents on page 6.

6. Prepare three sample spikes. Fill three Mixing Cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

7. Analyze each standard addition sample as described above (use a 5-mL aliquot of the spiked sample as the sample). Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use a 3.0-mg/L phosphate standard solution in place of the sample. Perform the procedure as describe above.

2. To adjust the calibration curve using the reading obtained with the 3.0-mg/L PO_4^{3-} Phosphate Standard Solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphates by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color. Test results are measured at 880 nm.

* See Optional Reagents on page 6.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Total Phosphorus Test 'N Tube™ Reagent Set, 50 tests, includes:	—	—	27426-45
PhosVer® 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Potassium Persulfate Powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N	2 mL	100 mL	27430-42
Total and Acid Hydrolyzable Test Vials[1]	1	50/pkg	—
Water, deionized	varies	4 L	272-56

[1] Not sold separately

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Funnel, micro	1	each	25843-35
Light Shield	1	each	LZV646
Pipet, TenSette®, 1.0 to 10 mL	1	each	19700-10
Pipet Tips for TenSette Pipet 19700-10	1	250/pkg	21997-25
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Test Tube Rack	1–2	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
Drinking Water Standard, Mixed Parameter, Inorganic for F-, NO_3, PO_4, SO_4	500 mL	28330-49
Phosphate Standard Solution, 10-mL Voluette® Ampule, 50-mg/L as PO_4^{3-}	16/pkg	171-10
Phosphate Standard Solution, 1-mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, 3 mg/L as PO_4^{3-}	946 mL	20597-16
Wastewater Standard, Effluent Inorganics, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49

Optional Reagents

Description	Cat. No.
Cylinder, mixing, 25 mL	1896-40
Hydrochloric Acid Solution, 1:1, 500 mL	884-49
Sodium Hydroxide, 5.0 N, 1000 mL	2450-53
Sulfuric Acid, concentrated, 500 mL	979-49

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Updated November 2005 Edition 1

Phosphorus, Total

Molybdovanadate Method with Acid Persulfate Digestion[1]

Method 10127

Test ‘N Tube™ Vials

HR (1.0 to 100.0 mg/L PO_4^{3-})

Scope and Application: For water and wastewater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

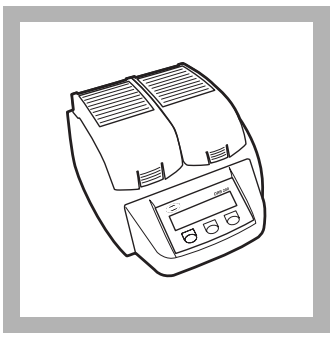
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Reagent blanks can be used more than once, but should not be used more than one day.
The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.

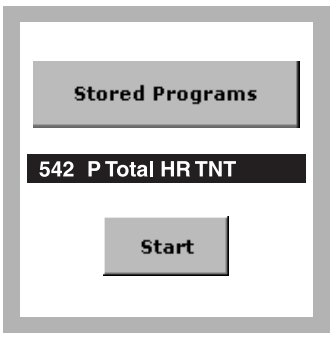
Collect the following items:	**Quantity**
Total High Range Phosphorus Test ’N Tube™ Reagent Set	1
DRB200 Reactor, 15 x 16 mm	1
Light Shield	1
Pipet, TenSette®, 1 to 10 mL, plus tips	1
Test Tube Rack	1–3

Note: *Reorder information for consumables and replacement items is on page 5.*

Test ‘N Tube — Method 10127

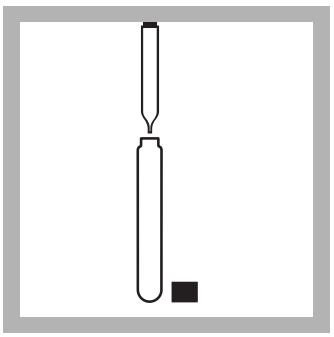


1. Turn on the DRB 200 Reactor. Heat to 150 °C.

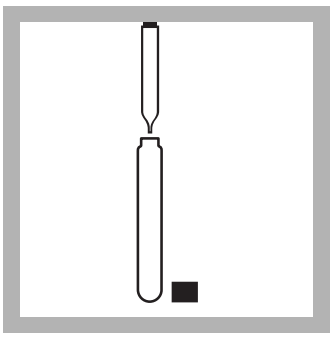


2. Select the test.

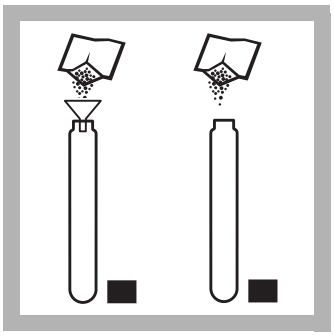
Install the Light Shield in Cell Compartment #2.



3. Blank Preparation: Use a TenSette® Pipet to add 5.0 mL of deionized water to a Total Phosphorus Test ‘N Tube Vial.



4. Prepared Sample: Use a TenSette Pipet to add 5.0 mL of sample to a Total Phosphorus Test ‘N Tube Vial.



5. Use a funnel to add the contents of one Potassium Persulfate Powder Pillow to each vial.

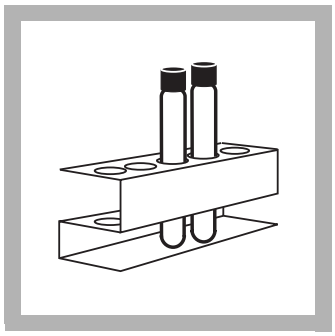
Cap tightly and shake to dissolve.

6. Insert the vials in the DRB 200 Reactor.

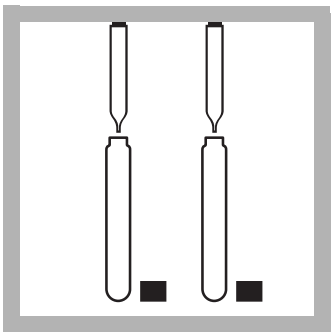
Close the protective cover.

7. Press **TIMER>OK**.

A 30-minute heating period will begin.

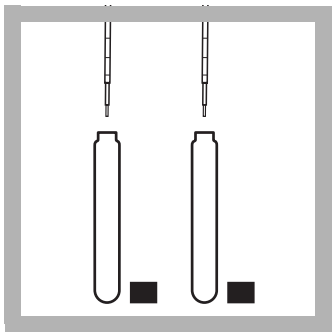


8. After the timer expires, carefully remove the hot vials from the reactor. Insert them in a test tube rack and allow to cool to room temperature (18–25 °C).



9. Use a TenSette Pipet to add 2.0 mL of 1.54 N sodium hydroxide to each vial.

Cap and invert to mix.

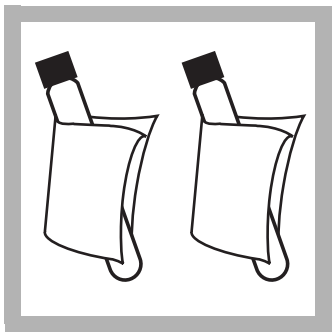


10. Use a polyethylene dropper to add 0.5 mL of Molybdovanadate Reagent to each vial.

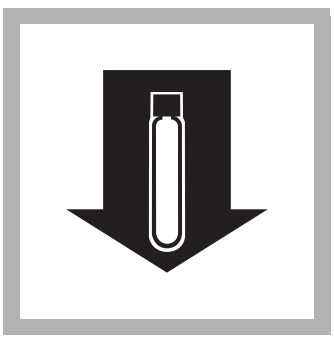
Cap and invert to mix.

11. Press **TIMER>OK**.

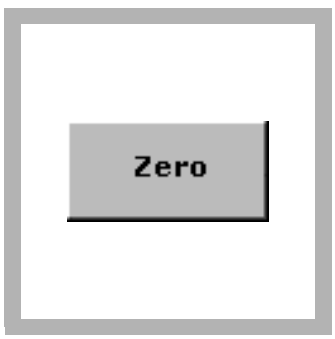
A 7-minute reaction period will begin. Read the sample within seven to nine minutes after adding the Molybdovanadate Reagent.



12. Wipe the vials with a damp towel, followed by a dry one, to remove fingerprints or other marks.



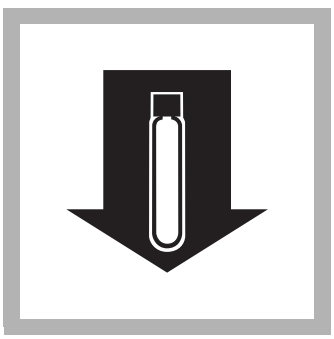
13. When the timer expires, insert the blank into the 16-mm cell holder.



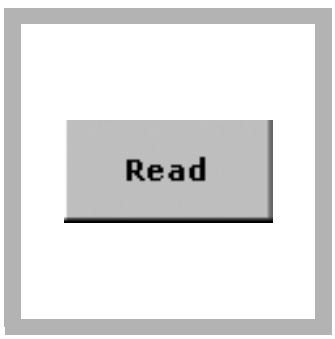
14. Press **ZERO**.

The display will show:

0.0 mg/L PO_4^{3-}



15. Insert the prepared sample into the 16-mm cell holder.



16. Press **READ**.

Results are in mg/L PO_4^{3-}.

Interferences

Large amounts of sample turbidity may cause inconsistent results in the test because the acid present in the reagents may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Arsenate	Causes positive interference if the sample is warm when the molybdovanadate reagent is added (after the digestion).[1] Cool the sample after digestion before adding reagent.
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Causes positive interference if the sample is warm when the molybdovanadate reagent is added (after the digestion). Cool the sample after digestion before adding reagent.
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, Cold (less than 18 °C)	Cause a negative interference.
Temperature, Hot (greater than 25 °C)	Causes a positive interference. Post-digestion samples should be brought to room temperature (18–25 °C) before the addition of the Molybdovanadate Reagent or sodium hydroxide.

[1] Gentle warming of the sample to reach room temperature will not cause this substance to interfere.

Table 2 Noninterfering Below 1000 mg/L

Pyrophosphate	Tetraborate	Selenate	Benzoate
Citrate	Oxalate	Lactate	Tartrate
Formate	Salicylate	Al^{3+}	Fe^{3+}
Mg^{2+}	Ca^{2+}	Ba^{2+}	Sr^{2+}
Li^{+}	Na^{+}	K^{+}	NH_4^{+}
Cd^{2+}	Mn^{2+}	NO_3^{-}	NO_2^{-}
SO_4^{2-}	SO_3^{2-}	Pb^{2+}	Hg^{+}
Hg^{2+}	Sn^{2+}	Cu^{2+}	Ni^{2+}
Ag^{+}	U^{4+}	Zr^{4+}	AsO_3^{-}
Br^{-}	CO_3^{2-}	ClO_4^{-}	CN^{-}
IO_3^{-}	SiO_4^{4-}	—	

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid washed with 1:1 Hydrochloric Acid Solution* and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning the glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 28 days by adjusting the pH to 2 or less with concentrated Sulfuric Acid* (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N Sodium Hydroxide* before analysis. Correct for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. Clean glassware with 1:1 Hydrochloric Acid Standard Solution. Rinse again with deionized water. Do not use detergents containing phosphate to clean glassware.
2. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.
3. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
4. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
5. Snap the neck off a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-}.
6. Prepare three sample spikes. Fill three mixing cylinders* with 10 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to each cylinder. Mix well.
7. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use a 50-mg/L Phosphate standard in place of the sample. Perform the procedure as described.
2. To adjust the calibration curve using the reading obtained with the 50-mg/L PO_4^{3-} Phosphate Standard Solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 5.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro-, or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphates by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a mixed phosphate/molybdate complex. In the presence of vanadium, yellow molybdovanadophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration. Test results are measured at 420 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Total High Range Phosphorus Test 'N Tube™ Reagent Set, includes:	—	50 vials	27672-45
(1) Molybdovanadate Reagent	0.5 mL	25 mL	20760-26
(1) Potassium Persulfate powder Pillows	1	50/pkg	20847-66
(1) Sodium Hydroxide Solution, 1.54 N	2 mL	100 mL	27430-42
(1) Total Phosphorus Test Vials[1]	1	50/pkg	—
(2) Water, deionized	5 mL	100 mL	272-42

[1] Not available separately.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
DRB200 Reactor, 110 V, 15 x 16 mm	1	each	LTV082.53.40001
DRB200 Reactor, 220 V, 15 x 16 mm	1	each	LTV082.52.40001
Light Shield	1	each	LZV646
Pipet, TenSette®, 1 to 10 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	1	250/pkg	21997-25
Test Tube Rack	1–3	each	18641-00

Recommended Standards

Description	Unit	Cat. No.
Phosphate Standard Solution, Voluette® ampule, 500-mg/L as PO_4^{3-}, 10-mL	16/pkg	14242-10
Phosphate Standard Solution, 50-mg/L as PO_4^{3-}	500 mL	171-49
Wastewater Standard, Influent Inorganics for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28331-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Hydrochloric Acid Solution, 1:1	500 mL	884-49
Sodium Hydroxide, 5.0 N	1000 mL	2450-53
Sulfuric Acid, concentrated	500 mL	979-49

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Updated November 2005 Edition 1

Potassium

Method 8049 Tetraphenylborate Method

Powder Pillows (0.1 to 7.0 mg/L)

Scope and Application: For water, wastewater, and seawater

Test Preparation

Before starting the test:

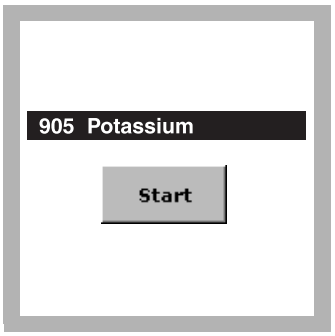
Program # 905 has a calibration curve for potassium; however, due to potential variation between lots of Potassium 3 Reagent, perform a new calibration for each lot of reagent to obtain best accuracy. Prepare and store the calibration as directed under Calibration on page 4.
Filter highly colored or turbid samples before analysis.
The final samples are highly acidic. Neutralize to pH 6–9 and flush to drain for disposal. Refer to a current MSDS for pollution prevention and waste management information.
After the test, clean the cells with soap and a brush.

Collect the following items:	Quantity
Potassium Reagent 1 Powder Pillow	1
Potassium Reagent 2 Powder Pillow	1
Potassium Reagent 3 Powder Pillow	1
Potassium Standard Solution, 100-mg/L	varies
Clippers	1
Cylinder, mixing, 25-mL	1
Flask, volumetric, 100-mL Class A	8
Pipet, TenSette®, 1–10 mL, plus tips	varies
Sample Cells, 1-inch square, 10-mL	2
Water, deionized	varies

Note: *Reorder information for consumables and replacement items is on page 6.*

Powder Pillows Method 8049

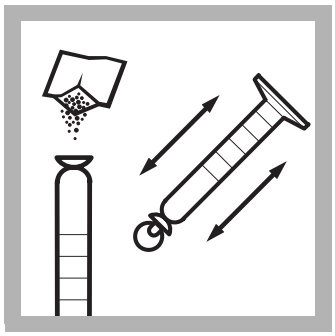
1. Press **USER PROGRAMS**.



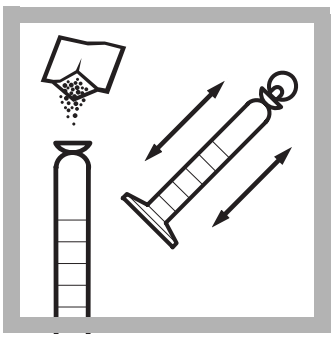
2. Select the test.

When performing this procedure for the first time the instrument must be programmed. See User Programming on page 4.

3. Fill a graduated mixing cylinder with 25 mL of sample.



4. Add the contents of one Potassium 1 Reagent Pillow. Add the contents of one Potassium 2 Reagent Pillow. Stopper and invert several times to mix.

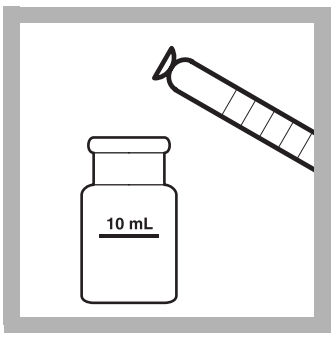


5. Add the contents of one Potassium 3 Reagent Pillow after the solution clears. Stopper and shake the solution for 30 seconds.

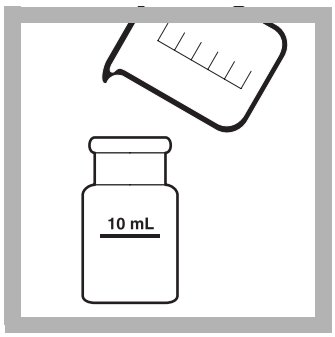
A white turbidity will form if potassium is present.

6. Press **TIMER>OK**.

A three-minute reaction period will begin.

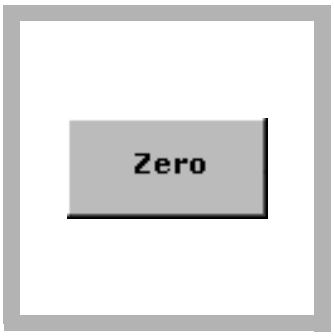


7. Prepared Sample: Pour at least 10-mL of the solution from the cylinder into a square sample cell.



8. Blank Preparation: When the timer expires, fill the second square sample cell with 10 mL of sample.

9. Wipe the blank and insert it into the cell holder with the fill line facing right.

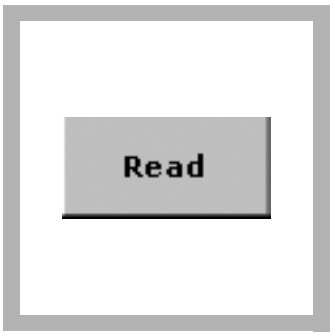


10. Press **ZERO**.

The display will show:

0.0 mg/L K

11. Within seven minutes after the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing right.



12. Press **READ**.

Results are in mg/L K.

Interferences

The substances listed below have been tested and will not interfere at or below the levels stated. If these substances are present at higher levels, conduct interference studies at the higher levels to determine if the substance interferes.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Ammonium Nitrogen	15 mg/L as N
Calcium	7000 mg/L as $CaCO_3$
Chloride	15,000 mg/L
Magnesium	6000 mg/L as $CaCO_3$

Sample Collection, Preservation, and Storage

Collect samples in acid-washed plastic bottles. Adjust the pH to 2 or less with Nitric Acid (about 2 mL per liter)*. Preserved samples may be stored at least six months at room temperature. Before analysis, adjust the pH to 4–5 with 5.0 N Sodium Hydroxide*. Do not measure pH in the sample container with a pH electrode, as this will introduce potassium from the filling solution. Use pH Paper* or pour off sample and test pH in a separate beaker. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

Note: *This procedure is applicable only to Stored Program 905, and not to User Programs.*

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

* See Optional Reagents and Apparatus on page 6.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Potassium Voluette® Ampule Standard, 250-mg/L K.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Calibration

Standard Preparation

An approximate calibration curve is preprogrammed within Program 905. For improved accuracy, a new calibration should be performed with each new lot of reagents. Prepare calibration standards containing 1, 2, 3, 4, 5, 6, 7, and 8 mg/L potassium as follows:

1. Into eight different 100-mL Class A volumetric flasks, pipet 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, and 8.0 mL of the 100-mg/L Potassium Standard Solution using class A glassware or TenSette Pipet.

2. Dilute to the mark with deionized water. Mix thoroughly.

3. Use deionized water for the 0-mg/L potassium standard.

User Programming

1. Press **USER PROGRAMS** on the main menu.

2. To perform the initial potassium calibration, press **PROGRAM OPTIONS** and **NEW**. Key in any available program number (950-999). Press **OK**.

3. Use the alphanumeric keys to enter a name for the potassium test into the "Program Name?" field. Press **NEXT**.

4. Set up the rest of the parameters as follows, pressing **NEXT** to move to the next screen:

   - Program Type: Single wavelength
   - Units: mg/L
   - Wavelength λ (nm): 650
   - Concentration Resolution: 0.1
   - Chemical Form: K
   - Calibration: Read Standards

5. Press **NEXT>EXIT**.

* See Optional Reagents and Apparatus on page 6.

6. To enter the remainder of the test parameters, press each line to highlight it, press **EDIT**, then enter the value specified below. Press **OK** to accept the value, and press **OK** again to return to the list. Set up the following parameters as:

   - Upper Limit: On, 8.0
   - Lower Limit: On, -0.2
   - Timer 1: Timer 3:00
   - Press Calibration: C=a + bA >Edit>OK

7. Enter the concentrations for the calibration, starting with 0.0, in the left column. (Press **+** and enter each value, then press **OK**.)

8. When all standard concentrations have been entered, press the **UP** arrow several times to move to the 0.0 line.

9. Insert the cell containing the blank (deionized water) and press **ZERO**.

10. Perform the potassium test on each standard and insert the first prepared standard into the cell holder. Press the **DOWN** arrow, if necessary, to highlight the line corresponding to this standard concentration. Press **READ**. Repeat for each standard concentration.

11. Press **GRAPH**. If the graph is acceptable, press **DONE>EXIT**. It may be possible to obtain a better fit to the data by pressing **NEXT CURVE**. The curve which results in the highest r2 value is generally the best fit. After selection of the best curve, press **DONE>EXIT**.

12. Press **YES** in response to the “Store Program?” prompt to save the calibration.

Summary of Method

Potassium in the sample reacts with sodium tetraphenylborate to form potassium tetraphenylborate, an insoluble white solid. The amount of turbidity produced is proportional to the potassium concentration. Test results are measured at 650.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Potassium Reagent Set:	—	—	24591-00
Potassium Reagent 1 Powder Pillow	1	25/pkg	14321-98
Potassium Reagent 2 Powder Pillow	1	25/pkg	14322-98
Potassium Reagent 3 Powder Pillow	1	100/pkg	14323-99
Potassium Standard Solution, 100-mg/L	varies	500 mL	23517-49
Water, deionized	varies	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers	1	each	968-00
Cylinder, mixing, 25-mL	1	each	1896-40
Flask, volumetric, 100-mL Class A	8	each	14574-42
Pipet, TenSette®, 1–10 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	varies	50/pkg	21997-96
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Potassium Standard Solution, 10-mL Voluette® Ampule, 250 mg/L	16/pkg	14790-10

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Nitric Acid, 1:1	500 mL	2540-49
pH Paper, 1.0–11.0	—	391-33
Sodium Hydroxide, 5.0 N	50 mL	2450-26

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Updated November 2005 Edition 1

Quaternary Ammonium Compounds

Method 8337

Powder Pillows

Direct Binary Complex Method

(0.2 to 5.0 mg/L as CTAB)

Scope and Application: For cooling tower water and pool/spa water

Test Preparation

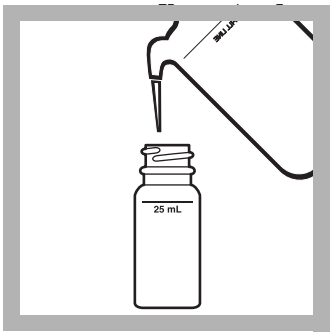
Collect the following items:	Quantity
QAC Reagent 1 Powder Pillows	2 pillows
QAC Reagent 2 Powder Pillows	2 pillows
Bottle, square, with 25 mL mark	2
Clippers, for opening powder pillows	1
Sample Cells, 1-inch square, 10 mL, matched pair	2

Note: *Reorder information for consumables and replacement items is on page 4.*

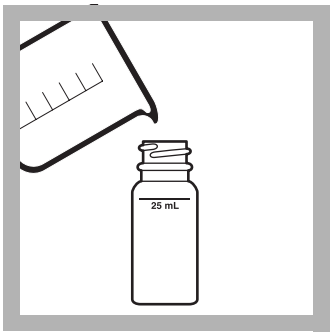
Powder Pillows Method 8337

1. Press **STORED PROGRAMS**.

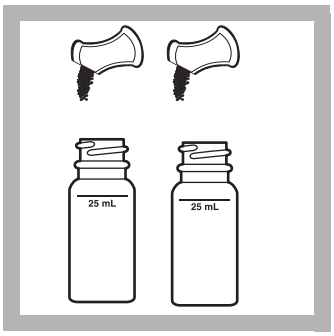
2. Select the test.



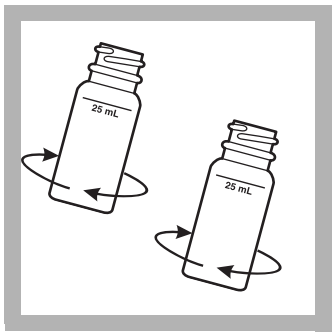
3. Blank Preparation: Fill one 25-mL mixing bottle with 25 mL of deionized water.



4. Prepared Sample: Fill another mixing bottle with 25 mL of sample.

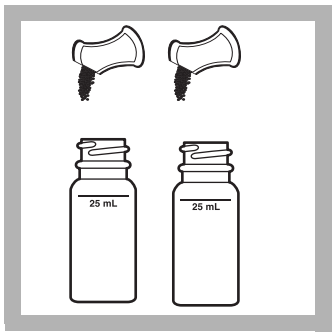


5. Add the contents of one QAC Reagent 1 Powder Pillow to each bottle.

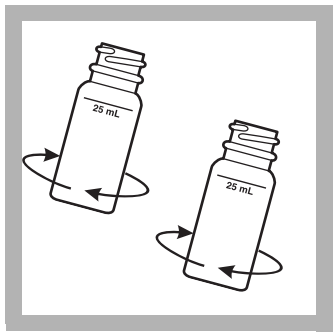


6. Swirl the bottles to dissolve the reagent.

Do not shake! Shaking creates air bubbles that interfere with test results.



7. Add the contents of one QAC Reagent 2 Powder Pillow to each bottle.

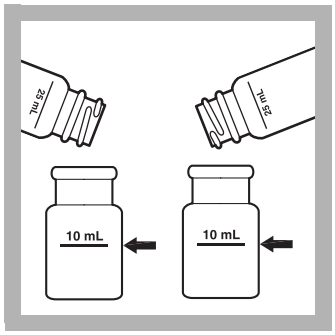


8. Swirl the bottles to dissolve the reagent. **Do not shake.**

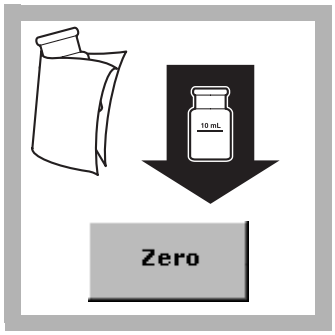
A purple color will form if a quaternary ammonium compound is present

9. Press **TIMER>OK**.

A two-minute reaction period will begin.



10. Pour at least 10 mL of the solutions from the bottles into square sample cells.



11. When the timer expires, insert the blank into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

0.0 mg/L CTAB

12. Insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L CTAB (cetyl-trimethylammonium bromide).

Interferences

Interference studies were conducted by preparing a CTAB standard solution of approximately 3 mg/L as well as a solution of the potential interference. The constituent was said to interfere when the resulting concentration changed by 10%.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium (as $CaCO_3$)	Positive interference above 1350 mg/L
Chlorine, HOCl and OCl^-	Positive interference above 7 mg/L
Cyanuric acid	Negative interference above 70 mg/L
Igepal™ nonionic surfactant	Positive interference above 3 mg/L
Iodine, I_3^-	Positive interference above 3 mg/L
Iron, Fe^{3+}	Positive interference above 80 mg/L
Liquimine™ 14–P, filming amine	Positive interference above 1825 mg/L

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Magnesium, Mg^{2+}	Positive interference above 1350 mg/L
Niaproof™ anionic surfactant	Negative interference above 11 mg/L
Polyacrylic acid	Negative interference above 16 mg/L
Sodium lauryl sulfate	Negative interference above 8 mg/L
Sodium polyphosphate	Positive interference above 1325 mg/L
Tribenzylamine	Positive interference above 7 mg/L
Triton X-100™ nonionic surfactant	Positive interference above 4 mg/L
Urea	Positive interference above 8 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment. Adjust the sample pH between 3 and 5 by using a pH meter or pH paper and adding dropwise an appropriate amount of acid or base such as 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution. If significant volumes of acid or base are used, a volume correction should be made.

Table 2 Noninterfering Substances

Non-interfering Substance	Highest Concentration Tested (mg/L)
Silica, SiO_2	400
Potassium alum, $AlKS_2O_8$	500
Sodium thiosulfate, $Na_2S_2O_3$	30

After several samples have been analyzed, the sample cells may exhibit a build-up of a pink or purple color. A rinse with 1.0 N Sodium Hydroxide Solution followed by an Alconox™ detergent wash and deionized water rinse will eliminate the build-up when it occurs.

Sample Collection, Storage, and Preservation

Collect samples in glass bottles that have been rinsed several times with sample before final sample filling. Do not use plastic containers as plastic adsorbs QACs. Acidify the sample to a pH of less than 2. Store at 4 ± 2 °C.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See in the user manual for more information.
4. Open a QAC Standard Solution, 100-mg/L CTAB.
5. Prepare three sample spikes. Use a graduated cylinder to fill three mixing bottles with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

Prepare a 5.0-mg/L CTAB standard solution as follows:

1. Pipet 5.0 mL of QAC Standard, 100-mg/L as CTAB, into a 100-mL volumetric flask. Dilute to the mark with deionized water. Mix well. Prepare this solution daily. Perform the quaternary ammonium compound procedure as described above.
2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.
3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

The test method makes use of a colorimetric chemistry in which a quaternary ammonium compound reacts with an indicator to produce a color change from pale pink to vivid purple. The test is conducted in a stabilized, acid-buffered solution containing a masking agent to eliminate potential interferences. This test is applicable to the monitoring of QACs in swimming pools and cooling towers. Test results are measured at 575 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Quaternary Ammonium Compounds Reagent Set (100 tests), includes:	—	—	24592-00
(4) QAC Reagent 1 Powder Pillows	2 pillows	50/pkg	24010-66
(8) QAC Reagent 2 Powder Pillows	2 pillows	25/pkg	24012-68

Required Apparatus

Description	Quantity/Test	Unit	Cat No.
Bottle, square, with 25 mL mark	2	each	17042-00
Clippers, for opening powder pillows	1	each	968-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
QAC Standard Solution, 100-mg/L as CTAB	100 mL	24153-42
Water, deionized	4 liters	272-56

Optional Reagents and Apparatus

Description	Cat. No.
Alconox™ detergent	20880-00
Sodium Hydroxide Standard Solution, 1.0 N	1045-32
Sodium Hydroxide Solution, 1.0 N	1045-53
Sulfuric Acid Standard Solution, 1.0 N	1270-32

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Updated November 2005 Edition 1

Selenium

Method 8194

Diaminobenzidine Method[1]

(0.01 to 1.00 mg/L)

Scope and Application: For water and wastewater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test Preparation

Before starting the test:

Distillation is required for determining total selenium. See Distillation on page 5 at the end of the procedure. Use the distillate as the sample in step 3.
Acetone[1] is a suitable solvent for removing toluene from glassware after results are measured.
Toluene (F005) solutions are regulated as hazardous waste by the Federal RCRA. Do not pour these materials down the drain. Water saturated with toluene, toluene solutions, and the cotton plug used in the delivery tube of the separatory funnel should be collected for disposal with laboratory solvent wastes. Refer to the current MSDS for safe disposal and handling information.
If there are visible water bubbles on the bottom of the cell, decant the top portion into a clean, dry 25-mL cell prior to reading the sample.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

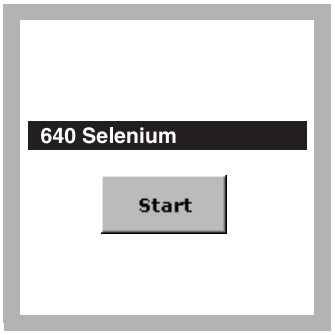
[1] See Optional Reagents and Apparatus on page 8.

Collect the following items:	Quantity
Buffer Solution, sulfate type, pH 2.0	10 mL
Cotton Ball	1
Cylinder, Graduated: 50- and 100-mL	1 of each
Diaminobenzidine, tetrahydrochloride	0.1 g
Distillation Reagents and Apparatus (page 8)	—
Dropper, 0.5 and 1.0 mL marks, one glass and one plastic	1 of each
Flask, Erlenmeyer, 500-mL	2
Funnel, separation	2
Hot Plate, 4-inch diameter	1
Pipet, volumetric, 5-mL, plus safety bulb filler	1
Potassium Hydroxide Standard Solution, 12 N	4 mL
Ring support (3-inch) and stand	1
Sample Cells, 1-inch square glass, 25-mL	2
Spoons, measuring, 0.2 and 0.05 g	1 of each
TitraVer® Hardness Reagent	0.4 g
Toluene, ACS	60 mL
Water, Deionized	100 mL

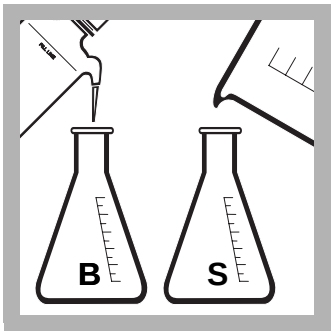
Note: *Reorder information for consumables and replacement items is on page 7.*

Diaminobenzidine Method 8194

1. Press **STORED PROGRAMS**.

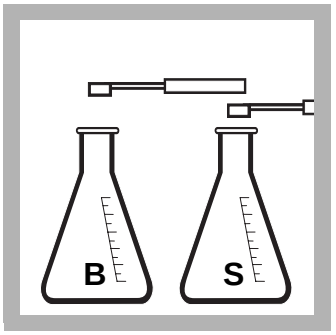


2. Select the test.

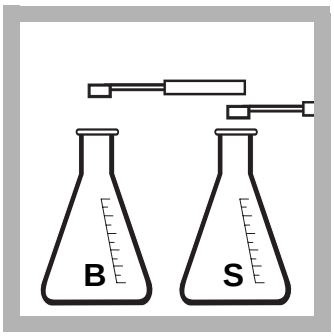


3. Measure 100 mL of deionized water into a 500-mL Erlenmeyer flask. Label the flask “blank”.

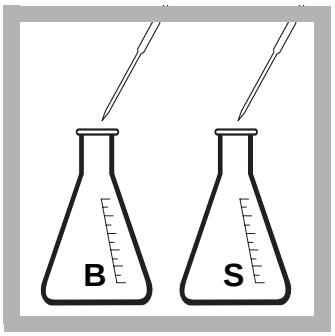
Measure 100 mL of sample into a 500-mL Erlenmeyer flask. Label the flask “sample”.



4. Add a 0.2-g spoonful of TitraVer® Hardness Reagent to each flask. Swirl to mix.

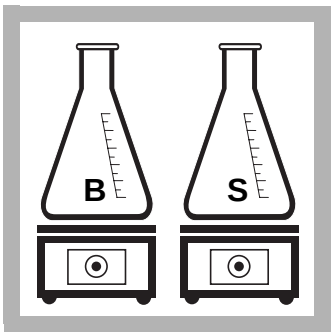


5. Add a 0.05-g spoonful of diaminobenzidene tetrahydrochloride to each flask. Swirl to mix.



6. If you have not distilled the sample, add 5.0 mL of Buffer Solution, sulfate type, pH 2.0 to each flask. Swirl to mix.

If the sample has been distilled, adjust the pH of the distillate to pH 2.7 ± 0.2 using 5 N Sodium Hydroxide Standard Solution. Adjust the blank to pH 2.7 ± 0.2 using 5.25 N Sulfuric Acid Standard Solution.



7. Heat each flask on a hot plate. Bring the contents to a gentle boil.

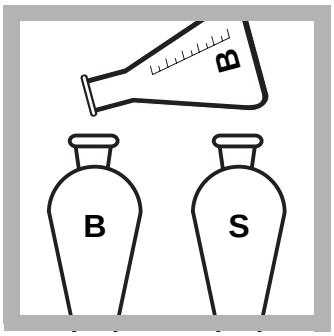
8. Press **TIMER>OK**.

A five-minute reaction period will begin. Continue to boil the contents gently during this time period.

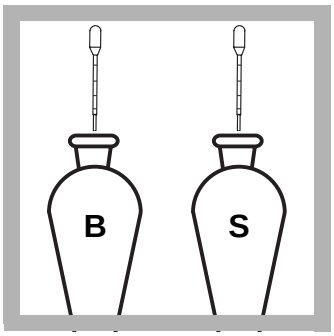
A yellow color will develop if selenium is present.

9. When the timer expires, remove both flasks. Cool to room temperature using a water bath.

Do not boil more than one minute after the timer expires.



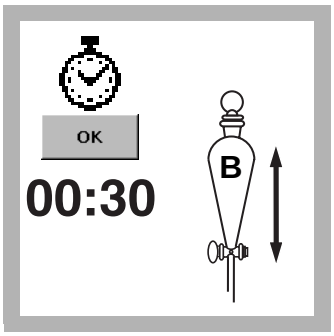
10. Transfer the contents of each flask to separate 250-mL separatory funnels. Label the funnels “blank” and “sample”.



11. Add 2.0 mL of 12 N Potassium Hydroxide Standard Solution to each funnel using a calibrated 1.0-mL plastic dropper. Stopper. Shake each funnel to mix.

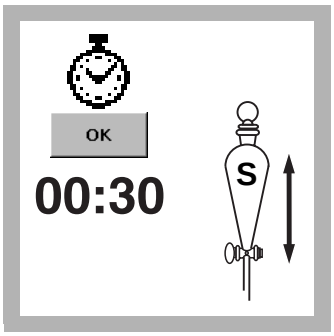
12. Add 30-mL of toluene to each funnel. Stopper. Swirl and invert each funnel, then open the stopcock to vent the funnel. Close the stopcock. Repeat twice with each funnel.

Use toluene only with adequate ventilation.



13. Press **TIMER>OK**.

A 30-second reaction period will begin. During this time, vigorously shake the funnel that contains the blank.

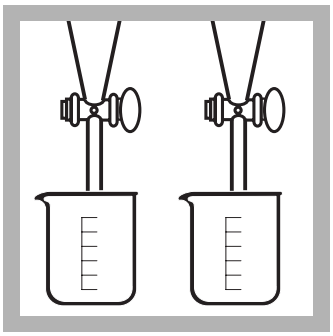


14. Press **TIMER>OK**.

A 30-second reaction period will begin. During this time, vigorously shake the funnel that contains the sample.

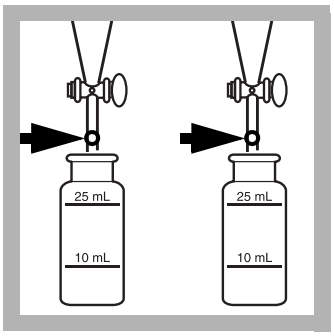
15. Press **TIMER>OK**.

A four-minute reaction period will begin.



16. When the timer expires, drain the lower water layer from each funnel and discard.

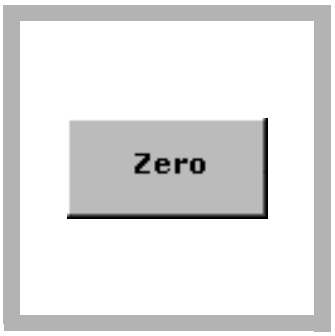
Complete steps 17–20 within five minutes after the timer expires. The developed color is stable, but should be measured as soon as possible.



17. Insert a cotton plug into the delivery tube of each separatory funnel. Slowly drain the toluene into respective sample cells labeled “blank” and “sample”. Stopper the sample cells.

Filtering the toluene through dry, absorbent cotton will remove water or suspended particles.

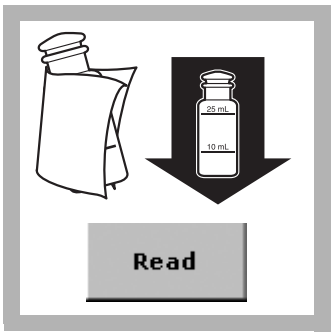
18. Wipe the blank and insert it into the cell holder with the fill line facing right.



19. Press **ZERO**.

The display will show:

0.00 mg/L Se



20. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Se.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Ferric iron	Up to 2.5 mg/L. Distill sample to eliminate interference.
Manganese	Will not interfere.
Strong oxidizing agents (i.e., iodine, bromine, or chlorine)	Can react with the indicator to give low results. Distill sample to eliminate interference.

Note: *There are no positive inorganic interferences with this method.*

Sample Collection, Storage, and Preservation

Collect samples in clean glass or plastic containers. Adjust the pH to 2 or less with Nitric Acid* (about 1.5 mL per liter). Preserved samples can be stored for up to six months at room temperature. Correct the test result for volume additions.

* See Optional Reagents and Apparatus on page 8.

Distillation

CAUTION
Always perform this procedure under a fume hood!

This distillation involves the use of a strong acid and oxidizer at high temperatures. To avoid personal injury, observe all laboratory safety precautions when operating the distilling apparatus.

1. Measure 500 mL of sample into a 1000-mL beaker.

2. Add 1 mL of Methyl Orange Indicator Solution. Stir with a glass rod.

3. Use a dropper to add 0.1 N Hydrochloric Acid Standard Solution dropwise until the solution becomes pink. Then add an additional 2 mL.

4. Use a pipet to add 5.0 mL Calcium Chloride Solution. Mix well.

5. Use a dropper to add 1-g/L Potassium Permanganate Standard Solution drop-wise until the solution is purple.

6. Place the beaker on a hot plate. Evaporate the solution to approximately 250 mL. Periodically add 1-g/L Potassium Permanganate Solution to keep the solution purple.

7. Any precipitate formed at this step is manganese dioxide, and may be ignored.

8. Cool the solution. While cooling, set up the distillation apparatus for the general purpose distillation as shown in the distillation manual.

9. Pour the treated sample solution into the distillation flask. Add a stirring bar to the flask.

10. Pipet 5.0 mL of 0.1 N Sodium Hydroxide Standard Solution into the flask. Turn the stirrer power switch to ON. Set the stir control to 5.

11. Turn on the water and adjust so a constant flow is maintained through the condenser. Set the heat control to 10.

12. When only a few milliliters are left in the distillation flask, turn the power switch off. The distillate in the Erlenmeyer flask may be discarded.

CAUTION
Perform step 13 under a fume hood.

13. When the flask has cooled, add 50 mL of 19.2 N Sulfuric Acid Standard Solution to the flask. Add the contents of one Potassium Bromide Powder Pillow to the flask.

14. Fill a 250-mL beaker to the 75-mL mark with deionized water. Place it under the drip tube. Elevate the beaker with a laboratory jack so the tube extends below the level of the water.

15. Add 1.0 mL of 30% hydrogen peroxide solution to the flask. Turn the stir control to 5 and the heat control to 10. Cap the distillation flask.

16. Heat the distillation flask until the yellow color is gone from the complete distillation apparatus, including the J-tube and condenser. Remove the beaker from under the drip tube.

17. Turn off the heater switch. When the J-tube and condenser have cooled, rinse them with deionized water. Add the washings to the 250-mL beaker. Total volume in the beaker should be approximately 100 mL.

18. Add the Phenol Solution drop-wise to the distilled sample to discharge the bromine color (a white precipitate of tribromophenol will form).

19. Allow the precipitate to settle. Using a dropper, collect about 5 mL of the clear, colorless distillate and transfer to a test tube.

20. Test the solution for completeness of precipitation by adding 2 drops of Phenol Solution. If the solution becomes cloudy or white precipitate forms, residual bromine is still present (proceed to next step). If no cloudiness occurs, the sample is ready for analysis.

21. Transfer the 5-mL aliquot back to the beaker and continue to add Phenol Solution until no turbidity is formed in subsequent 5-mL aliquots.

22. Transfer the entire sample into a 500-mL volumetric flask. Rinse the beaker with deionized water and add to the flask.

23. Dilute to volume with deionized water, stopper and mix well. The distillate is now ready for analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row.

4. Prepare a 100 mg/L selenium standard solution by pipetting 10 mL of 1000 mg/L Selenium Standard Solution into a 100 mL volumetric flask, and diluting to volume with demineralized water.

5. Prepare three sample spikes. Fill three mixing cylinders with 100 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **VIEW: FIT**, then select **IDEAL LINE** and press **OK** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method
Prepare a 0.5-mg/L Se standard solution as follows:

1. Prepare a 100 mg/L selenium standard solution by pipetting 10 mL of 1000 mg/L Selenium Standard Solution into a 100 mL volumetric flask, and diluting to volume with demineralized water. Pipet 1.00 mL of this 100 mg/L standard into a 200 mL volumetric flask. Dilute to volume with deionized water. Transfer 100 mL of the standard into a 500-mL Erlenmeyer flask. Perform the test as described above.

2. To adjust the calibration curve using the reading obtained with the 0.5-mg/L standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

An EDTA masking agent is added to the sample to remove interferences such as iron prior to the test. The addition of a sulfate buffer adjusts the sample to the optimum pH of 1 to 2. Under these conditions, diaminobenzidine reacts with all selenium present as selenite (Se^{4+}) to give a yellow-colored piazselenol complex which is extracted and the color intensity measured colorimetrically. Selenium present as Se^{2+} and Se^{6+} is not detected unless the sample is distilled. Test results are measured at 420 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Selenium Reagent Set (100 tests), includes:			22442-00
(1) Buffer Solution, sulfate type, pH 2.0	10 mL	500 mL	452-49
(1) Diaminobenzidine, tetrahydrochloride	0.1 g	5 g	7062-22
(2) Potassium Hydroxide Standard Solution, 12 N	4 mL	100 mL	230-32
(1) TitraVer® Hardness Reagent, ACS	0.4 g	100 g	204-26
(1) Toluene, ACS	60 mL	4 L	14470-17
Water, deionized	100 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cotton Balls, absorbent	1	100/pkg	2572-01
Cylinder, graduated, 50-mL	1	each	508-41
Cylinder, graduated, 100-mL	1	each	508-42
Dropper, 0.5 & 1.0 mL marks, glass	1	5/pkg	14197-05
Dropper, 0.5 & 1.0 mL marks, plastic	1	20/pkg	21247-20
Flask, Erlenmeyer, 500-mL	2	each	505-49
Funnel, separatory, 250-mL	2	each	520-46
Hot Plate, 4-inch diameter, 120 VAC	1	each	12067-01
OR			
Hot Plate, 4-inch diameter, 240 VAC	1	each	12067-02
Pipet, volumetric, 5-mL	1	each	14515-37

Required Apparatus (continued)

Description	Quantity/Test	Unit	Cat. No.
Pipet filler, safety bulb	1	each	14651-00
Ring, support, (3-inch) 83-mm	1	each	580-00
Sample Cells, 1-inch square, 25 mL with stopper, matched pair	2	2/pkg	26126-02
Spoon, measuring, 0.05-g	1	each	492-00
Spoon, measuring, 0.2-g	1	each	638-00
Support, ring stand, (5 x 8 inch) 127 x 203 mm	1	each	563-00

Distillation Reagents and Apparatus

Description	Unit	Cat. No.
Calcium Chloride Solution	1000 mL	428-53
Hydrochloric Acid Standard Solution, 0.1 N	1000 mL	14812-53
Hydrogen Peroxide, 30%, ACS	473 mL	144-11
Methyl Orange Indicator Solution, (0.50-g/L)	500 mL	148-49
Phenol Solution, 30-g/L	29 mL	2112-20
Potassium Permanganate Standard Solution, 1-g/L	100 mL	14164-42
Sodium Hydroxide Standard Solution, 0.100 N	1000 mL	191-53
Sulfuric Acid Standard Solution, 19.2 N	500 mL	2038-49
Distillation Apparatus Set, general purpose	each	22653-00
Distillation Apparatus Heater, 115 VAC	each	22744-00
Distillation Apparatus Heater, 230 VAC	each	22744-02

Recommended Standards

Description	Unit	Cat. No.
Selenium Standard Solution, 1000-mg/L	100 mL	22407-42

Optional Reagents and Apparatus

Description	Cat. No.
Acetone	14429-49
Nitric Acid	152-49
Sodium Hydroxide, 5.0 N	2450-32
Sulfuric Acid, 5.25 N	2449-32

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Updated November 2005 Edition 1

Silica

Method 8282

Heteropoly Blue Method[1]

Pour-Thru Cell

ULR(3 to 1000 µg/L as SiO_2)

Scope and Application: For testing trace levels of soluble silica in pure and ultrapure water

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test Preparation

Before starting the test:

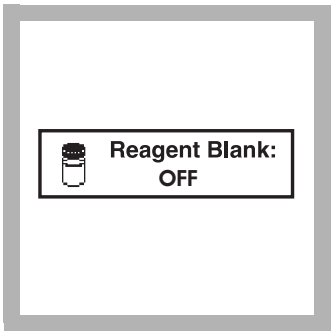
See the user manual for Pour-Thru Module installation instructions.
Clean the Pour-Thru cell and all labware as specified in Labware on page 4
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel.
See Reagent Preparation on page 4 for instructions on preparing the Amino Acid F Reagent.
Reagent blank values printed on analyzer reagent containers vary because the reagents' dilutions vary according to instrument. For this method, use the 1234D analyzer reagent blank value for a 3.78 L volume of Molybdate 3 Reagent. For a Series 5000, 2.9 L volume of Molybdate 3 Reagent, multiply the reagent blank on the label by 1.09. For 100-mL Molybdate 3 Reagent and 1 L Molybdate 3 Reagent, use the lab blank values on the bottle labels.
The four-minute reaction time in step 11 is for samples at 20 °C; for samples at 10 °C, wait eight minutes; for samples at 30 °C, wait two minutes.
The one-minute reaction time in step13 is for samples at 20 °C; for samples at 10 °C, wait two minutes; for samples at 30 °C, wait 30 seconds.

Collect the following items:	**Quantity**
Amino Acid F Reagent Solution	1 mL
Citric Acid F Reagent	1 mL
Molybdate 3 Reagent	1 mL
Cylinder, graduated, 50-mL, poly	1
Flask, Erlenmeyer, 250-mL, PMP, with cap	2
Pipet, TenSette®, 0.1 to 1.0 mL with tips	1
Pour-Thru Cell Module	1

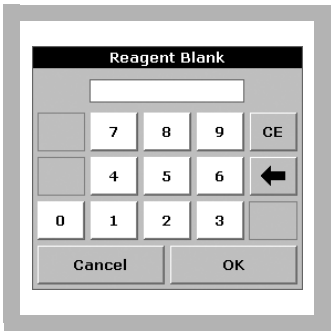
Note: *Reorder information for consumables and replacement items is on page 6.*

Pour-Thru Cell — Method 8282

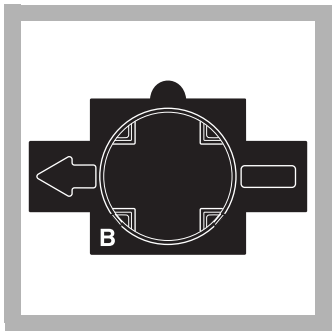
1. Select the test.



2. Account for the Molybdate 3 reagent blank by pressing **OPTIONS>MORE> REAGENT BLANK>ON**.



3. To adjust the reagent blank value, press the existing value and adjust it with the numeric keypad. Press **OK>OK>RETURN**.

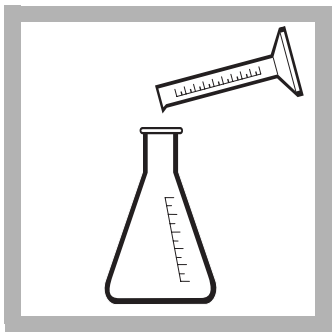


4. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch (round) path in line with the adapter arrow. Flush the Pour-Thru cell with 50 mL of low-silica deionized water.

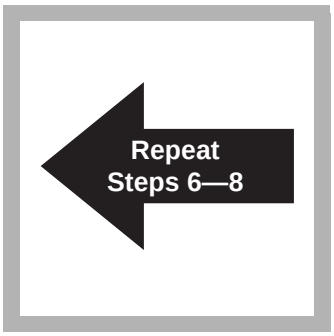
5. Fill two clean 250-mL Erlenmeyer flasks to overflowing with sample.

6. Fill a clean 50-mL plastic graduated cylinder with sample from one of the flasks; then discard the contents of the cylinder. Repeat three times.

7. Fill the rinsed cylinder to the 50-mL mark with sample from the same flask. Discard any remaining sample in the flask.



8. Pour the contents of the 50-mL cylinder back into the original flask.

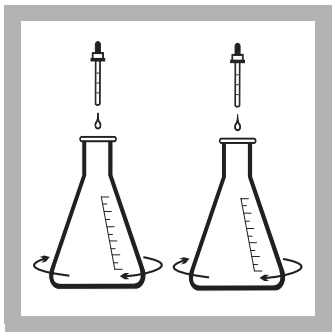


9. Repeat steps 6 through 8 for the second flask containing sample.

10. Use a TenSette® Pipet to add 1.0 mL of Molybdate 3 Reagent to each flask. Swirl to mix.

11. Press **TIMER>OK**.

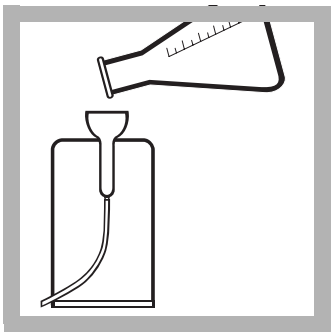
A four-minute reaction period will begin.



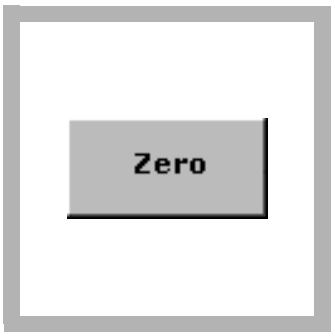
12. When the timer expires, add 1.0 mL of Citric Acid F Reagent to each flask. Swirl to mix.

13. Press **TIMER>OK**.

A one-minute reaction period will begin. The destruction of possible phosphate interference occurs during this period.



14. When the timer expires, pour the contents of one flask into the Pour-Thru Cell.



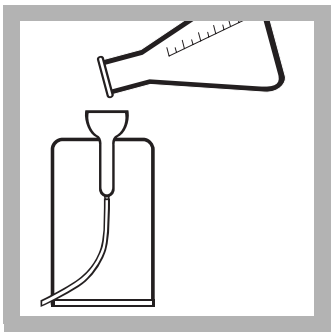
15. After the flow stops, press **ZERO**.

The display will show:

0 µg/L SiO_2

16. Add 1.0 mL of Amino Acid F Reagent to the remaining flask. Swirl to mix.

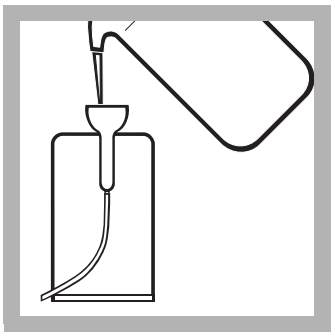
A faint blue color will develop if silica is present.



17. Wait at least 15 seconds, then pour the contents of the second flask into the Pour-Thru Cell.

18. Press **READ**.

Results are in µg/L SiO_2.



19. Flush the Pour-Thru Cell with at least 50 mL of deionized water immediately after use.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample (follow procedure).
Iron	Interferes at high levels.
pH (extreme)	Adjust pH to less than 7.
Phosphate (PO_4^{3-})	Interferes at levels greater than 50 mg/L PO_4^{3-}.
Sulfides	Interfere at all levels.
Turbidity	Eliminated by zeroing the instrument with the original sample (follow procedure).

Sample Collection, Storage, and Preservation

Use only plastic containers with tight-fitting closures. Do not use glass containers; they will contaminate the sample with silica. Soak sampling containers with a solution made of one part Molybdate 3 Reagent to 50 parts of high quality deionized water of low silica concentration. Fill completely and let stand for several hours. Rinse thoroughly with low-level silica water, drain and close. Repeat this cleaning periodically.

Allow the sample stream to flow for 1–2 minutes before collection. Do not adjust the flow during the sampling period as this may introduce particulates. Rinse the container well with sample before collecting the portion for analysis. Analyze as soon as possible.

Reagent Preparation

Amino Acid F Reagent Solution is available in either 100-mL bottles or a package of 20 unit-dose ampules. The bottled reagent is stable for up to one year if the bottle is kept closed when not in use. The ampuled reagent is sealed under argon and is more stable with a shelf life greater than 1 year. Reduced sensitivity at high concentrations (1000 µg/L) indicates reagent instability. Check the bottled reagent on a routine basis by performing an analysis on a 1-mg/L Silica Standard Solution. If the concentration is less than 950 µg/L, use a fresh bottle of Amino Acid F Reagent Solution.

Prepare larger or smaller volumes of Amino Acid F Reagent by dissolving Amino Acid F Reagent Powder in Amino Acid F Reagent Solvent at a ratio of 11 grams per 100 mL of reagent solvent. These reagents are available as the Amino Acid F Reagent Package. This prepared solution has limited stability; test routinely with the 1-mg/L Silica Standard Solution.

Labware

All containers used in this test must be cleansed thoroughly to remove any traces of silica. Use plastic containers for all analysis and storage because glass can contaminate the sample with silica. Small bottles or flasks with screw-type closures work well.

Clean containers by normal means (do not use phosphate detergents), then rinse with high quality deionized water of low-level silica concentration. Soak for 10 minutes with a 1:50 dilution of Molybdate 3 Reagent in low-level silica water. Rinse repeatedly with either low-level silica water or the sample before use. Keep containers tightly closed when not in use. Fill the Pour-Thru Cell with this same mixture of Molybdate 3 and water, and let stand for several minutes before use. Rinse with low-level silica water.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of ammonium hydroxide, followed by several deionized water rinses. Cover the Pour-Thru Cell when it is not in use.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare three samples as instructed in the procedure. Fill three 250-mL Erlenmeyer flasks with 50 mL of sample. Use the TenSette® Pipet to add 0.2 mL, 0.4 mL, and 0.6 mL of 1-mg/L (1000-µg/L) Silica standard, respectively, to each flask and mix thoroughly.

5. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

6. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use the 500-µg/L SiO_2 Standard Solution listed under Recommended Standards in place of the sample. Perform the silica procedure as described above.

2. To adjust the calibration curve using the reading obtained with the 500-µg/L Standard Solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

A number of modifications are necessary to adapt the Low Range Silica method for analyzing trace levels in the Ultra Low Range method. It is absolutely necessary to use the one-inch Pour-Thru Cell and liquid reagents. The Pour-Thru Cell increases the reproducibility of the optics and reduces the instability of the readings that result from moveable sample cells. Liquid reagents produce more reproducible readings and lower blank values by eliminating slight turbidity that may remain when using powdered reagents. Use of liquid reagents in continuous monitors for silica provides a means of confirming the analyzer performance.

Silica and phosphate in the sample react with molybdate ions under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Amino Acid F Reagent is then added to reduce the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration. Test results are measured at 815 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
ULR Silica Reagent Set (using Amino Acid F solution, 100 tests)	—	—	25535-00
Includes: (2) 1995-32, (2) 22542-32, (1) 23864-42			
ULR Silica Reagent Set (using Amino Acid F ampules, 40 tests)	—	—	25814-00
Includes: (1) 1995-32, (1) 22542-32, (2) 23864-20			
Amino Acid F Reagent Solution	1.0 mL	100 mL	23864-42
OR			
Amino Acid F Reagent Solution, 1.2-mL Ampules	1 each	20/pkg	23864-20
Citric Acid Reagent Solution	2 mL	500 mL	22542-49
Molybdate 3 Reagent Solution	2.0 mL	500 mL	1995-49

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 50-mL, poly	1	each	1081-41
Flask, Erlenmeyer, 250-mL, PMP, w/cap	2	each	20898-46
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	5	50/pkg	21856-96
Pour-Thru Cell Kit	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Silica Standard Solution, 1-mg/L SiO_2	500 mL	1106-49
Silica Standard Solution, 10-mg/L SiO_2	500 mL	1403-49
Silica Standard Solution, 500-µg/L SiO_2	3.78 L	21008-17
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Ammonium Hydroxide, 58%	500 mL	106-49
Molybdate 3 Reagent	2.9 L	1995-03
Molybdate 3 Reagent	3.78 L	1995-17
Molybdate 3 Reagent	100 mL	1995-32
Molybdate 3 Reagent	1 L	1995-53

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Updated November 2005 Edition 1

Silica

Method 8282
Pour-Thru Cell

Heteropoly Blue Rapid Liquid Method[1]
ULR(3 to 1000 µg/L as SiO_2)

Scope and Application: For testing trace levels of soluble silica in pure and ultrapure water

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

Before starting the test:

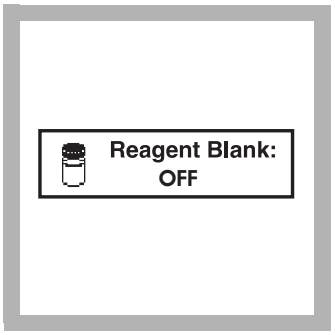
See the User Manual for Pour-Thru Module installation instructions.
Clean the Pour-Thru cell and all labware as specified in Labware on page 4
Protect the Pour-Thru Cell from contamination when not in use by inverting a small beaker over the top of the glass funnel.
See Reagent Preparation on page 4 for instructions on preparing the Amino Acid F Reagent.
Reagent blank values printed on analyzer reagent containers vary because the reagents' dilutions vary according to instrument. For this method, use the 1234D analyzer reagent blank value for a 3.78 L volume of Molybdate 3 Reagent. For a Series 5000, 2.9 L volume of Molybdate 3 Reagent, multiply the reagent blank on the label by 1.09. For 100-mL Molybdate 3 Reagent and 1 L Molybdate 3 Reagent, use the lab blank values on the bottle labels.
The four-minute reaction time in step 11 is for samples at 20 °C; for samples at 10 °C, wait eight minutes; for samples at 30 °C, wait two minutes.
The one-minute reaction time in step13 is for samples at 20 °C; for samples at 10 °C, wait two minutes; for samples at 30 °C, wait 30 seconds.

Collect the following items:	**Quantity**
Amino Acid F Reagent Powder	varies
Amino Acid Reagent Dilution Solvent	varies
Citric Acid F Reagent	1 mL
Molybdate 3 Reagent	1 mL
Cylinder, graduated, 50-mL, poly	1
Dispenser, fixed volume, 1.0-mL, w/bottle	3
Flask, Erlenmeyer, 250-mL, PMP, w/cap	2
Pour-Thru Cell Module	1

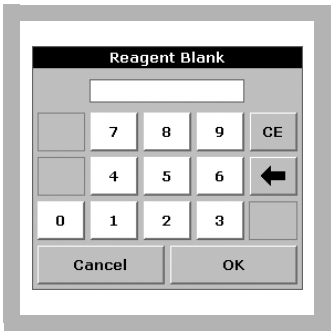
Note: *Reorder information for consumables and replacement items is on page 5.*

Pour-Thru Cell — Method 8282

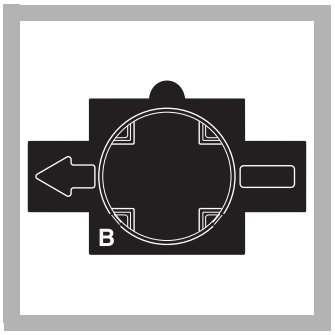
1. Select the test.



2. Account for the Molybdate 3 reagent blank by pressing **OPTIONS>MORE> REAGENT BLANK>ON**.



3. To adjust the reagent blank value, press the existing value and adjust it with the numeric keypad. Press **OK>OK>RETURN**.

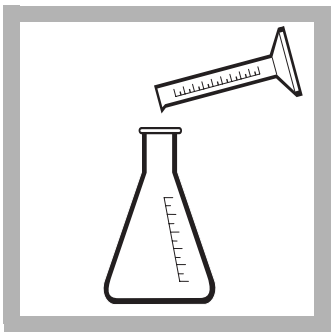


4. Insert Adapter B. Install the Pour-Thru Cell with the 1-inch (round) path in line with the adapter arrow. Flush the Pour-Thru cell with 50 mL of low-silica deionized water.

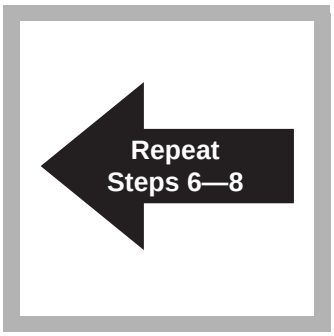
5. Fill two clean 250-mL Erlenmeyer flasks to overflowing with sample.

6. Fill a clean 50-mL plastic graduated cylinder with sample from one of the flasks; then discard the contents of the cylinder. Repeat three times.

7. Fill the rinsed cylinder to the 50-mL mark with sample from the same flask. Discard any remaining sample in the flask.



8. Pour the contents of the 50-mL cylinder back into the original flask.



9. Repeat *step 6* through *step 8* for the second flask containing sample.

10. Add 1.0 mL of Molybdate 3 Reagent to each flask. Swirl to mix.

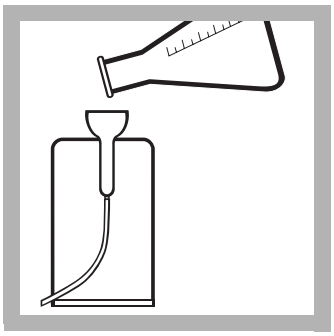
11. Press **TIMER>OK**.

A four-minute reaction period will begin.

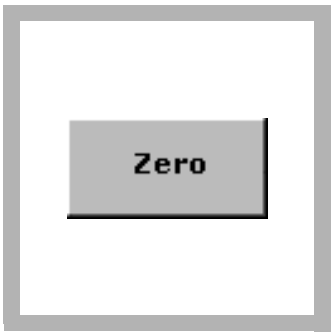
12. When the timer expires, add 1.0 mL of Citric Acid F Reagent to each flask. Swirl to mix.

13. Press **TIMER>OK**.

A one-minute reaction period will begin. The destruction of possible phosphate interference occurs during this period.



14. When the timer expires, pour the contents of one flask into the Pour-Thru Cell.



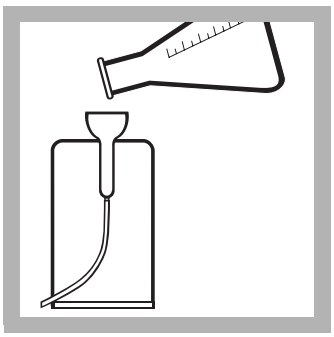
15. After the flow stops, press **ZERO**.

The display will show:

0 µg/L SiO_2

16. Add 1.0 mL of Amino Acid F Reagent to the remaining flask. Swirl to mix.

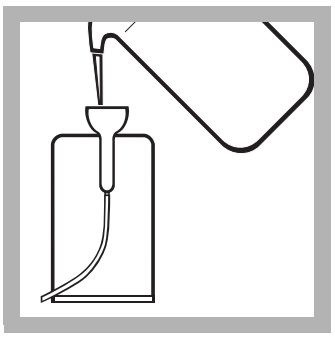
A faint blue color will develop if silica is present.



17. Wait at least 15 seconds, then pour the contents of the second flask into the Pour-Thru Cell.

18. Press **READ**.

Results are in µg/L SiO_2.



19. Flush the Pour-Thru Cell with at least 50 mL of deionized water immediately after use.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample (follow procedure).
Iron	Interferes at high levels.
pH (extreme)	Adjust pH to less than 7.
Phosphate (PO_4^{3-})	Interferes at levels greater than 50 mg/L PO_4^{3-}.
Sulfides	Interfere at all levels.
Turbidity	Eliminated by zeroing the instrument with the original sample (follow procedure).

Sample Collection, Storage, and Preservation

Use only plastic containers with tight-fitting closures. Do not use glass containers; they will contaminate the sample with silica. Soak sampling containers with a solution made of one part Molybdate 3 Reagent to 50 parts of high quality deionized water of low silica concentration. Fill completely and let stand for several hours. Rinse thoroughly with low-level silica water, drain and close. Repeat this cleaning periodically.

Allow the sample stream to flow for 1–2 minutes before collection. Do not adjust the flow during the sampling period as this may introduce particulates. Rinse the container well with sample before collecting the portion for analysis. Analyze as soon as possible.

Reagent Preparation

Dissolve the contents of one bottle of Amino Acid F Reagent Powder in one bottle of Amino Acid Reagent Dilution Solvent. Install a bottle-top dispenser on this bottle, as well as on the Molybdate 3 Reagent and Citric Acid Reagent bottles.

Prepare smaller volumes of Amino Acid F Reagent by dissolving Amino Acid F Reagent Powder in Amino Acid F Reagent Solvent at a ratio of 11 grams per 100 mL of reagent solvent. This prepared solution has limited stability; test routinely with the 1-mg/L Silica Standard Solution.

Reduced sensitivity at high concentrations (1000 µg/L) indicates reagent instability. Check the reagent on a routine basis by performing an analysis on a 1-mg/L Silica Standard Solution. If the concentration is less than 950 µg/L, use fresh Amino Acid F Reagent Solution.

Labware

All containers used in this test must be cleansed thoroughly to remove any traces of silica. Use plastic containers for all analysis and storage because glass can contaminate the sample with silica. Small bottles or flasks with screw-type closures work well.

Clean containers by normal means (do not use phosphate detergents), then rinse with high quality deionized water of low-level silica concentration. Soak for 10 minutes with a 1:50 dilution of Molybdate 3 Reagent in low-level silica water. Rinse repeatedly with either low-level silica water or the sample before use. Keep containers tightly closed when not in use. Fill the Pour-Thru Cell with this same mixture of Molybdate 3 and water, and let stand for several minutes before use. Rinse with low-level silica water.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of colored products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of ammonium hydroxide, followed by several deionized water rinses. Cover the Pour-Thru Cell when it is not in use.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Prepare three samples as instructed in the procedure. Fill three 250-mL Erlenmeyer flasks with 50 mL of sample. Use the TenSette® Pipet to add 0.2 mL, 0.4 mL, and 0.6 mL of 1-mg/L (1000-µg/L) Silica standard, respectively, to each flask and mix thoroughly.

5. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

6. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use the 500-µg/L SiO_2 Standard Solution listed under Recommended Standards in place of the sample. Perform the silica procedure as described above.

2. To adjust the calibration curve using the reading obtained with the 500-µg/L Standard Solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration (the value depends on the selected chemical form). If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

A number of modifications are necessary to adapt the Low Range Silica method for analyzing trace levels in the Ultra Low Range method. It is absolutely necessary to use the one-inch Pour-Thru Cell and liquid reagents. The Pour-Thru Cell increases the reproducibility of the optics and reduces the instability of the readings that result from moveable sample cells. Liquid reagents produce more reproducible readings and lower blank values by eliminating slight turbidity that may remain when using powdered reagents. Use of liquid reagents in continuous monitors for silica provides a means of confirming the analyzer performance.

Silica and phosphate in the sample react with molybdate ions under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Amino Acid F Reagent is then added to reduce the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration. Test results are measured at 815 nm.

Consumables and Replacement Items

Required Reagent

Description	Quantity/Test	Unit	Cat. No.
Rapid Liquid ULR Silica Reagent Set, includes:			26785-00
Amino Acid F Reagent Powder	varies	55 g	26511-55
Amino Acid Reagent Dilution Solvent	varies	475 mL	23530-11
Citric Acid F Reagent	1 mL	500 mL	22542-49
Molybdate 3 Reagent	1 mL	500 mL	1995-49

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Adapter, for Multi-path and Pour-Thru cells	1	each	LZV585
Cylinder, graduated, 50-mL, poly	1	each	1081-41
Dispenser, fixed volume, 1.0-mL, w/bottle	3	each	21113-02
Flask, Erlenmeyer, 250-mL, PMP, w/cap	2	each	20898-46

Silica ULR(3 to 1000 µg/L as SiO_2)

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Pour-Thru Cell Kit	1	each	59404-00

Recommended Standards

Description	Unit	Cat. No.
Silica Standard Solution, 1-mg/L SiO_2	500 mL	1106-49
Silica Standard Solution, 10-mg/L SiO_2	500 mL	1403-49
Silica Standard Solution, 500-µg/L SiO_2	3.78 L	21008-17
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Ammonium Hydroxide 58%	500 mL	106-49
Molybdate 3 Reagent	2.9 L	1995-03
Molybdate 3 Reagent	3.78 L	1995-17
Molybdate 3 Reagent	100 mL	1995-32
Molybdate 3 Reagent	1 L	1995-53
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet tips for TenSette Pipet 19700-01	50/pkg	21856-96

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Updated November 2005 Edition 1

Silica

Method 8186 — Heteropoly Blue Method[1]

Powder Pillows — LR (0.010 to 1.600 mg/L as SiO_2)

Scope and Application: For water and seawater

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

Test Preparation

Before starting the test:

The four-minute reaction time in step 5 is for samples at 20 °C; for samples at 10 °C, wait eight minutes; for samples at 30 °C, wait two minutes.
The one-minute reaction time in step 7 is for samples at 20 °C; for samples at 10 °C, wait two minutes; for samples at 30 °C, wait 30 seconds.
If testing for very low levels of silica, use Method 8282.

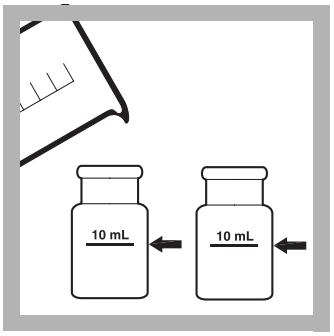
Collect the following items:	**Quantity**
Amino Acid F Reagent Powder Pillows (for 10-mL sample)	1 pillow
Citric Acid Powder Pillows	2 pillows
Molybdate 3 Reagent Solution	1 mL
Sample Cells, 1-inch square, 10 mL, matched pair	2

Note: *Reorder information for consumables and replacement items is on page 4.*

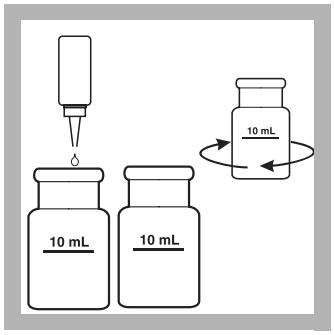
Powder Pillows — Method 8186

1. Press **STORED PROGRAMS**.

2. Select the test.



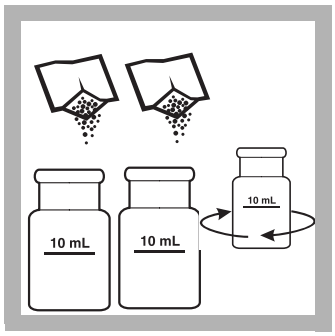
3. Fill two square sample cells with 10 mL of sample.



4. Add 14 drops of Molybdate 3 Reagent to each sample cell. Swirl to mix.

5. Press **TIMER>OK**.

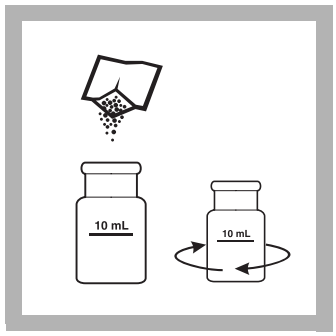
A four-minute reaction period will begin.



6. When the timer expires, add the contents of one Citric Acid Reagent Powder Pillow to each sample cell. Swirl to mix.

7. Press **TIMER>OK**.

A one-minute reaction period will begin. The destruction of possible phosphate interference occurs during this period.



8. Prepared Sample: When the timer expires, add the contents of one Amino Acid F Reagent Powder Pillow to one of the sample cells. Swirl to mix.

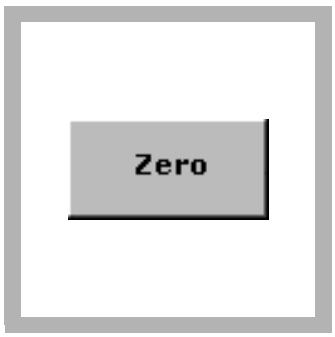
Blank Preparation: The sample without the Amino Acid F Reagent is the blank.

9. Press **TIMER>OK**.

A two-minute reaction period will begin.

A blue color will develop if silica is present.

10. When the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.



11. Press **Zero**.

The display will show:

0.000 mg/L SiO_2

12. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L SiO_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	Large amounts interfere.
Phosphate	Does not interfere at levels less than 50 mg/L PO_4. At 60 mg/L PO_4, an interference of –2% occurs. At 75 mg/L PO_4 the interference is –11%.
Slow reacting forms of silica	Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these “molybdate-unreactive” forms is not known. A pretreatment with Sodium Bicarbonate, then Sulfuric Acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help instead of the bicarbonate pretreatment.
Sulfides	Interfere at all levels.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 7 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open a 25-mg/L Silica Standard Solution Bottle.
5. Prepare three sample spikes. Fill three sample cells with 10 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

1. Use the 1.00-mg/L SiO_2 Standard Solution listed under Consumables and Replacement Items on page 4 in place of the sample. Perform the silica procedure as described above.

2. To adjust the calibration curve using the reading obtained with the 1.00 mg/L Standard Solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. An Amino Acid is then added to reduce the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration. Test results are measured at 815 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Low Range Silica Reagent Set (100 tests), includes:	—	—	24593-00
(1) Amino Acid F Reagent Powder Pillows (for 10-mL sample)	1 pillow	100/pkg	22540-69
(2) Citric Acid Powder Pillows	2 pillows	100/pkg	21062-69
(2) Molybdate 3 Reagent Solution	1 mL	50 mL	1995-26

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standard

Description	Unit	Cat. No.
Deionized Water	4 L	272-56
Silica Standard Solution, 1-mg/L SiO_2	500 mL	1106-49
Silica Standard Solution, 25-mg/L as SiO_2	236 mL	21225-31

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Sodium Bicarbonate	454 g	776-01
Sulfuric Acid, 1.00 N	1000 mL	1270-53
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet Tips for TenSette Pipet 19700-01	50/pkg	21856-96

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Updated November 2005 Edition 1

Silica

Method 8185
Powder Pillows

Silicomolybdate Method
HR (1 to 100 mg/L)

Scope and Application: For water and seawater

Test Preparation

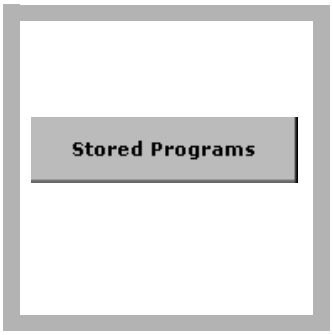
Before starting the test:

Sample temperature should be 15–25 °C (59–77 °F)

Collect the following items:	**Quantity**
High Range Silica Reagent Set	1
Deionized Water	10 mL
Sample Cell, 1-inch square, 10-mL	2

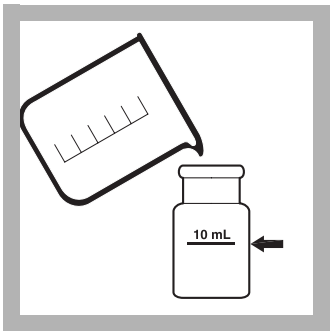
Note: *Reorder information for consumables and replacement items is on page 4.*

Powder Pillows Method 8185

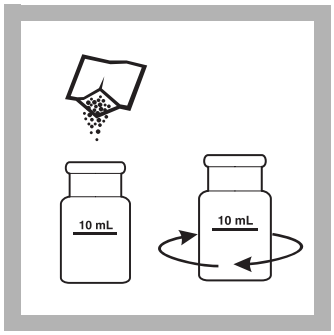


1. Press **STORED PROGRAMS**.

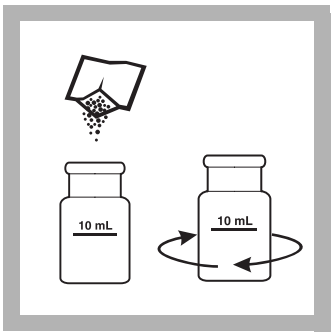
2. Select the test.



3. Fill a square sample cell with 10-mL of sample.



4. Prepared Sample: Add the contents of one Molybdate Reagent Powder Pillow for High Range Silica to the sample cell. Swirl until completely dissolved.

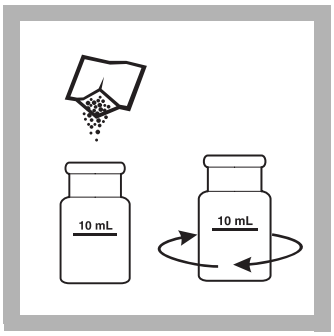


5. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. Swirl to mix.

A yellow color will develop if silica or phosphorus is present.

6. Press **TIMER>OK**.

A ten-minute reaction period will begin.



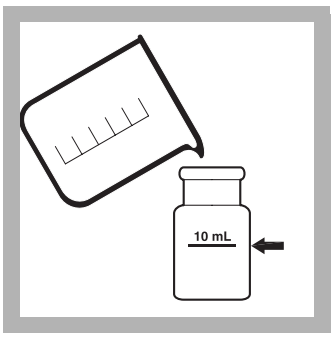
7. When the timer expires, add the contents of one Citric Acid Powder Pillow to the sample cell. Swirl to mix.

Any yellow color due to phosphorus is removed in this step.

8. Press **TIMER>OK**.

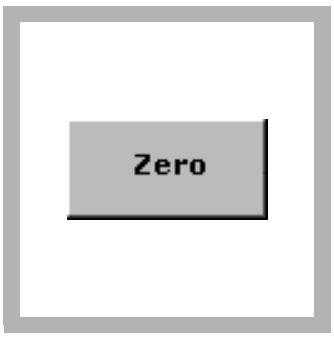
A two-minute reaction period will begin.

Perform steps 9–12 within three minutes after the timer expires.



9. Blank Preparation: Fill a second sample cell with 10 mL of the original sample.

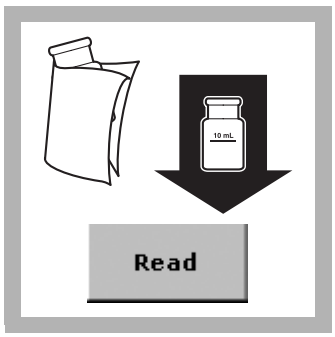
10. Wipe the blank and insert the blank into the cell holder with the fill line facing right.



11. Press **ZERO**.

The display will show:

0 mg/L SiO_2



12. Wipe the prepared sample and insert the prepared sample into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L SiO_2.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	High levels of Fe^{2+} and Fe^{3+} interfere.
Phosphate	Does not interfere below 50 mg/L PO_4^{3-}. At 60 mg/L PO_4^{3-}, a negative 2% interference occurs. At 75 mg/L PO_4^{3-}, the interference is negative 11%.
Sulfides (S^{2-})	All levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these “molybdate-unreactive” forms is not known. A pretreatment with Sodium Bicarbonate*, then Sulfuric Acid† will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help instead of the bicarbonate treatment.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples at 4 °C (39 °F) for up to 28 days. Warm samples to room temperature before analyzing.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Open a 1000 mg/L Silica Standard Solution.

5. Prepare three sample spikes. Fill three sample cells with 10 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery

Standard Solution Method

1. To check test accuracy, use the 50-mg/L Silica Standard Solution. Analyze according to the HR Silica procedure described above using deionized water as the blank.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 4.
† See Optional Reagents and Apparatus on page 4.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color. Test results are measured at 452 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
High Range Silica Reagent Set for 10-mL samples (100 tests), includes:			24296-00
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	21074-69
Citric Acid Powder Pillows	1	100/pkg	21062-69
Molybdate Reagent Powder Pillows for High Range Silica	1	100/pkg	21073-69
Water, deionized	10 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Silica Standard Solution, 50-mg/L	200 mL	1117-29
Silica Standard Solution, 1000-mg/L	500 mL	194-49

Optional Reagents and Apparatus

Description	Cat. No.
Sodium Bicarbonate, 454 grams	776-01
Sulfuric Acid 1.00 N, 100 mL	1270-32

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Updated November 2005 Edition 1

Silver

Method 8120 Colorimetric Method
Powder Pillows (0.005 to 0.700 mg/L)

Scope and Application: For water and wastewater.

Test Preparation

Before starting the test:

Digestion is required for samples with interferences. See Digestion on page 5.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
The graduated cylinder must be completely dry before beginning the test. If the Silver 1 Powder becomes moist, it will not dissolve completely, which will inhibit color development.
The sample pH for this test must be between 9 and 10. Do not use a pH meter to adjust the sample pH as it will cause contamination. See Digestion on page 5 for the procedure to adjust pH.
Generate a blank for each sample.

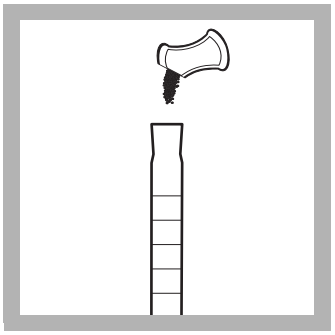
Collect the following items:	**Quantity**
Silver 1 Reagent Powder Pillow	1
Silver 2 Reagent Powder Pillow	1
Sodium Thiosulfate Powder Pillow	1
Clippers	1
Cylinder, graduated, 50-mL	1
Cylinder, graduated mixing, 50-mL	1
Sample Cells, 10-mL square, 1-inch	2

Note: *Reorder information for consumables and replacement items is on page 7.*

Powder Pillows — Method 8120

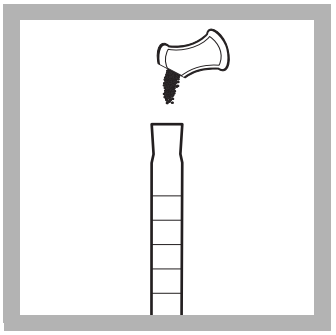
1. Press **STORED PROGRAMS**.

2. Select the test.



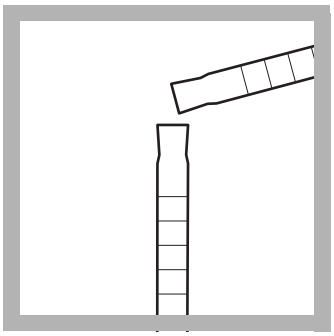
3. Add the contents of one Silver 1 Powder Pillow to a dry 50-mL graduated mixing cylinder.

If the Silver 1 Powder becomes wet at this point, the powder will not dissolve completely, which will inhibit color development.

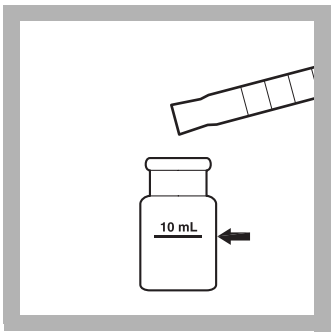


4. Add the contents of one Silver 2 Reagent Solution Pillow to the cylinder. Swirl to completely wet the powder.

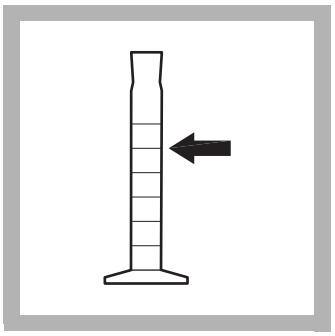
If clumps of dry powder are present when the sample is poured in, the powder will not dissolve completely, which will inhibit color development.



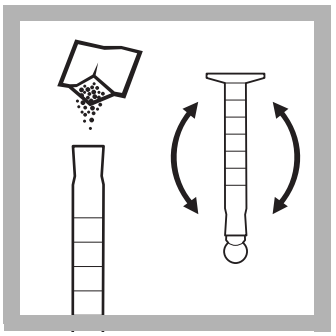
5. Use a 50-mL graduated cylinder to add 50 mL of sample to the 50-mL graduated mixing cylinder. Stopper and invert repeatedly for one minute.



6. Prepared Sample: Fill a square sample cell to the 10-mL mark with the mixture.



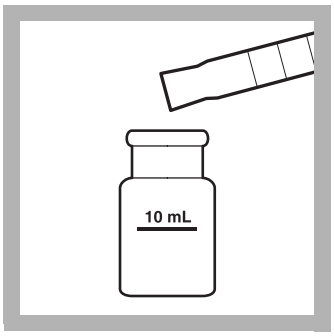
7. Blank Preparation: Discard all but 25-mL of the sample from step 5.



8. Add the contents of one Sodium Thiosulfate Reagent Powder Pillow to the remaining 25-mL of sample. Stopper and invert to mix.

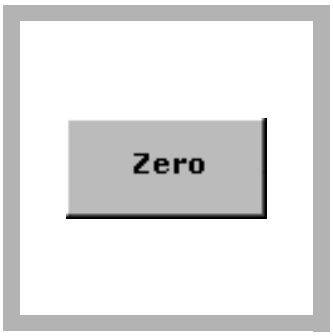
9. Press **TIMER>OK.**

A two-minute reaction period will begin.



10. Pour 10 mL of the blank into a second square sample cell.

11. When the timer expires, insert the blank into the cell holder with the fill line facing right.



12. Press **ZERO**.

The display will show:

0.000 mg/L Ag

13. insert the prepared sample into the cell holder with the fill line facing right.

14. Press **READ**.

Results are in mg/L Ag.

Interferences

Interference studies were conducted by preparing a known silver solution (about 0.4 mg/L) and the potential interfering ion. The ion was said to interfere when the resulting concentration changed by ±10%.

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels
Aluminum	Negative interference above 30 mg/L
Ammonia	Negative interference above 750 mg/L
Cadmium	Negative interference above 15 mg/L
Calcium	Positive interference above 600 mg/L
Chloride	Negative interference above 19 mg/L
Chromium^{6+}	Negative interference above 90 mg/L
Copper	Negative interference above 7 mg/L
Iron	Negative interference above 30 mg/L
Lead	Negative interference above 13 mg/L
Manganese	Negative interference above 19 mg/L
Magnesium	Positive interference above 2000 mg/L

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels
Mercury	Positive interference above 2 mg/L
Nickel	Negative interference above 19 mg/L
Zinc	Negative interference above 70 mg/L

Sample Collection, Storage, and Preservation

Collect samples in acid-cleaned glass or plastic bottles. Using pH paper, adjust the pH to 2 or less with concentrated Nitric Acid* (about 2 mL/liter). Store preserved samples at room temperature for up to 6 months. If the sample contains particulates or only dissolved metal content is being determined, filter through a 0.45 µm filter at collection. After filtration, adjust the pH to 2 or less as described above for storage.

Before analysis, adjust the pH to 9–10 with 5.0 N Sodium Hydroxide*. (See steps 13–14 of Digestion on page 5.) Do not use a pH meter because of silver contamination from the electrode.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Add 5.00 mL of 1000 mg/L Silver Standard Solution to a 100-mL volumetric Class A flask. Dilute to volume with deionized water. This is a 50.0 mg/L standard solution.
5. Prepare three sample spikes. Fill three mixing cylinders* with 50-mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.
7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method

Prepare a 0.5 mg/L silver standard solution as follows:

1. Pipet 0.50 mL of Silver Standard Solution, 1000 mg/L, into a 1000-mL volumetric flask using a Class A volumetric pipet. Dilute to the mark with deionized water. Prepare this solution daily. Perform the silver procedure as described above.

* See Optional Reagents and Apparatus on page 7.

2. To adjust the calibration curve using the reading obtained with the 0.5-mg/L silver standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **OK** to accept the displayed concentration. If an alternate concentration is used, press the number to enter the actual concentration. Press **OK**. Press **ADJUST**.

Digestion

This digestion is for samples containing organic matter, thiosulfate or cyanide. Possible sources for these compounds are wastewater, silver electroplating baths and silver strike solutions. Digestion should be done with a Digesdahl Digestion Apparatus.

DANGER
Perform this digestion under a fume hood. Poisonous hydrogen cyanide gas may be generated.

CAUTION
Always wear safety glasses and use a safety shield, or operate the Digesdahl within a closed fume hood. Follow the additional safety precautions in the Digesdahl Digestion Apparatus Manual.

1. Add an appropriate size sample to the 100-mL digestion flask for use with the Digesdahl. Add several boiling chips to prevent bumping.

Note: *Appropriate sample size is determined experimentally. The final sample concentration (after dilution to 100 mL) should be 0–0.6 mg/L. Larger dilutions may be necessary for electroplating baths and silver strike solutions. Do not exceed the maximum sample volume of 25 mL. Several 25-mL aliquots may be digested in succession to concentrate a very dilute sample.*

2. Turn on the water aspirator and make sure there is suction in the fractionating head.

3. Add 3 mL of concentrated sulfuric acid to the sample in the volumetric flask. Immediately place the head on the digestion flask. Never use less than 3 mL of acid.

4. Place the digestion flask on the heater. Turn the temperature dial to 440 °C (825 °F).

5. After the sample begins to char or the sulfuric acid reflux line becomes visible, wait 3–5 minutes.

6. Visually confirm the presence of acid in the flask before adding hydrogen peroxide!

7. Add 10 mL of 50% hydrogen peroxide to the sample via the capillary funnel in the fractionating head.

8. After the hydrogen peroxide has boiled off, heat the sample until heavy white sulfuric acid fumes are present. Continue heating and reduce the sample volume to near dryness. Do not let the sample go completely dry at any time.

Note: *If the sample evaporates, turn the Digesdahl off and cool completely. Add water to flask before handling. Repeat digestion from the beginning.*

Note: *If only thiosulfate is present in the sample, proceed to step 1 of the Colorimetric procedure.*

9. Add another 3 mL of sulfuric acid via the capillary funnel.

10. Add another 5 mL of hydrogen peroxide. Check the solution for digestion completion. If digestion is not complete, continue adding hydrogen peroxide in 5 to 10 mL portions. Several portions may be necessary.

Note: *Digestion is complete when the digestate is colorless or the color of the digestate does not change upon addition of hydrogen peroxide. Also, a completely digested sample will not foam.*

11. After digestion is complete and all the hydrogen peroxide is boiled off, reduce the volume of the digestate to near dryness. Do not allow the sample to become completely dry. Remove the flask from the heater. Cool to room temperature.

12. Slowly add about 25 mL of deionized water to the cooled flask.

13. Add 2 drops of 1 g/L Phenolphthalein Indicator Solution. Add 2 drops of 1 g/L Thymolphthalein Indicator Solution.

14. Using sodium hydroxide, adjust the pH of the solution to 9–10. The solution will be pink in this pH range.

Note: *A purple color indicates a pH greater than 10. If this occurs, add a drop of sulfuric acid and 2 drops of each indicator; repeat pH adjustment. Initially, use 50% sodium hydroxide, then 1 N sodium hydroxide as the end point is approached.*

15. Filter turbid digestates. Quantitatively transfer the filtrate (or unfiltered sample) to a clean 100-mL volumetric flask. Dilute to the mark with deionized water. The sample is ready for analysis.

Summary of Method

Silver ions in basic solution react with cadion 2B to form a green to brown to red-purple complex. The sodium thiosulfate acts as a decolorizing agent for the blank. The Silver 1 and Silver 2 reagents contain the buffer, indicator, and masking agents. Organic extractions are not necessary and this method does not have as many interferences as the traditional dithizone method. Test results are measured at 560 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Silver Reagent Set (50 tests), includes:			22966-00
Silver 1 Reagent Powder Pillow	1	50/pkg	22935-66
Silver 2 Reagent Solution Pillow	1	50/pkg	22936-66
Sodium Thiosulfate Powder Pillow	1	50/pkg	22937-66

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers	1	each	968-00
Cylinder, graduated, 50-mL	1	each	21179-41
Cylinder, graduated, mixing, 50-mL	1	each	1896-41
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Digestion Reagents and Apparatus

Description	Unit	Cat. No.
Hydrogen Peroxide, 50%	490 mL	21196-49
Phenolphthalein Indicator Solution, 1 g/L	15 mL SCDB	1897-36
Sodium Hydroxide Solution, 50%	500 mL	2180-49
Sodium Hydroxide Solution, 1.00 N	100 mL MDB	1045-32
Sulfuric Acid, ACS, concentrated	2.5 L	979-09
Thymolphthalein Indicator Solution, 1 g/L	15 mL SCDB	21853-36
Water, deionized	4 L	272-56
Boiling Chips, silicon carbide	500 g	20557-34
Digesdahl Digestion Apparatus, 115 V ac, 50/60 Hz	each	23130-20
Digesdahl Digestion Apparatus, 230 V ac, 50/60 Hz	each	23130-21
Safety Shield, for Digesdahl	each	50030-00

Recommended Standards

Description	Unit	Cat. No.
Silver Standard Solution, 1000 mg/L Ag	100 mL	14613-42
Water, deionized	4 L	272-56

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Cylinder, mixing	50 mL	1896-41
Nitric Acid, concentrated ACS	500 mL	152-49
Sodium Hydroxide, 5.0 N	100 mL	2450-32

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Updated November 2005 Edition 1

Sulfate

★Method 8051 — SulfaVer 4 Method[1]

Powder Pillows or AccuVac® Ampuls — (2 to 70 mg/L)

Scope and Application: For water, wastewater, and seawater; USEPA accepted for reporting wastewater analyses

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*. Procedure is equivalent to USEPA method 375.4 for wastewater.

Test Preparation

Before starting the test:

Adjust the standard curve for each new lot of reagent (Standard Solutions on page 4).
For best results, perform a new calibration for each lot of reagent (Calibration Standard Preparation on page 5).
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Filter highly colored or turbid samples using filter paper[1] and a funnel[1]. Use this sample in step 3 and 6. Undissolved reagent powder that has settled does not affect accuracy.
SulfaVer® 4 contains barium chloride. The final solution will contain barium chloride (D005) at a concentration regulated as a hazardous waste by the Federal RCRA. Refer to a current MSDS for safe handling and disposal instructions.

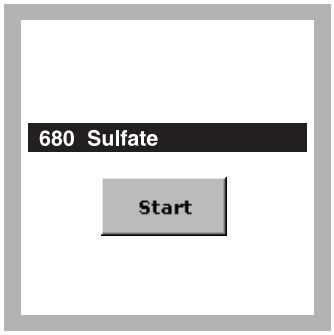
Collect the following items:	**Quantity**
Powder Pillow Test:	
SulfaVer® 4 Reagent Powder Pillows	1
Sample Cells, 1-inch square, 10-mL	2
AccuVac Test:	
SulfaVer® 4 Reagent AccuVac® Ampuls	1
Beaker, 50-mL	1
Sample Cell, 10-mL round, with cap	1
Stopper for 18 mm Tube	1

Note: *Reorder information for consumables and replacement items is on page 5.*

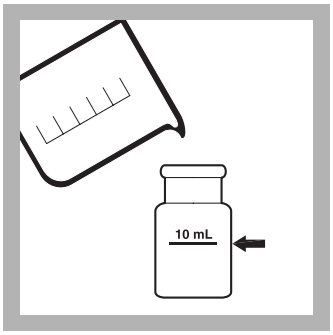
[1] See Optional Reagents and Apparatus on page 5.

Powder Pillows — Method 8051

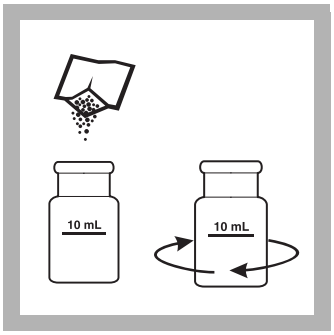
1. Press **STORED PROGRAMS**.



2. Select the test.



3. Fill a square sample cell with 10 mL of sample.

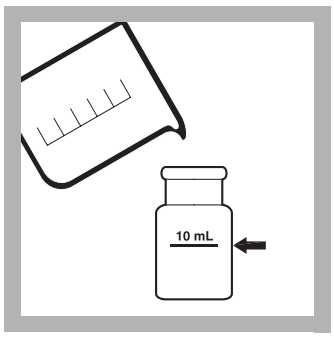


4. Prepared Sample: Add the contents of one SulfaVer 4 Reagent Powder Pillow to the sample cell. Swirl vigorously to dissolve powder.

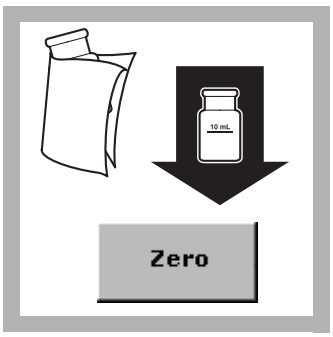
White turbidity will form if sulfate is present.

5. Press **TIMER>OK**.

A five-minute reaction period will begin. Do not disturb the cell during this time.



6. Blank Preparation: Fill a second square sample cell with 10 mL of sample.

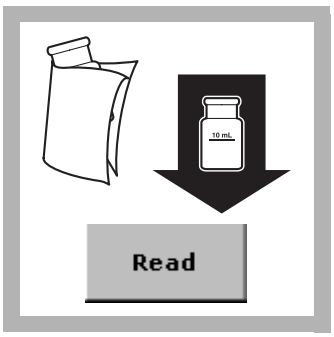


7. When the timer expires, insert the blank into the cell holder with the fill line facing right.

Press **ZERO**.

The display will show:

0 mg/L SO_4^{2-}

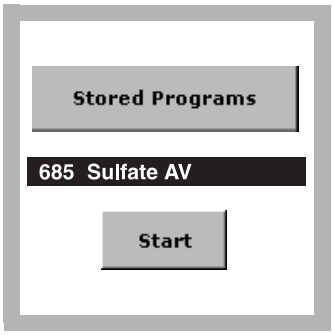


8. Within five minutes after the timer expires, insert the prepared sample into the cell holder with the fill line facing right.

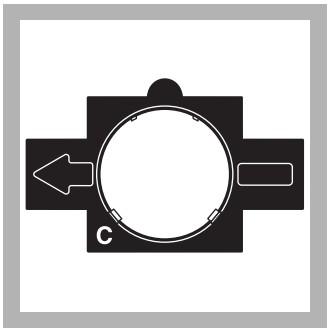
Press **READ**. Results are in mg/L SO_4^{2-}.

Clean sample cells with a soap and brush.

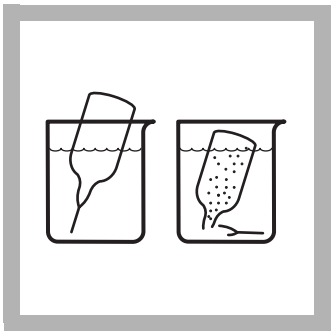
AccuVac Ampul — Method 8051



1. Select the test.

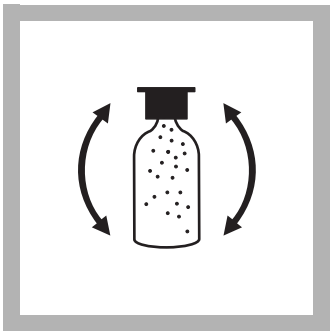


2. Insert Adapter C.



3. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample. Keep the tip immersed until the Ampul fills completely.

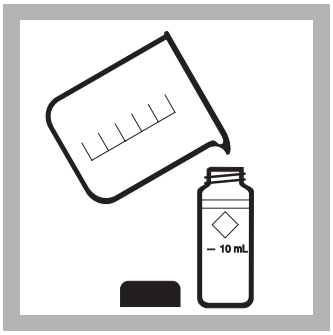


4. Quickly invert the Ampul several times to mix.

White turbidity will form if sulfate is present.

5. Press **TIMER>OK**.

A five-minute reaction period will begin. Do not disturb the cell during this time.



6. Fill a clean sample cell with 10 mL of sample.

7. When the timer expires, insert the blank into the cell holder.

Press **ZERO**.

The display will show:

0 mg/L SO_4^{2-}

8. Within five minutes after the timer expires, insert the Ampul into the cell holder.

Press **READ**. Results are in mg/L SO_4^{2-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Calcium	Greater than 20,000 mg/L as $CaCO_3$
Chloride	Greater than 40,000 mg/L as Cl^-
Magnesium	Greater than 10,000 mg/L as $CaCO_3$
Silica	Greater than 500 mg/L as SiO_2

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Samples may be stored up to 7 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Sulfate Ampule Standard, 2500-mg/L sulfate.

5. Prepare three sample spikes. Fill three mixing cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Transfer 10 mL of each sample spike to a clean sample cell and analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

Note: *For AccuVac® Ampuls, fill three Mixing Cylinders* with 50 mL of sample and spike with 0.2 mL, 0.4 mL, and 0.6 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL Beakers†. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solutions
Prepare a 70-mg/L sulfate standard solution as follows:

1. Using Class A glassware, Pipet 7 mL of Sulfate Standard Solution, 1000-mg/L, into a 100-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the SulfaVer procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

* See Optional Reagents and Apparatus on page 5.
†See Required Apparatus (AccuVac) on page 5.

Calibration Standard Preparation

To perform a sulfate calibration using the SulfaVer method, use Class A glassware to prepare calibration standards containing 10, 20, 30, 40, 50, 60, and 70 mg/L SO_4^{2-} as follows:

1. Into seven different 100-mL Class A volumetric flasks, pipet 1, 2, 3, 4, 5, 6, and 7 mL of the 1000-mg/L Sulfate Standard Solution.
2. Dilute to the mark with deionized water. Mix thoroughly.
3. Using the SulfaVer method and the calibration procedure described in the User Programs section of the user manual, generate a calibration curve from the calibration standards prepared above.

Summary of Method

Sulfate ions in the sample react with barium in the SulfaVer 4 and form a precipitate of barium sulfate. The amount of turbidity formed is proportional to the sulfate concentration. Test results are measured at 450 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
SulfaVer® 4 Reagent Powder Pillows	1	100/pkg	21067-69
OR			
SulfaVer® 4 Sulfate Reagent AccuVac® Ampuls	1	25/pkg	25090-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVac)

Description	Quantity/Test	Unit	Cat. No.
Adapter, 1-inch round, for AccuVac Ampuls	1	each	LZV584
Beaker, 50-mL	1	each	500-41H
Sample Cell, 10-mL, with cap	1	each	21228-00
Stopper for 18 mm Tube	1/test	6/pkg	1731-06

Recommended Standards

Description	Unit	Cat. No.
Sulfate Standard Solution, 1000-mg/L	500 mL	21757-49
Sulfate Standard Solution, 2500-mg/L, 10-mL Ampules	16/pkg	14252-10
Standard, Drinking Water, Mixed Parameter, Inorganic for F^-, NO_3, PO_4, SO_4	500 mL	28330-49

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Cylinder, mixing, 25-mL	each	1896-40
Cylinder, mixing, 50 mL	each	1896-41
Stopper for 18 mm Tube	25/pkg	

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Sulfide

★Method 8131

Methylene Blue Method[1]

(5 to 800 µg/L)

Scope and Application: For testing total sulfides, H_2S, HS^-, and certain metal sulfides in groundwater, wastewater brines, and seawater; USEPA Approved for reporting wastewater analysis[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

[2] Procedure is equivalent to USEPA method 376.2 and Standard Method 4500-S^{2-} D for wastewater.

Test Preparation

Before starting the test:

Analyze samples immediately. Do not preserve for later analysis.
Avoid excessive agitation of samples to minimize sulfide loss.
Some sulfide loss may occur if dilution is necessary.
Sulfide 2 reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration regulated as a hazardous waste by Federal RCRA. Refer to the current MSDS for safe handling and disposal instructions.

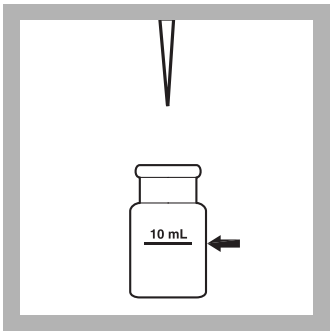
Collect the following items:	**Quantity**
Sulfide 1 Reagent	2 mL
Sulfide 2 Reagent	2 mL
Water, deionized	25 mL
Pipet, serological, 10-mL	1
Pipet Filler, safety bulb	1
Sample Cells, 1-inch square, 10 mL, matched pair	2
Stopper for 18-mm tube	2

Note: *Reorder information for consumables and replacement items is on page 3.*

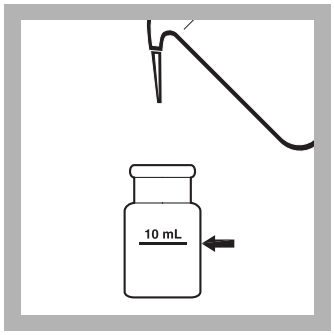
Method 8131

1. Press **STORED PROGRAMS**.

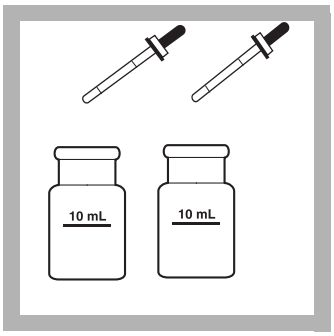
2. Select the test.



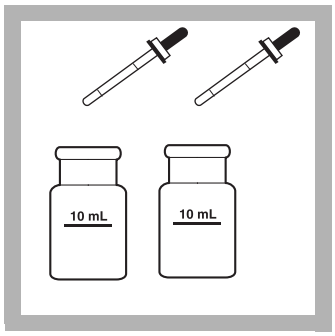
3. Prepared Sample: Avoiding excess agitation of the sample, use a pipet add 10 mL of sample to a square sample cell.



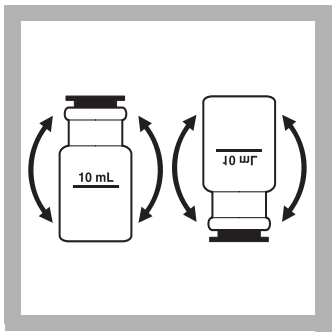
4. Blank Preparation: Measure 10 mL of deionized water into a second square sample cell.



5. Use the calibrated dropper to add 0.5 mL of Sulfide 1 Reagent to each cell. Swirl to mix.



6. Use the calibrated dropper to add 0.5 mL of Sulfide 2 Reagent to each cell.



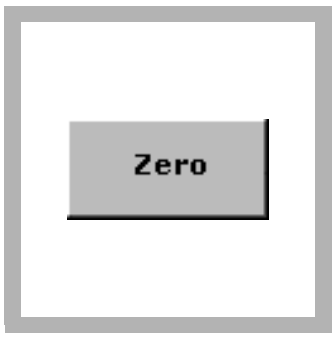
7. Cap the cell and immediately invert to mix.

A pink color will develop, then the solution will turn blue if sulfide is present.

8. Press **TIMER>OK**.

A five-minute reaction period will begin.

9. When the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.



10. Press **ZERO**.

The display will show:

0 µg/L S^{2-}

11. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

12. Press **READ**.

Results are in µg/L S^{2-}.

Determining Soluble Sulfides

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Strong reducing substances (sulfite, thiosulfate and hydrosulfite)	Interferes by reducing the blue color or preventing its development.
Sulfide, high levels	High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Turbidity	For turbid samples, prepare a sulfide-free blank as follows. Use it in place of the deionized water blank in the procedure. **1.** Measure 25 mL of sample into a 50-mL Erlenmeyer flask. **2.** Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears. **3.** Add Phenol Solution dropwise until the yellow color just disappears. Use this solution to replace the deionized water in step 4 of the procedure.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

Summary of Method

Hydrogen sulfide and acid-soluble metal sulfides react with N,N-dimethyl-p-phenylenediamine sulfate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. High sulfide levels in oil field waters may be determined after proper dilution. Test results are measured at 665 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Sulfide Reagent Set (100 tests), includes:	—	—	22445-00
(2) Sulfide 1 Reagent	2 mL	100 mL MDB	1816-32
(2) Sulfide 2 Reagent	2 mL	100 mL MDB	1817-32
Water, deionized	25 mL	4 liters	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Pipet, serological, 10-mL	1	each	532-38
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Stopper, for 18-mm Tube	2	6/pkg	1731-06

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Bromine Water	29 mL	2211-20
Phenol Solution	29 mL	2112-20
Stopper, for 18-mm Tube	25/pkg	1731-25

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Updated November 2005 Edition 1

Sulfite

Colorimetric Method[1]
(0.10 to 5.00 mg/L)

Scope and Application: For boiler water, foodstuffs

[1] Reagent sets for this method are only available in Europe.

Test Preparation

Before starting the test:

Samples must be analyzed immediately.
Sample and Reagent temperature must be between 15–25 °C (59–77 °F).
Adjust the sample pH between 3–10.

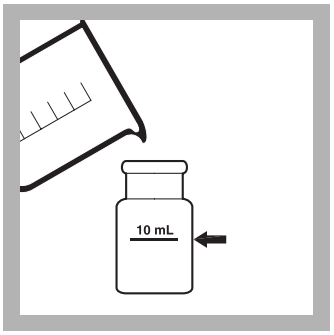
Collect the following items:	**Quantity**
Sulfite Colorimetric Reagent Set:	
Sulfite Reagent A	5 drops
Sulfite Reagent B	2 drops
Water, deionized	varies
Pipet, 10-mL serological	1
Pipet Filler, safety bulb	1
Sample Cells, 1-inch square, 10 mL, matched pair	2

Note: *Reorder information for consumables and replacement items is on page 3.*

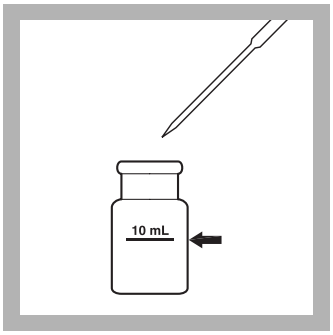
Colorimetric Method

1. Press **STORED PROGRAMS**.

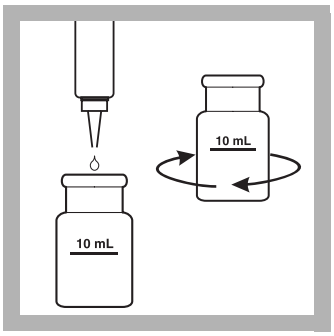
2. Select the test.



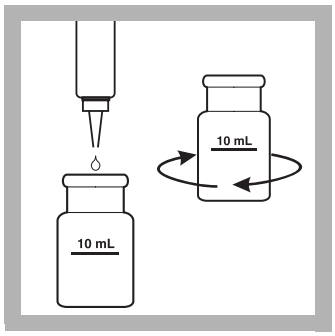
3. Blank Preparation: Fill a clean square sample cell with 10 mL of sample.



4. Prepared Sample: Pipet 10 mL of sample into a second clean square sample cell.



5. Add 5 drops of Sulfite Reagent A (HPT 430 A) to the prepared sample. Swirl to mix.

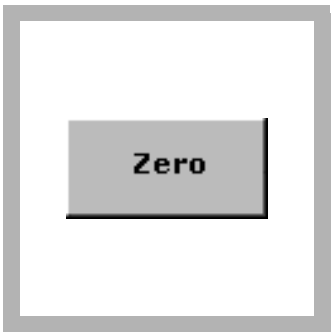


6. Add 2 drops of Sulfite Reagent B (HPT 430 B) to the prepared sample. Swirl to mix.

7. Press **TIMER>OK**.

A 3-minute reaction period will begin. Do not disturb the cell during this time.

8. Wipe the blank and insert the it into the cell holder with the fill line facing right.

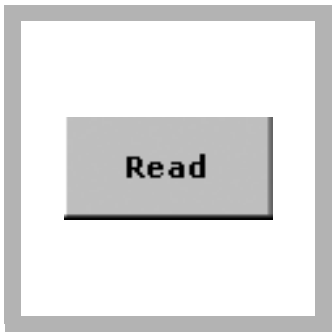


9. Press **ZERO**.

The display will show:

0.00 mg/L SO_3^{2-}

10. When the timer expires, wipe the prepared sample and insert it into the cell holder with the fill line facing right.



11. Press **READ**.

Results are in mg/L SO_3^{2-}.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels
Sulfide	Greater than 5 mg/L

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Samples may be stored up to 7 days by cooling to 4 °C (39 °F) or lower. Warm to 15–25 °C (59–77 °F) before analysis.

Summary of Method

The reagents react with sulfite to form a yellow complex. The samples are measured at 435 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Sulfite Colorimetric Reagent Set, includes:	—	100/pkg	HPT430
Sulfite Reagent A[1]	5 drops	28 mL	—
Sulfite Reagent B[1]	2 drops	8.7 mL	—

[1] Not available separately

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Pipet, 10-mL serological	1	each	532-38
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

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Surfactants, Anionic (Detergents)

Method 8028

Crystal Violet Method[1]

(0.002 to 0.275 mg/L as LAS)

Scope and Application: For water, wastewater, and seawater

[1] *Analytical Chemistry*, 38, 791 (1966).

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Test Preparation

Before starting the test:

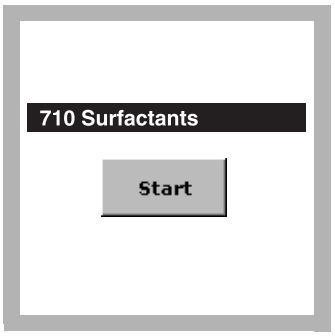
Use benzene only in a well-ventilated area.
Benzene (D018) solutions are regulated as hazardous waste by the Federal RCRA. Do not pour these materials down the drain. Collect water saturated with benzene and benzene solutions for disposal with laboratory solvent wastes. Refer to the current MSDS for safe handling and disposal instructions.
To prevent water droplets from forming in the sample cells, use only dry sample cells and discard the first few mL of benzene. Additionally, it helps to transfer the liquid from the funnel to a sample cell, let it sit for a few seconds, and decant to a second cell for reading.
Excessive agitation may cause an emulsion to form, which in turn makes the phases separate more slowly. Should this occur, remove most of the water layer, then gently agitate the contents of the funnel with a clean Teflon®-coated rod or other such inert tool.
Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.
Acetone may be used to clean benzene from glassware.
For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.
Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.

Collect the following items:	**Quantity**
Benzene, ACS	55 mL
Buffer Solution, sulfate-type	10 mL
Detergent Reagent Powder Pillows	1 pillow
Clippers, for opening powder pillows	1
Cylinder, graduated, 25-mL	1
Cylinder, graduated, 50-mL	1
Cylinder, graduated, 500-mL	1
Funnel, separatory, 500-mL	1
Sample Cells, 10, 25 mL stoppered	2
Support Ring, 4-inch	1
Support, Ring Stand, 5 x 8 inch base	1

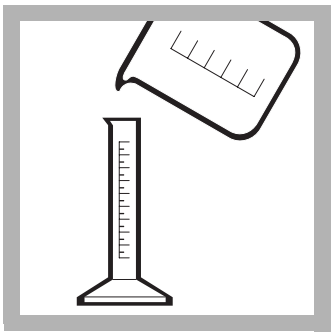
Note: *Reorder information for consumables and replacement items is on page 5.*

Method 8028

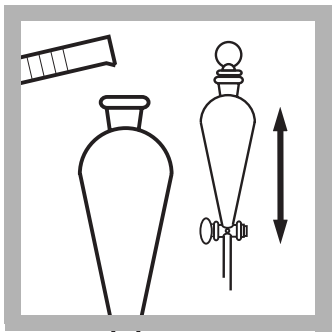
1. Press **STORED PROGRAMS**.



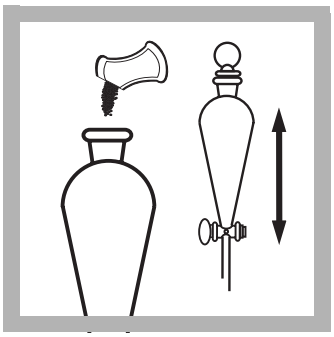
2. Select the test.



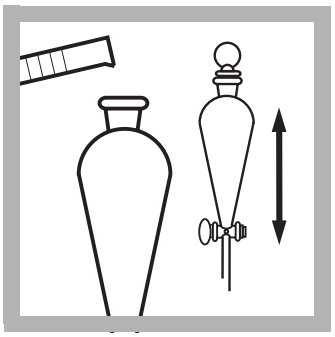
3. Fill a clean 500-mL graduated cylinder to the 300 mL mark with sample. Pour the sample into a clean 500-mL separatory funnel.



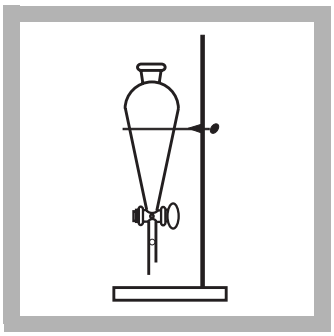
4. Add 10 mL of Sulfate Buffer Solution. Stopper the funnel. Shake the funnel for five seconds.



5. Add the contents of one Detergents Reagent Powder Pillow to the funnel. Stopper the funnel and shake until the powder dissolves completely.



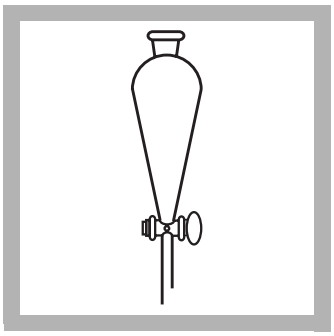
6. Add 30 mL of benzene to the funnel. Stopper the funnel and shake gently for one minute.



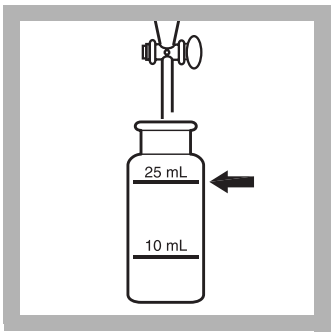
7. Place the separatory funnel in a support stand.

8. Press **TIMER>OK**.

A 30-minute reaction period will begin.

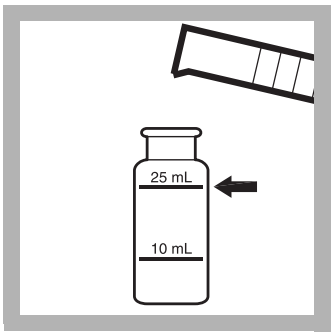


9. After the timer expires, remove the stopper and drain the bottom water layer. Discard this layer.



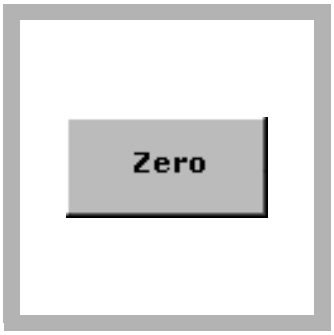
10. Prepared Sample: Drain the top benzene layer into a clean 25-mL sample cell.

Do not filter the benzene layer before color measurement. Filtration removes the blue color.



11. Blank Preparation: Fill another sample cell to the 25-mL mark with pure benzene.

12. Insert the blank into the cell holder with the fill line facing right.



13. Press **ZERO**.

The display will show:

0.000 mg/L LAS

14. Insert the prepared sample into the cell holder with the fill line facing right.

15. Press **READ**.

Results are in mg/L LAS.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chloride	High amounts of chloride, such as those levels found in brines and seawater, will cause low results.
Perchlorate ions	Interferes at all levels.
Periodate ions	Interferes at all levels.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible, but they may be stored at least 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify that the units displayed are in mg/L.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Detergent Voluette® Ampule Standard, 60-mg/L LAS.

5. Prepare three sample spikes. Fill three beakers with 300 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method
Prepare a 0.180-mg/L LAS standard solution as follows:

1. Pipet 3.0 mL of Detergent Standard, 60-mg/L as LAS, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Mix well. Prepare this solution daily. Perform the surfactants procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

Detergents, ABS (alkyl benzene sulfonate), or LAS (linear alkylate sulfonate) are determined by association with crystal violet dye and extraction of the ion-pair complex into benzene. Test results are measured at 605 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Detergents Reagent Set, includes:	—	—	24468-00
(1) Benzene, ACS	55 mL	4 liters	14440-17
(2) Buffer Solution, sulfate-type	10 mL	500 mL	452-49
(3) Detergent Reagent Powder Pillows	1 pillow	25/pkg	1008-68

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25-mL	1	each	508-40
Cylinder, graduated, 50-mL	1	each	508-41
Cylinder, graduated, 500-mL	1	each	508-49
Funnel, separatory, 500-mL	1	each	520-49
Sample Cells, 10, 25 mL stoppered	2	2/pkg	26126-02
Support Ring, 4-inch	1	each	580-01
Support, Ring Stand, 5 x 8 inch base	1	each	563-00

Recommended Standards

Description	Unit	Cat. No.
Detergent Standard Solution, 10-mL Voluette® Ampule, 60-mg/L LAS	16/pkg	14271-10

Optional Reagents and Apparatus

Description	Cat. No.
Acetone	14429-49
Beaker, 600 mL	500-52
Flask, volumetric, 1000 mL	14574-53
Pipet filler	14651-00
Pipet, TenSette®, 0.1 to 1.0 mL	19700-01
Pipet tips for TenSette Pipet 19700-01	21856-96
Pipet, volumetric, 3.00 mL	14515-03

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Updated November 2005 Edition 1

Suspended Solids

Method 8006

Photometric Method[1]

(5 to 750 mg/L)

Scope and Application: For water and wastewater

[1] Adapted from *Sewage and Industrial Wastes*, 31, 1159 (1959).

Test Preparation

Collect the following items:	Quantity
Beaker, 600-mL, polypropylene	1
Blender	1
Cylinder, 500-mL polypropylene, graduated	1
Pipet, serological, 25-mL	1
Pipet Filler, safety bulb	1
Sample Cells, 1-inch square, 10 mL	2

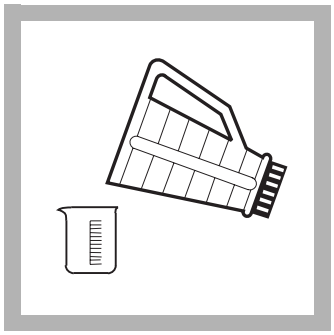
Note: *Reorder information for consumables and replacement items is on page 3.*

Method 8006

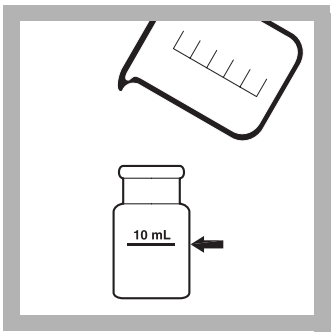
1. Press **STORED PROGRAMS**.

2. Select the test.

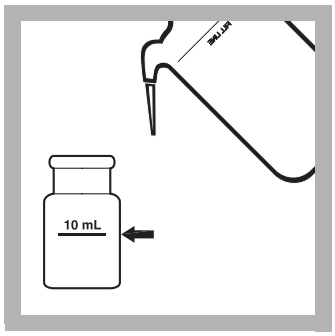
3. Blend 500 mL of sample in a blender at high speed for exactly two minutes.



4. Pour the blended sample into a 600-mL beaker.



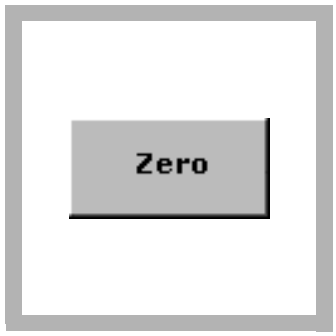
5. Prepared Sample: Stir the sample and immediately pour 10 mL of the blended sample into a sample cell.



6. Blank Preparation: Fill a second sample cell with 10 mL of tap water or deionized water.

Remove gas bubbles in the water by swirling or tapping the bottom of the cell on a table.

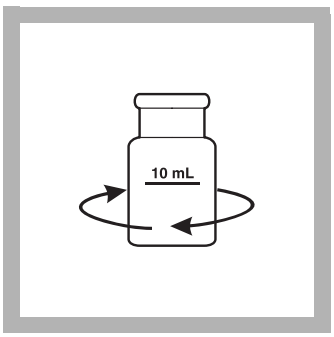
7. Insert the blank into the cell holder with the fill line facing right.



8. Press **ZERO**.

The display will show:

0 mg/L TSS



9. Swirl the prepared sample to remove any gas bubbles and uniformly suspend any residue.

10. Insert the prepared sample into the cell holder with the fill line facing right.

11. Press **READ**.

Results are in mg/L TSS.

Interferences

Samples that absorb strongly at 810 nm, such as blue dyes, may give false, high-bias readings. A user-entered calibration is advised for these samples.

Calibration for this test is based on parallel samples using the gravimetric technique on sewage samples from a municipal sewage plant. For most samples, this calibration will provide satisfactory results. When higher accuracy is required, run parallel spectrophotometric and gravimetric determinations with portions of the same sample. The new calibration should be made on your particular sample using a gravimetric technique as a basis.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. The sample may be stored seven days by cooling to 4 °C (39 °F).

Summary of Method

This method of determining suspended solids is a simple, direct measurement which does not require the filtration or ignition/weighing steps that gravimetric procedures do. The USEPA specifies the gravimetric method for solids determinations, while this method is often used for checking in-plant processes. Test results as mg/L total suspended solids (TSS) are measured at 810 nm.

Consumables and Replacement Items

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Beaker, 600-mL, polypropylene	1	each	1080-52
Blender, 1.2-L, 120 VAC	1	each	26161-00
Blender, 1.2 L, 240 VAC	1	each	26161-02
Cylinder, 500-mL graduated, polypropylene	1	each	1081-49
Pipet, serological, 25-mL	1	each	2066-40
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

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Updated November 2005 Edition 1

Tannin and Lignin

Method 8193

Tyrosine Method[1] (0.1 to 9.0 mg/L)

Scope and Application: For water, wastewater, and boiler water

[1] Adapted from Kloster, M.B., *Journal American Water Works Association*, Vol. 66, No. 1, p. 44 (1974)

Test Preparation

Before starting the test:

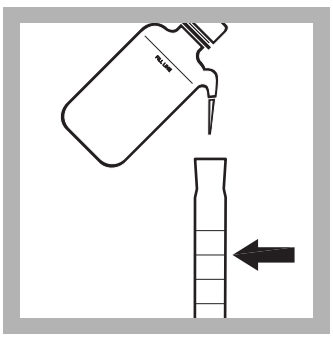
Filter turbid samples and report results as mg/L soluble tannic acid.
Results will be given in mg/L tannins (as tannic acid).
For best accuracy, use a pipet to add the TanniVer® 3 solution.

Collect the following items:	**Quantity**
Tannin and Lignin Reagent Set:	
Sodium Carbonate Solution	10 mL
TanniVer® 3 Tannin-Lignin Reagent	1 mL
Cylinder, graduated mixing, 25-mL	2
Pipet Filler	1
Pipet, volumetric Class A, 5.0-mL	1
Pipet, volumetric Class A, 0.5-mL	1
Sample Cells, 1-inch square glass, 10-mL	2
Water, deionized	25 mL

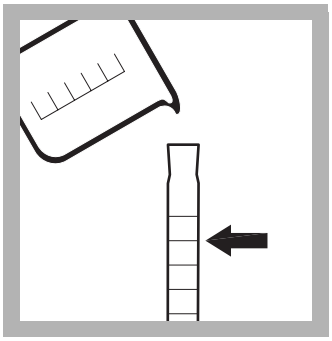
Note: *Reorder information for consumables and replacement items is on page 4.*

Tyrosine Method 8193

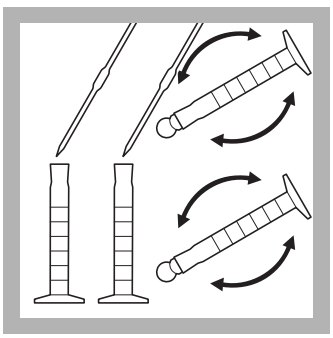
1. Select the test.



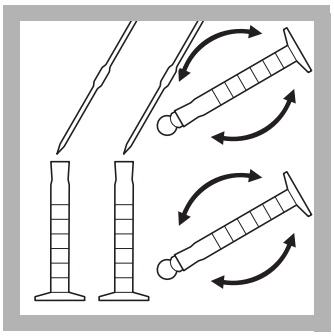
2. Blank Preparation: Fill a 25-mL graduated mixing cylinder to the 25-mL mark with deionized water.



3. Prepared Sample: Fill a second 25-mL graduated mixing cylinder to the 25-mL mark with sample.

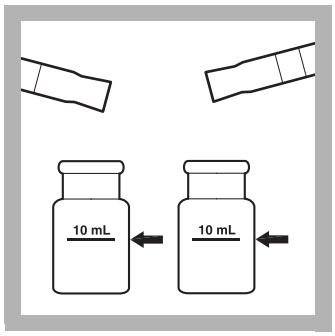


4. Pipet 0.5 mL of TanniVer® 3 Tannin-Lignin Reagent into each cylinder. Insert stopper and invert to mix.



5. Pipet 5.0 mL of Sodium Carbonate Solution into each cylinder. Insert stopper and invert to mix.

A blue color will develop if tannins and/or lignins are present.

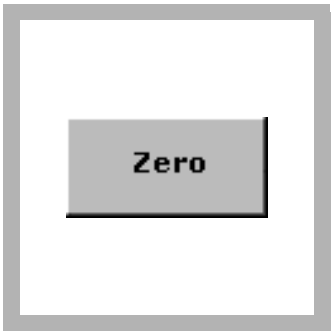


6. Pour 10 mL of each solution into two square sample cells.

7. Press **TIMER>OK**.

A 25-minute reaction period will begin.

8. When the timer expires, insert the blank into the cell holder with the fill line facing right.



9. Press **ZERO**.

The display will show:

0.0 mg/L Tannins
(as Tannic Acid)

10. Insert the prepared sample into the cell holder with the fill line facing right.

11. Press **READ**.

Results are in mg/L Tannins.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Ferrous iron	Causes a positive interference. (2 mg/L of ferrous iron produces a color equivalent to about 1 mg/L of tannic acid.) To eliminate interference of ferrous iron up to 20 mg/L, add one 0.2 g scoop of Sodium Pyrophosphate[1] to the sample before testing.
Sulfite	Interference is eliminated by adding 1 mL of formaldehyde[1] to the sample before testing the sample.

[1] See Optional Reagents and Apparatus on page 4.

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles.

Accuracy Check

Standard Solution Method
Prepare a 200-mg/L tannic acid standard solution as follows:

1. Dissolve 0.200 grams of tannic acid in deionized water and dilute to 1000 mL. Prepare this solution monthly. Further prepare a 6.0-mg/L tannic acid standard by diluting 15.00 mL of the stock solution to 500 mL with deionized water. Prepare this standard daily. Perform the tannin and lignin test on the standard solution as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Summary of Method

This test measures all hydroxylated aromatic compounds, including tannin, lignin, phenol, and cresol. This method produces a blue color proportional to the amount of these compounds present in the sample. The results are reported as total tannin and lignin and expressed as mg/L tannic acid. Test results are measured at 700 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Tannin and Lignin Reagent Set (up to 100 tests), includes:			22446-00
(2) Sodium Carbonate Solution	10 mL	500 mL	675-49
(1) TanniVer® 3 Tannin-Lignin Reagent	1 mL	100 mL	2560-32
Water, deionized	25 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, mixing, with stopper, 25-mL	2	each	20886-40
Pipet filler, safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 5.0-mL	1	each	14515-37
Pipet, volumetric, Class A, 0.5-mL	1	each	14515-34
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Balance, analytical	each	28014-01
Flask, volumetric, 1000 mL	each	14574-53
Flask, volumetric, 500 mL	each	14574-49
Pipet, volumetric, 15.0 mL	each	14515-39
Tannic Acid	113 g	791-14

Optional Reagents and Apparatus

Description	Unit	Cat. No.
Formaldehyde	100 mL	2059-32
Sodium Pyrophosphate	50 g	14295-25

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Updated November 2005 Edition 1

Toxicity

Method 10017

ToxTrak™ Method [1, 2 ,3]
(0 to 100% Inhibition)

Scope and Application: For drinking water, wastewater and natural waters

[1] U.S. Pat. No. 5,413,916
[2] Liu, D., Bull. Environ. Contm. Toxicol. 26, 145-149 (1981)
[3] Environmental Technology Verification ETV Program evaluated, November, 2003

Test Preparation

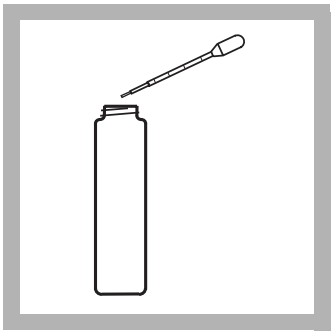
Before starting the test:

Important Note: *Install the light shield in Cell Compartment #2 before performing this test.*
Do not leave the cells in the instrument during incubation. All samples and control cells should be allowed to react under similar conditions of temperature and light.
When testing chlorinated samples, the blank and the samples should be dechlorinated by adding two drops of sodium thiosulfate.
When testing drinking water, the control should be taken from a reservoir of tap water known to be free of toxicants, if possible.

Collect the following items:	**Quantity**
ToxTrak™ Reagent Set:	
Media Set, Bacterial Count Broth Tubes	1 set
Pipet, transfer, sterile	1
Reaction tubes, with cap	1
Sodium Thiosulfate	varies
ToxTrak™ Reagent Powder Pillows	2
ToxTrak™ Accelerator Solution	4 drops
Water, deionized	varies
Clippers	1
Dropper with 0.5 and 1.0 mL marks	1
Forceps	1
Incubator	1
Light Shield	1
Pipet, volumetric, Class A, 5.00 mL and pipet filler	1

Note: *Reorder information for consumables and replacement items is on page 6.*

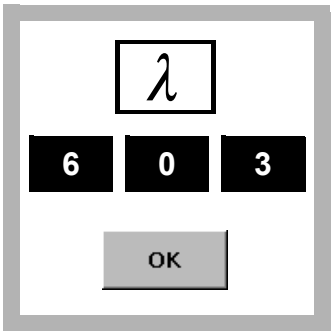
Inoculum Development Using Indigenous Biomass



1. Using one of the dropper pipets provided, add 1.0 mL of source culture to a Total Bacteria Count Broth Tube.

2. Incubate the tube contents at 35 °C (95 °F) until the medium is visibly turbid (approximately 12 hours). Turbidity develops much faster in the incubator than at room temperature.
Note: *Commercial sources of freeze-dried bacteria may also be used.*

Reaction Tube Colorimetric Test

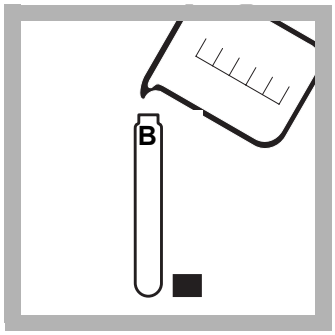


1. Press

SINGLE WAVELENGTH>OPTIONS

then press the λ button. Type in

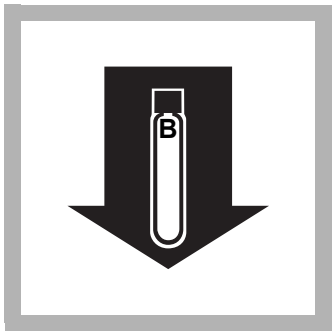
603 nm and press **OK.**



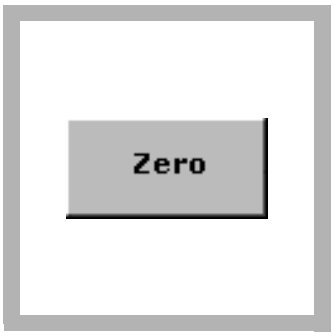
2. Blank Preparation: Fill an empty reaction sample cell with deionized water.

3. Wipe the outside of all the cells with a tissue to remove fingerprints and other marks.

Install the Light Shield in Cell Compartment #2.



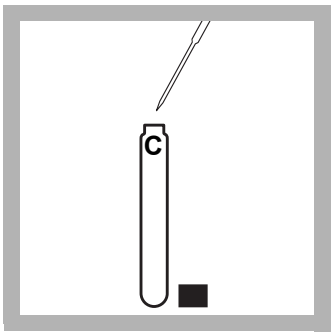
4. Wipe the blank and insert it into the 16-mm round cell holder.



5. Touch **ZERO**.

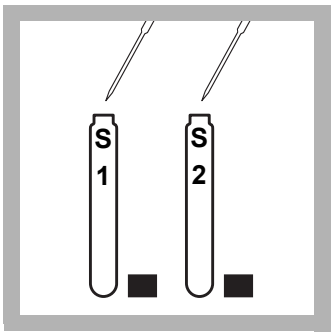
The display will show:

0.000 ABS



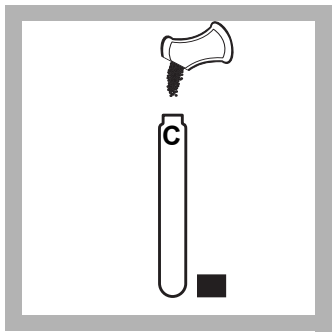
6. Add 5.0 mL of deionized water to the control cell.

Use deionized water that is free of toxicity or another water source that represents baseline toxicity.

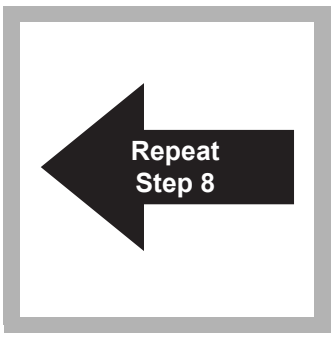


7. Add 5.0 mL of sample (or dilutions) to each sample cell.

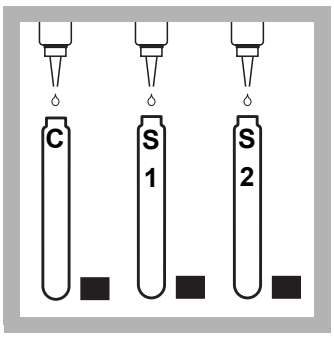
To determine the approximate threshold level of toxicity for a sample, dilute 1 mL of sample to 10 mL with deionized water and run the test. Continue to make serial 1/10 dilutions of the sample until a level is reached that gives 0% Inhibition in the final calculation.



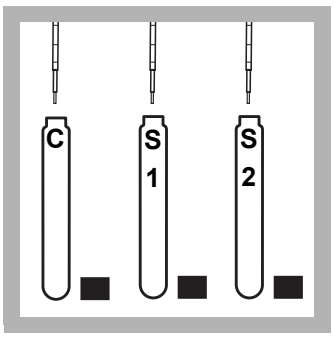
8. Label a cell “control”. Open one ToxTrak Reagent Powder Pillow and add the contents to the empty reaction cell.



9. For each sample or dilution, repeat step 8 and label each cell.



10. Add two drops of Accelerator Solution to each cell.



11. Add 0.5 mL of inoculum (previously prepared) to each cell. Cap and shake to mix.

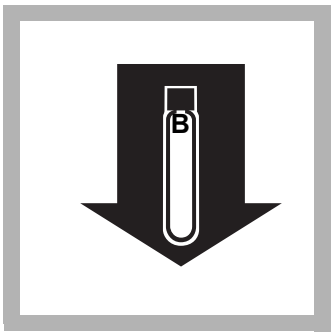
Shaking fully oxygenates the samples and assures that the oxygen concentration is not a factor in determining the respiration rate.

12. Insert the control in the cell holder. Record the absorbance.

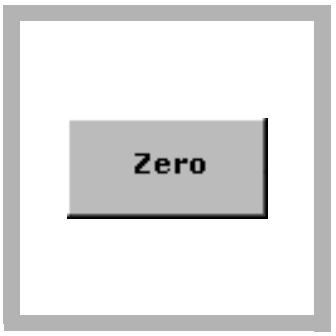
13. Repeat *step 12* for all samples and dilutions. Be sure to record each absorbance.

14. Allow the solutions in the tubes to react until the absorbance of the control decreases 0.60 ±0.10 abs. This takes 45–75 minutes. Invert occasionally.

Reaction time varies according to temperature, age of the culture, bacteria concentrations, etc.



15. After the absorbance of the control has decreased by 0.60 ± 0.10 ABS, insert the blank into the cell holder.



16. Press **ZERO**.

The display will show:

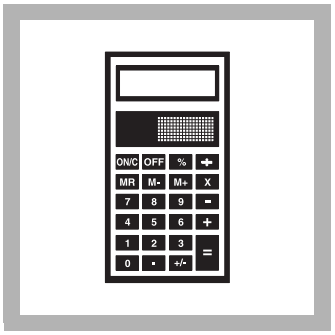
0.000

17. Insert the control into the cell holder.

Press **READ**. The absorbance value of the control will appear.

Record this value.

18. Repeat *step 17* for each sample or dilution. Record each absorbance value.



19. Calculate the % Inhibition:

$$\%I = \left[1 - \left(\frac{\Delta A_{sample}}{\Delta A_{control}}\right)\right] \times 100$$

Where:

ΔA = Initial absorbance value – Final absorbance value

Some toxins increase respiration and will give a negative percent Inhibition on all respiration-based toxicity tests. After repeated testing, samples that give a percent Inhibition that is more negative than –10% should be considered toxic.

Interpreting Results

The percent Inhibition (%I) results obtained are only a relative measurement. They do not represent a true quantitative measurement of toxic concentration. The percent Inhibition does not necessarily increase in direct proportion to the concentration of toxins. To determine the minimum inhibition concentration of a toxin, it is possible to make tenfold dilutions of the sample and determine the percent Inhibition for the dilutions until the sample is diluted sufficiently so that no inhibition is observed. This is the No Observed Effect Concentration (NOEC).

Due to the many variables involved in the test, the limits of detection are on the order of 10% Inhibition. This would correlate to the Lowest Observable Effect Concentration (LOEC). If a sample shows less than 10% Inhibition, repeat the test. After several repetitions, look at the series of data to determine the likelihood of toxicity. Results below 10% are not reliable, but can be used to surmise some presence of toxicity if they are consistent. See examples below:

Data Points: Percent Inhibition	Conclusion
7%, 9%, 5%, 8%, 5%	May be slightly toxic
7%, –4%, –5%, 5%, 1%	Most likely not toxic
–7%, –9%, –5%, –8%, –5%	May be slightly toxic

Some toxins will increase respiration and will give a negative percent Inhibition on this and all other respiration-based toxicity tests. After repeated testing, samples that always give a percent Inhibition that is more negative than -10percent should be considered toxic.

Disposal of Test Cultures

Dispose of active bacterial cultures grown during incubation by using one of these methods:

- Autoclave used test containers at 121 °C (250 °F) for 15 minutes at 15 pounds of pressure. Once the containers are sterile, pour the contents down the drain with running water. The reaction tubes may be washed and re-used.

- Sterilize test containers by using a 1:10 dilution of commercial laundry bleach. Pour the test container contents and test containers into the bleach solution. Allow 10–15 minutes of contact time with the bleach solution. Then pour the liquid down the drain and wash the reaction tubes for reuse.

Summary of Method

This method is based on the reduction of resazurin, a redox-active dye, by bacterial respiration. When it is reduced, resazurin changes color from blue to pink. Toxic substances can inhibit the rate of resazurin reduction. A chemical accelerant has been added to shorten the reaction time. The absorbance of the color change is measured at 603 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
ToxTrak™ Reagent Set, includes:	1	25/set	25972-00
Media Set, Total Bacteria Count Tubes	1	15/pkg	22777-00
Pipet, transfer, sterile	1	15/pkg	22325-12
ToxTrak Reagent Powder Pillows	2	50/pkg	25607-66
ToxTrak Accelerator Solution	4 drops	15 mL SCDB	25608-36
Tubes, 16 x 100 mm	1	30 tubes	22758-06
Tube caps for 22758-00	1	30 caps	22411-06
Water, deionized	varies	500 mL	272-49

Required Apparatus

Description	Unit	Cat. No.
Clippers	each	936-00
Dropper, 0.5 and 1.0 mL marks	20/pkg	21247-20
Forceps, flat square tip	each	14537-00
Light Shield	each	LZV646
Incubator Body, 120 V	each	22816-00
Incubator block, Dri-Bath, 12 well	each	22817-00
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00

Required Apparatus

Description	Cat. No.
Pipet, TenSette®, 0.1 to 1.0 mL	19700-01
Pipet Tips for TenSette Pipet 19700-01	21856-96
Pipet, TenSette, 1.0 to 10.0 mL	19700-10
Pipet Tips for TenSette Pipet 19700-10	21997-96
Rack, test tube	18641-00

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Updated November 2005 Edition 1

TPH (Total Petroleum Hydrocarbons)

Method 10050 **Immunoassay Method[1]**

Scope and Application: For soil and water

[1] This test is semi-quantitative. Results are expressed as greater or less than the threshold value used.

Test Preparation

This TPH test can be used for both soil and water testing. When testing soil, perform the Soil Extraction Procedure on page 2. See Soil Extraction Reagents and Apparatus on page 10. When testing water samples only, proceed directly with Immunoassay Procedure for Soil Extracts and Water Samples.The test requires about 20 to 30 minutes for complete analysis. As many as 10 cuvettes can be run simultaneously.

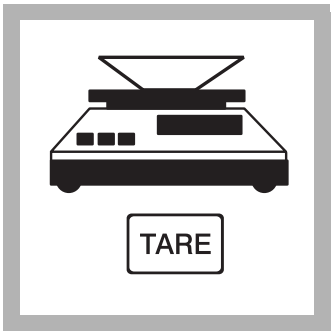
Before starting the test:

Read the entire procedure before starting. Identify and make ready all the necessary reagents, cuvettes, and other apparatus before beginning the analysis.
Timing is critical; follow instructions carefully.
A consistent technique when mixing the cuvettes is critical to this test. The best results come from using the cuvette rack and mixing as described in Using the 1-cm MicroCuvette Rack on page 6. Cuvettes can be mixed individually, but test results may not be as consistent.
Handle the cuvettes carefully. Scratches on the inside or outside may cause erroneous results. Carefully clean the outside of the cuvettes with a clean absorbent cloth or tissue before placing them into the instrument.
Antibody cuvettes and enzyme conjugate are made in matched lots. Do not mix reagent lots.
Twenty Antibody Cuvettes are provided with each reagent set. One Antibody Cuvette will be used for each calibrator or sample. Antibody Cuvettes are not reusable.
To avoid damaging the Color Developing Solution, do not expose it to direct sunlight.
Store the reagents at 4 °C when they are not in use. Allow the reagents to reach room temperature before using them in an analysis. Actual testing may be done at temperatures ranging from 1– 38 °C.
The Soil Extractant contains methyl alcohol which is poisonous and flammable. Before using this and other reagents, read the Material Safety Data Sheet (MSDS) for proper use of protective equipment and other safety information.
Protective nitrile gloves are recommended for this procedure.

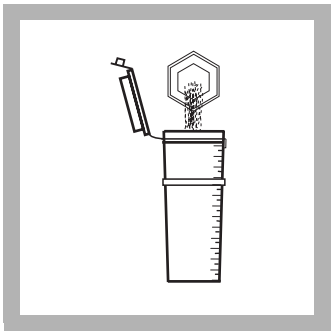
Collect the following items:	**Quantity**
TPH Reagent Set	1
Water, deionized	varies
Caps, flip spout	1
Marker, laboratory	1
Rack, for 1-cm Micro Cuvettes	1
Wipes, disposable	1
Pipet, TenSette®, 0.1–1.0 mL and pipet tips	1
Soil Extraction Kit and Soil Scoop	1

Note: *Reorder information for consumables and replacement items is on page 10.*

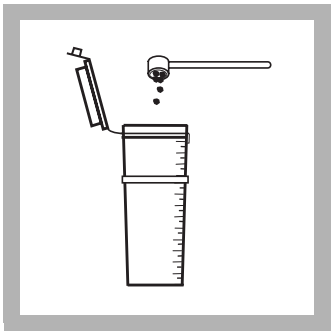
Soil Extraction Procedure



1. Weigh out 5 g of soil in the plastic weighing boat.



2. Carefully pour the soil into an extraction vial.

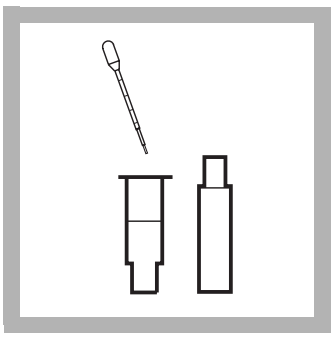


3. Use the 5-gram scoop to add one scoop of sodium sulfate to the extraction vial.

4. Use the graduated cylinder to transfer 10 mL of Soil Extractant into the extraction vial.

5. Cap the extraction vial tightly and shake vigorously for one minute.

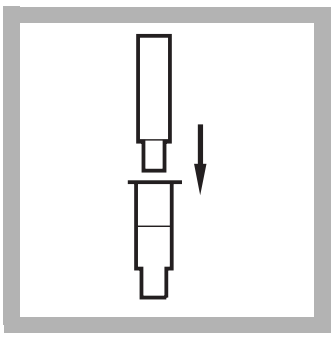
6. Allow to settle for at least one minute. Carefully open the extraction vial.



7. Using the disposable bulb pipet, withdraw 1.0–1.5 mL from the liquid layer at the top of the extraction vial.

Transfer it into the filtration barrel (the bottom part of the filtering assembly into which the plunger inserts).

Do not use more than 1.5 mL. The bulb is marked in 0.25-mL increments.

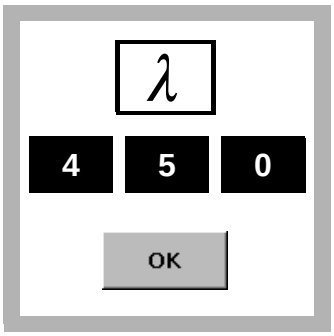


8. Insert the filtration plunger into the filtration barrel. Press firmly on the plunger until the sample extract is forced upward into the center of the plunger.

Use the resultant filtrate for the immunoassay in the Immunoassay for Soil Extracts and Water Samples on page 3.

It may be necessary to place the filtration assembly on a table and press down on the plunger.

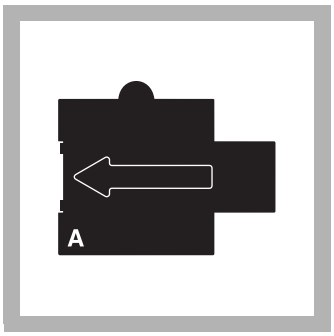
Immunoassay for Soil Extracts and Water Samples



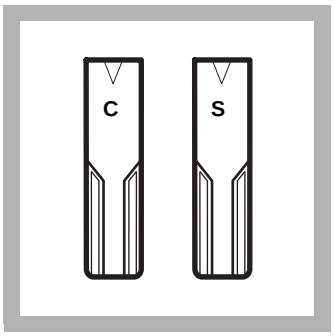
1. Press

SINGLE WAVELENGTH>OPTIONS

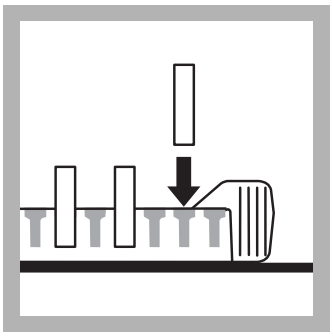
then press the λ button. Type in **450 nm** and press **OK.**



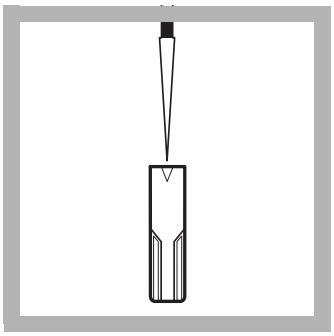
2. Insert Adapter A.



Label an Antibody Cuvette for each calibrator and each sample to be tested. To select the proper calibrators, see Table 2 on page 8 or Table 3 on page 8.

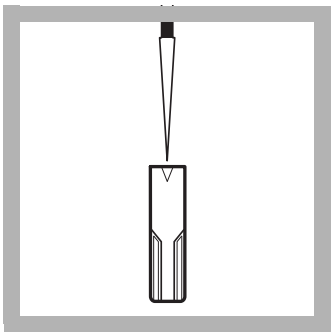


3. Insert the cuvettes into the rack snugly.



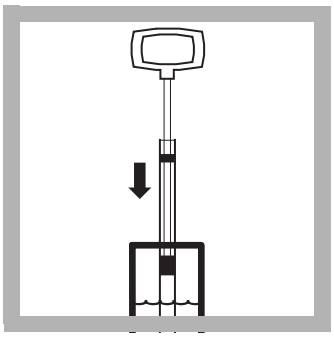
4. Pipet 0.5 mL of Diluent Solution into each Calibrator cuvette.

The same pipette tip can be used repeatedly for this step.



5. If testing soil: Pipet 0.5 mL of *Diluent Solution* into each sample cuvette.

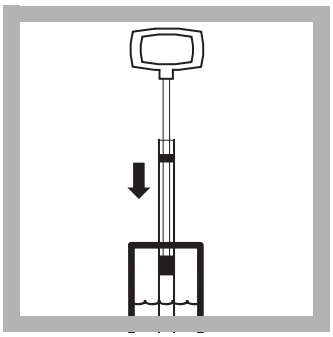
If testing water: Pipet 0.5 mL of each *water sample* into the appropriate cuvette. Use a new pipette tip for each water sample.



6. Have the necessary apparatus at hand for the next four steps as they must be done without delay.

Use a Wiretrol® pipet to transfer 50 µL of each calibrator to be used into the calibrator cuvettes.

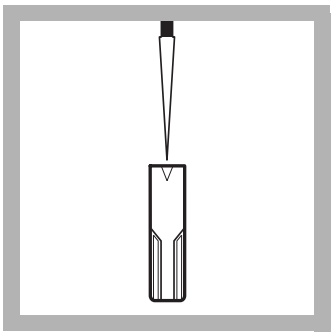
Mix the contents of the cuvettes after each addition. Use a separate capillary tube for each solution.



7. If testing soil: Use a Wiretrol pipet to transfer 50 µL of the filtered extract from step 8 of the Soil Extraction Procedure into the appropriately labeled cuvette. Use a separate capillary tube for each solution.

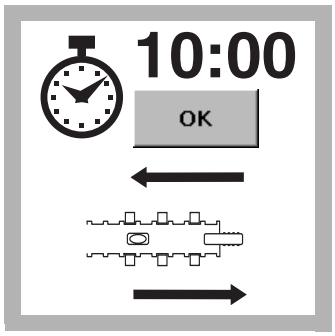
Mix the contents of the cuvettes after the addition of each sample.

If testing water: Use a Wiretrol pipet to transfer 50 µL of methanol into each sample cuvette



8. Immediately pipet 0.5 mL of TPH Enzyme Conjugate into each calibrator and sample cuvette.

The same pipette tip can be used repeatedly for this step.

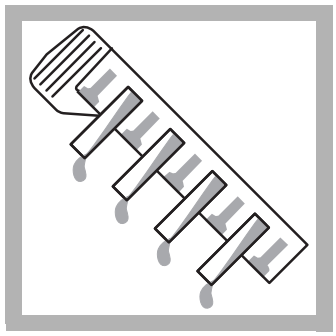


9. Press **TIMER**. Enter **10:00** minutes and press **OK.**

A 10-minute reaction time will begin. Proceed immediately to the next step.

Mix the contents of the cuvettes for 30 seconds using the technique described in Using the 1-cm MicroCuvette Rack on page 6.

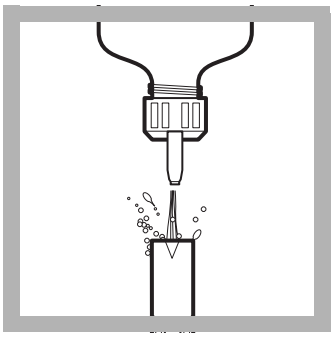
10. After 5 minutes, mix the contents of the rack a second time for a period of 30 seconds using the same technique.



11. At the end of the 10-minute period, discard the contents of all the cuvettes into an appropriate waste container.

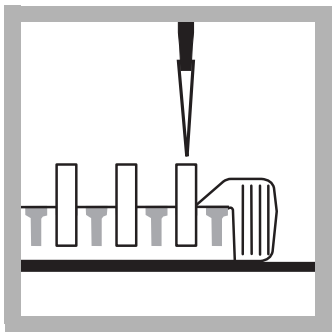
Color Development

Important Note: *Timing is critical. Follow instructions carefully.*



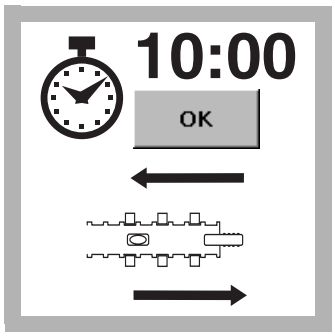
12. Wash each cuvette forcefully and thoroughly four times with deionized water. Empty the rinse water into the waste container.

Ensure most of the water is drained from the cuvettes by turning the cuvettes upside down and tapping them lightly on a paper towel.



13. With the cuvettes still held snugly in the rack, pipet 0.5 mL of Color Developing Solution into each Antibody Cuvette.

Use a new pipette tip for each cuvette.



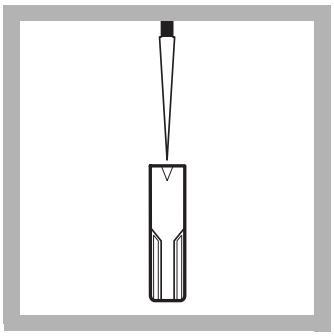
14. Press **OPTIONS>TIMER**. Enter **10:00** minutes and press **OK.**

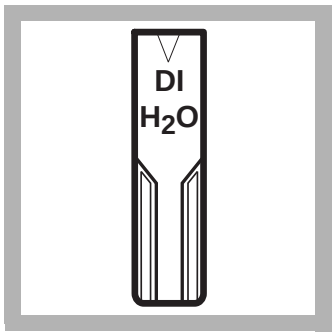
A reaction period will begin. Mix, using the instructions in Using the 1-cm MicroCuvette Rack on page 6.

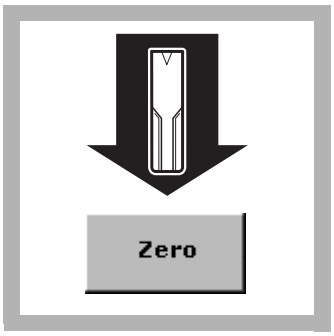
15. After 5 minutes, mix the contents of the rack a second time for a period of 30 seconds using the same technique.

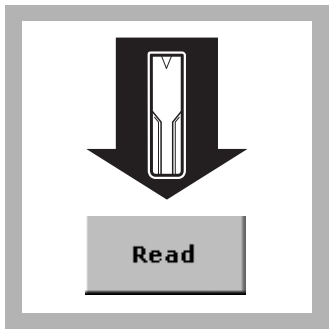
Solutions will turn blue in some or all of the cuvettes.

Measuring the Color









16. At the end of the 10-minute reaction period, pipette 0.5 mL of Stop Solution into each cuvette in the same order as the Color Developing Solution was added in step 12. Use the same pipette tip repeatedly for this step.

Slide the rack for 20 seconds (Using the 1-cm MicroCuvette Rack on page 6.)

Blue solutions will turn yellow with the addition of the Stop Solution.

17. Label and fill a Zeroing Cuvette with deionized water. Wipe the outside of all the cuvettes with a tissue to remove water, smudges, and fingerprints.

18. Insert the filled zeroing cuvette into the cell holder with the arrow to the right.

Orient the arrow in the same direction for all cuvettes.

Press **ZERO**.

The display will show:

0.000 Abs

19. Insert the first calibrator into the cell holder.

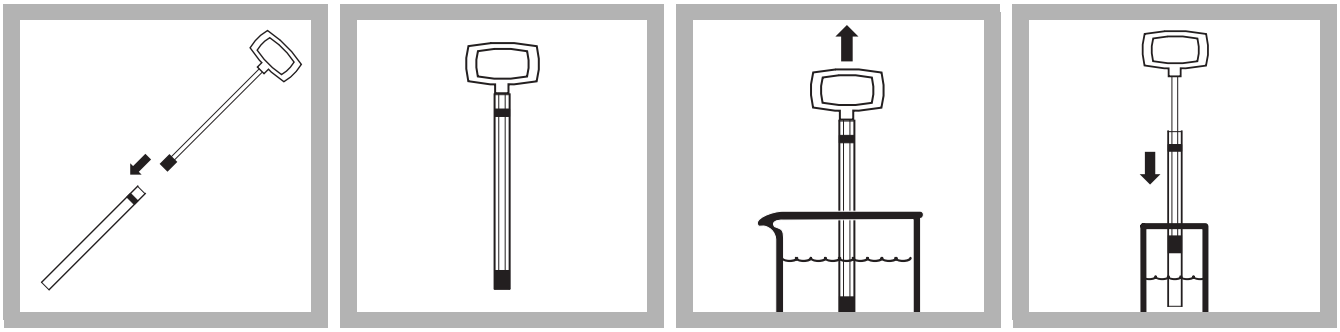
Press **READ**. The display will give an absorbance reading. Record the results for each calibrator and sample.

Repeat this step for all remaining calibrators and samples.

See Interpreting and Reporting Results for help with interpretation of results.

Using the Wiretrol®* Pipet

The Wiretrol Pipet can accurately measure small quantities of liquids. It consists of two parts: a Teflon®-tipped plunger and a calibrated capillary tube. The plunger can be reused; the capillary tubes must be discarded after one use..



1. Wet the orange Teflon® tip of the Wiretrol plunger in the sample and carefully insert it into the end of the capillary tube with the colored band.

2. Push the tip to the other end of the capillary tube until it barely extends beyond the end of the capillary tube.

3. Submerge the capillary tube below the surface of the liquid to be pipetted. Slowly and smoothly draw the Wiretrol plunger up until the bottom of the plunger tips reaches the appropriate volume line.

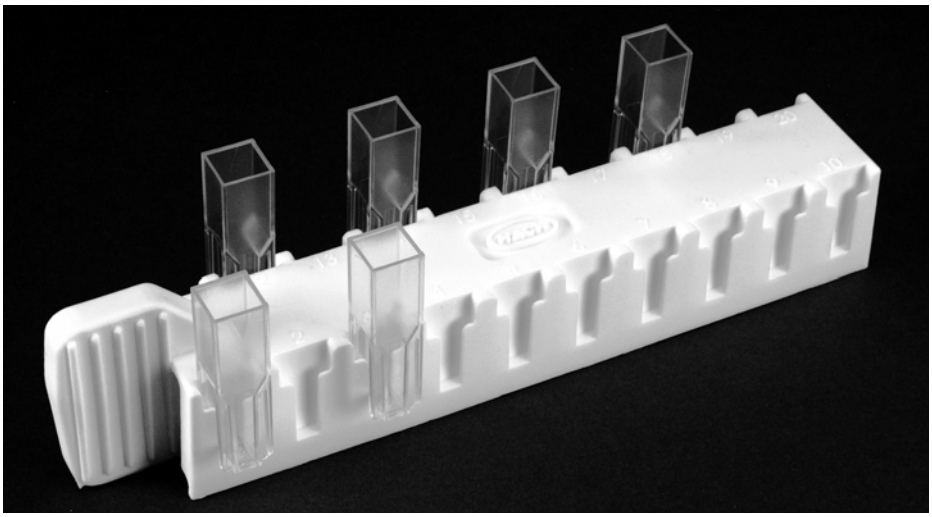
Touch the end of the tube to the side of the vessel to release remaining drops on the capillary tube tip.

4. To discharge the pipet, place the tip of the capillary tube below the surface of the solution and push the Wiretrol plunger down in one smooth motion. Change capillary tubes for each calibrator and sample.

Using the 1-cm MicroCuvette Rack

This rack (Figure 1) has been designed specifically to aid in achieving precise and accurate results when using the immunoassay technique to analyze several samples at the same time.

Figure 1 The 1-cm MicroCuvette Rack



* Wiretrol is a registered trademark of Drummond Scientific.

Loading the Rack—The cuvette rack is designed so that it may be inverted with the cuvettes in place. Identify each cuvette with a sample or calibrator number and insert all the cuvettes in the rack before beginning the procedure. Fit the cuvettes snugly into the rack, but do not force them or they may be difficult to remove and their contents may spill. The cuvettes should remain in place when the rack is inverted and tapped lightly.

Mixing—Set the rack on a hard, flat surface that is at least twice the length of the rack. Hold the rack by one end and vigorously slide it back and forth along its long axis for 30 seconds. The rack should move through a distance equal to its own length in each direction.

Interpreting and Reporting Results

There is an inverse relationship between the concentration of TPH and the reading. In other words, the higher the reading, the lower the concentration of TPH.

Table 1 Relative TPH Concentration

If the sample reading is...	the sample TPH Concentration is...
…less than calibrator reading	…greater than the calibrator concentration
…greater than calibrator reading	…less than the calibrator concentration

Example
Readings:

TPH Calibrator #1: **0.480 Abs**

TPH Calibrator #2: **0.360 Abs**

Sample #1: **0.200 Abs**

Sample #2: **0.400 Abs**

Sample #3: **0.550 Abs**

Interpretation for a Soil Sample

Sample #1—Sample reading is less than the readings for both calibrators. Therefore the sample concentration of TPH is greater than both 20 ppm and 50 ppm diesel fuel.

Sample #2—Sample reading is between the readings for the TPH calibrators. Therefore the sample concentration of TPH is between 20 ppm and 50 ppm diesel fuel.

Sample #3—Sample reading is greater than the readings for both calibrators. Therefore the sample concentration of TPH is less than both 20 ppm and 50 ppm diesel fuel.

Interpretation for a Water Sample

Sample #1—Sample reading is less than the readings for both calibrators. Therefore the sample concentration of TPH is greater than both 2 ppm and 5 ppm diesel fuel.

Sample #2—Sample reading is between the readings for the TPH calibrators. Therefore the sample concentration of TPH is between 2 ppm and 5 ppm diesel fuel.

Sample #3—Sample reading is greater than the readings for both calibrators. Therefore the sample concentration of TPH is less than both 2 ppm and 5 ppm diesel fuel.

Storing and Handling Reagents

- Wear protective gloves and eyewear.
- When storing reagent sets for extended periods of time, keep them out of direct sunlight. Store reagents at a temperature of 4 °C when not in use.

- Keep the foil pouch containing the Antibody Cuvettes sealed when not in use.
- If Stop Solution comes in contact with eyes, wash thoroughly for 15 minutes with cold water and seek immediate medical help.

Sensitivity

The antibodies used in the TPH Test Kit react with a variety of compounds found in petroleum fuels; however, each TPH calibrator has been formulated to represent a specific concentration of diesel fuel. To use the calibrators for other TPH compounds, see *Table 1* or *Table 2* to select the proper TPH calibrator for the compound, sample, and range you want to test.

Example:
To use the TPH calibrators for gasoline, find “Gasoline” in the first column of Table 2 or Table 3. Read across the column to find the ppm represented by each calibrator. For gasoline, calibrator #1 = 15 ppm, calibrator #2 = 35 ppm, and so forth.

Table 2 Various TPHs in Soil

Compound	TPH calibrator #1	TPH calibrator #2	TPH calibrator #3	TPH calibrator #4
	ppm			
Diesel	20	50	100	200
Gasoline	15	35	70	140
Kerosene	35	75	140	250
Benzene	20	45	85	160
Toluene	15	30	50	90
Ethylbenzene	5	15	35	75
m-Xylene	9	20	35	70
o-Xylene	10	20	40	80
p-Xylene	3	5	9	16
BTEX	5	15	25	45

Table 3 Various TPHs in Water

Compound	TPH calibrator #1	TPH calibrator #2	TPH calibrator #3	TPH calibrator #4[1]
	ppm			
Diesel	2	5	10	20
Gasoline	1.5	3.5	7	14
Kerosene	3.5	7.5	14	25
Benzene	2	4.5	8.5	16
Toluene	1.5	3	5	9
Ethylbenzene	0.5	1.5	3.5	7.5
m-Xylene	0.9	2	3.5	7
o-Xylene	1	2	4	8
p-Xylene	0.3	0.5	0.9	16
BTEX	0.5	1.5	2.5	4.5

[1] To test concentrations in water higher than those covered by the calibrators, dilute the original sample as described below.

Diluting Water Samples
Higher concentrations in water can be tested by first diluting the sample with deionized water (see Sensitivity on page 8). Test for other TPH compounds (i.e., gasoline) by using the

conversion factors given in Table 2 and Table 3. Dilute the sample to 50 mL with deionized water in a graduated cylinder.

Choose the mL of sample from Table 4. Use the multiplier value for the chosen quantity to multiply the value from Table 3.

Table 4 Sample Multipliers

mL Sample	Multiplier
0.5	100
1.0	50
2.0	25
5.0	10
10.0	5
25.0	2

For example: If a 0.5 mL water sample is diluted to 50 mL and tested, the calibrator levels for diesel fuel in water would represent 200, 500, 1000, and 2000 ppm respectively.

Interferences

Table 5 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chlorine in water samples	Interferes above 2 ppm. Remove with sodium thiosulfate

Sample Collection and Storage

Analyze the samples as soon as possible after collection. If the samples must be stored, collect them in glass or Teflon® containers that have been washed with soap and water and rinsed with methanol. The container should be capped with a Teflon-lined cap. If a Teflon cap is not available, aluminum foil rinsed in methanol may be used as a substitute cap liner.

When collecting water samples, fill the container completely (no head space) and cover the container with a tightly-sealed lid immediately after collection.

For Soil: Store the samples at 4 °C (40 °F) for no longer than 14 days.

For Water: Chill the sample in an ice bath or refrigerator to limit the loss of volatile compounds. Store samples no longer than 24 hours.

Summary of Method

This method provides semi-quantitative screening based on thresholds for TPH as diesel fuel in the following concentrations:

Soil 20, 50, 100, 200 ppm as diesel fuel

Water 2, 5, 10, 20 ppm as diesel fuel

Immunoassay tests use antigen/antibody reactions to test for specific organic compounds in water and soil. Antibodies specific for TPH are attached to the walls of plastic cuvettes. They selectively bind and remove TPH from complex sample matrices. A prepared sample and a reagent containing enzyme-conjugate molecules (analyte molecules attached to molecules of an enzyme) are added to the Antibody Cuvettes. During incubation, enzyme-conjugate molecules and TPH compete for binding sites on the antibodies. Samples with higher levels of

analyte will have more antibody sites occupied by TPH and fewer antibody sites occupied by the enzyme-conjugate molecules.

After incubation, the sample and unbound enzyme conjugate are washed from the cuvette and a color-development reagent is added. The enzyme in the conjugate catalyzes the development of color. Therefore, there is an inverse relationship between color intensity and the amount of TPH in the sample. The resulting color is then compared with a calibrator to determine whether the TPH concentration in the sample is greater or less than the threshold levels. The TPH concentration is inversely proportional to the color development: the lighter the color, the higher the TPH concentration. Test results are measured at 450 nm.

Consumables and Replacement Items

Required Reagents

Description	Unit	Cat. No.
TPH Reagent Set[1]	20 cuvettes	27743-00
Deionized Water	500 mL	272-48

[1] Immunoassay components are manufactured by Beacon Analytical Systems, Inc.

Required Apparatus

Description	Unit	Cat. No.
Adapter, 1-cm square cell	each	
Caps, flip spout	2/pkg	25818-02
Marker, laboratory	each	20920-00
Rack, for 1-cm Micro Cuvettes	each	48799-00
Wipes, disposable	box	20970-00
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28

Soil Extraction Reagents and Apparatus

Description	Unit	Cat. No.
Balance, AccuLab Pocket Pro 250 B	each	27969-00
Gloves, disposable, Nitrile, medium	each	25505-02[1]
Pipet tips for 19700-01	50/pkg	21856-96
Soil Scoop, 5-g, 4.25-cc	20/pkg	26572-05
Soil Extraction Refill Kit, includes:	each	27752-00
Dropper, LDPE, 0.5 and 1.0-mL	20/pkg	21247-20
Filter and Barrel Assembly	20/pkg	25676-20
Sodium Sulfate, anhydrous	250 g	7099-29
Soil Extractant Solution	200 mL	25677-29
Soil Sample Container	20/pkg	25929-20
Weighing Boat, 8.9-cm square	20/pkg	21790-20
Spatula, disposable	2/pkg	25693-20

[1] Other sizes available.

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Updated November 2005 Edition 1

Trihalomethanes

Method 10132
Water Bath Method

THM Plus™ Method
(10 to 600 ppb as Chloroform)

Scope and Application: For screening THMs in drinking water samples and Formation Potential tests.

Test Preparation

Before starting the test:

If analyzing more than four samples, use 450 mL of water in the water bath.
THM Plus Reagent 2 **must** be at room temperature before use.
A Repipet Jr. may be used in place of the TenSette® Pipet.
Trihalomethane compounds are extremely volatile. Immediately cap sample cells after filling with sample.
Reagent blank is stable for 1–2 hours and need not be prepared for each test.

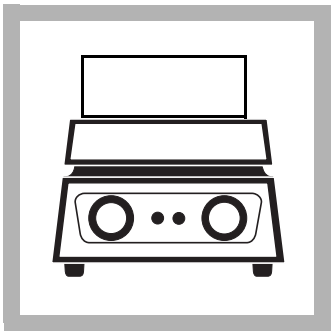
Collect the following items:	**Quantity**
THM Plus Reagent Set	varies
Beaker, 600-mL	1
Cell Holder Assembly, TTHM	1
Evaporating Dish, 125 mm x 65 mm	1
Hot Plate, 7 x 7 inch	1
Pipet, TenSette®, 0.1–1.0 mL and tips	1
Pipet, TenSette®, 1–10 mL and tips	1
Sample Cells, 10 mL, with caps.	2
Sample Cells, 10-mL, 1-inch square, matched pair	2
Wipers, disposable, KimWipes®	varies

Note: *Reorder information for consumables and replacement items is on page 7.*

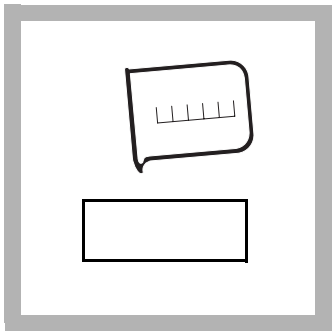
Method 10132

1. Press **STORED PROGRAMS.**

2. Select the test.



3. Prepare a hot water bath by adding 500 mL of water to an evaporating dish. Put the dish on a hot plate and turn the heater on high.

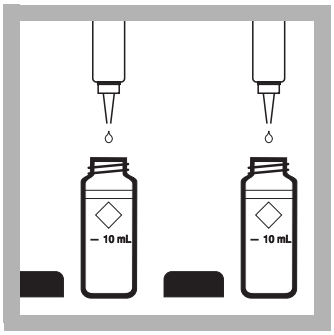


4. Prepare a cooling bath by adding 500 mL of cold (18–25 °C) tap water to a second evaporating dish. Maintain the temperature in this range.

Important Note: *Perform steps 5–10 rapidly to avoid loss of THMs from the sample. When testing more than one sample, complete steps 5–10 for one sample before going on to another. If dispensing sample with a pipette, the pipette must dispense quickly without causing aeration or back pressure.*

5. Prepared Sample: Fill one round sample cell to the 10-mL mark with sample. Cap and label as “sample”.

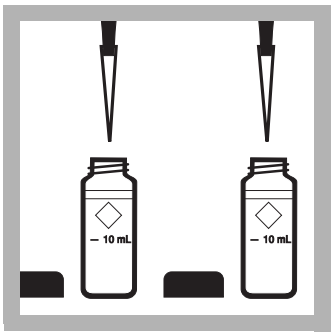
6. Blank Preparation: Fill the second sample cell with deionized water. Cap and label as “blank”.



7. Add three drops of THM Plus Reagent 1 to each cell.

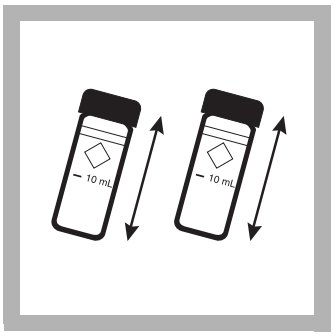
8. Cap tightly and mix gently by swirling each cell three times.

Vigorous shaking can cause loss of THMs into the sample cell headspace.



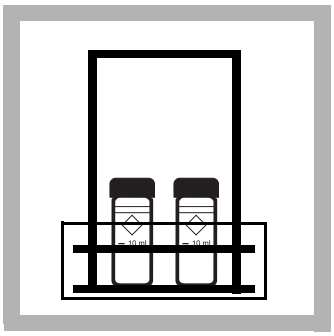
9. Use a TenSette® Pipet to add 3 mL of THM Plus Reagent 2 to each cell. Avoid excess agitation of the sample when dispensing the reagent.

The reagent is viscous and a small amount may remain on the tip after dispensing. This will not affect the results.

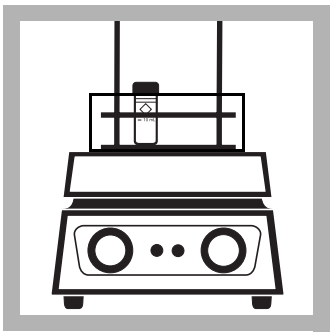


10. Cap tightly and mix by shaking.

Thorough mixing ensures that all of the THM goes into the liquid and does not accumulate in the air above the sample.



11. Place the sample cells in the cell holder assembly.

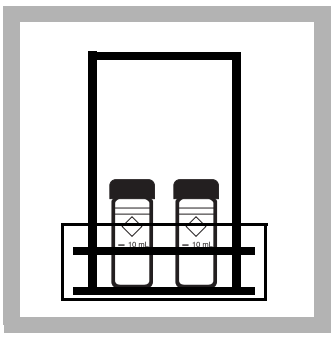


12. Place the assembly in the hot water bath when the water is boiling rapidly.

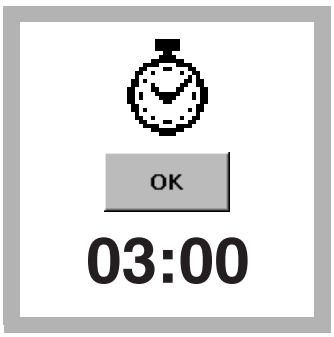
Do not allow the water to rise above the white “diamond” near the top of the sample cells.

13. Press **TIMER>OK**.

A five-minute reaction period will begin.



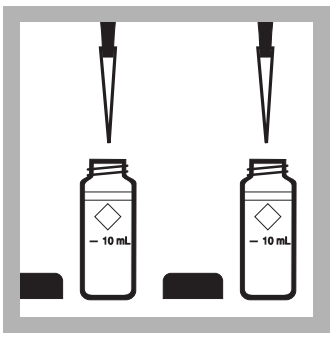
14. When the timer expires, remove the assembly and sample cells from the hot water bath. Place in the cooling bath.



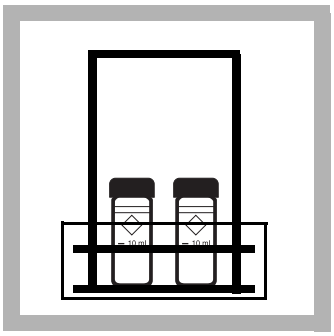
15. Press **TIMER>OK**.

A three-minute cooling period will begin.

When the timer expires, remove the cells from the cooling bath.



16. Use a TenSette Pipet to add 1 mL of THM Plus Reagent 3 to each cell. The sample and blank will become warm.



17. Replace the cooling water with fresh, cold tap water. Place the assembly that contains the sample and blank cells into the cooling bath.

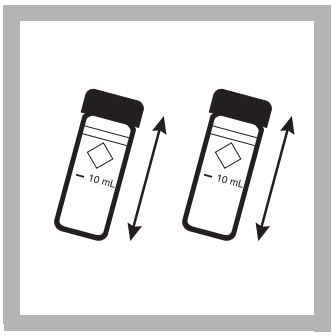
18. Press **TIMER>OK**.

A second three-minute cooling period will begin.

After the timer expires, remove the cells from the cooling bath.

The temperature of the sample should be 15–25 °C.

19. Add the contents of one THM Plus Reagent 4 Powder Pillow to the sample cell and one to the blank.

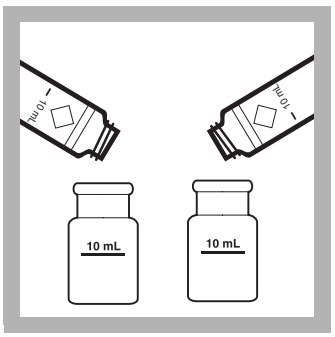


20. Cap each cell tightly and mix by shaking until all the powder dissolves.

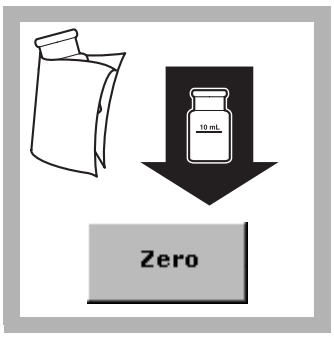
The powder dissolves slowly. Intermittent shaking during the first five minutes of the color development period will help dissolve the reagent powder.

21. Press **TIMER>OK**.

A 15-minute development time will begin.



22. After the timer expires, pour the prepared sample and prepared blank into two square sample cells.

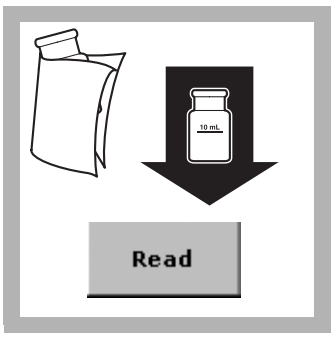


23. Insert the blank into the cell holder with the fill line facing right. Close the cover.

Press **ZERO**.

The display will show:

0 ppb $CHCl_3$



24. Insert the prepared sample into the cell holder with the fill line facing right. Close the cover.

Press **READ**.

Results are in ppb chloroform.

“Underrange” will appear in the display if results are below the Estimated Detection Limit.

Interferences

The substances in Table 1 have been tested and found not to interfere up to the indicated levels (in ppm):

Table 1 Interferences That Have No Effect Up to the Maximum Level Tested

Interfering Substance	Interference Levels and Treatments
Chlorine	<10 ppm
Copper	<1000 ppm
Hardness, Ca	<1000 ppm as $CaCO_3$ May have some turbidity until Reagent 3 is added
Hardness, Mg	<4000 ppm as $CaCO_3$ May have some turbidity until Reagent 3 is added
Iron	<10 ppm
Lead	<2 ppm
Mercury	<10 ppm
Monochloramine	<20 ppm
Nickel	<10ppm
Sodium Bisulfite	<100 ppm
EDTA	Interferes negatively at all levels

Table 2 Additional Disinfection By-Products That React

Compound	Effect
1,1,1-trichloro-2-propanone	Interferes positively
1,1,1-tricholoacetonitrile	Interferes positively
Chloral hydrate	Interferes positively
Dibromochloroacetic acid	Interferes positively
Dichlorobromoacetic acid	Interferes positively
Tribromoacetic acid	Interferes positively
Trichloroacetic acid	Interferes positively

Sampling and Storage

Collect samples in 40-mL glass bottles sealed with Teflon®-lined septa caps. Fill the bottles slowly to overflowing so that no air is included with the sample. Seal the bottles tightly and invert to check that no air has been trapped.

Because trihalomethane compounds (THMs) are extremely volatile, immediate analysis yields the greatest accuracy. If the samples cannot be analyzed immediately, cool samples to 4 °C. This will slow the formation of any additional THM compounds in chlorinated samples. Store the samples at 4 °C in an atmosphere free of organic vapors. Samples should not be held more than 14 days. Allow the samples to equilibrate to 15–25 °C before analyzing.

Accuracy Check

Standard Additions Method (Sample Spike)
Prepare the standard additions sample at the same time as the unspiked water sample.

1. Snap the neck off a THM Standard Ampule, 10 ppm as chloroform.
2. Using a Wiretrol™ Pipet, add 0.050 mL of the standard to 10 mL of water sample. Immerse the tip of the pipet below the surface of the water sample and dispense the aliquot of chloroform standard.
3. Cap the sample cell immediately and swirl three times to mix. Prepare the sample and the spiked sample according to the procedure steps 7–24.
4. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify that the units displayed are in ppb.
5. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
6. Press **OK** to accept the default values for standard concentration, sample volume, and spike volume. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row
7. Insert the prepared spiked sample into the cell holder. Press **READ**.
8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery. The addition should reflect 80–120% recovery.

CAUTION
Chloroform is extremely volatile! Do not shake it when mixing.

Standard Solutions Method
Prepare a 99 ppb chloroform standard by pipetting 10.0 mL of organic-free water into a sample cell. Snap the neck off a THM Standard Ampule, 10 ppm as chloroform. Using a Wiretrol Pipette, transfer 0.100 mL of the chloroform standard into the organic-free water. Immerse the end of the pipet tip under the water to dispense the chloroform. Cap the sample cell immediately and swirl three times to mix. Immediately perform steps 7–24 of the procedure. Do not make up the standard in advance. Use the standard immediately upon preparation.

Summary of Method

The THM Plus method reacts with the trihalogenated disinfection by-products formed as the result of the disinfection of drinking water with chlorine in the presence of naturally occurring organic materials. These disinfection by-products (DBPs) may be produced in the treatment plant or the distribution system as long as the water is in contact with free chlorine residual. The formation of the DBPs is influenced by chlorine contact time, chlorine dose and residual, temperature, pH, precursor concentration, and bromide concentration.

The predominant DBPs formed by the chlorination of drinking water are the trihalomethanes or THMs. The four trihalogenated compounds that form are chloroform, bromoform, dichlorobromomethane, and dibromochloromethane. These four compounds comprise the Total Trihalomethanes (TTHMs) group which is regulated under the Safe Drinking Water Act. The combined concentration of the TTHMs, is regulated to be 80 ppb or less in drinking water samples. Other DBPs that may be present and react under the conditions of the THM Plus method are listed in Interferences.

In the THM Plus method, THM compounds present in the sample react with N, N,-diethylnicotinamide under heated alkaline conditions to form a dialdehyde intermediate.

The sample is then cooled and acidified to pH 2.5. The dialdehyde intermediate formed is then reacted with 7-amino-1,3 napthalene disulfonic acid to form a colored Schiff base. The color formed is directly proportional to the total amount of THM compounds present in the sample. Test results are measured at 515 nm and reported as ppb chloroform.

Consumables and Replacement Items

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Reagent Set (50 tests[1]), includes:			27908-00
THM Plus™ Reagent 1	6 drops	15 mL	27539-29
THM Plus™ Reagent 2	6 mL	330 mL	27540-48
THM Plus™ Reagent 3	2 mL	110 mL	27541-42
THM Plus™ Reagent 4	2 pillows	100 pillows	27566-99

[1] Fifty tests equals 25 samples and 25 individual blanks. Additional tests can be obtained when multiple samples are run using a single blank.

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Beaker, 600-mL	1	each	500-52
Cell Holder Assembly, TTHM	1	each	47880-00
Evaporating Dish, 125 mm x 65 mm	1	each	27647-00
Hot Plate, 7 x 7 in., 120 VAC, digital	1	each	28816-00
Hot Plate, 7 x 7 in., 240 VAC, digital	1	each	28816-02
Pipet, TenSette®, 0.1–1.0 mL	1	each	19700-01
Pipet Tips for TenSette Pipet 19700-01	varies	50/pkg	21856-96
Pipet, TenSette®, 1–10 mL	1	each	19700-10
Pipet Tips, for TenSette Pipet 19700-10	varies	50/pkg	25589-96
Sample Cells, 10 mL, with caps.	2	6/box	24276-06
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Wipers, disposable, KimWipes®	varies	280/pkg	20970-00

Recommended Standards

Description	Unit	Cat. No.
Chloroform, 10-ppm ampule	each	27567-07
Water, Reagent, Organic-free	500 mL	26415-49

Recommended Apparatus

Description	Unit	Cat. No.
Flask, volumetric, 100 mL, class A	.each	14574-42
Pipet, filler, safety bulb	each	14651-00
Pipet, volumetric, class A, 10 mL	each	14515-38
Pipettes, Wiretrol™, 50–100 µL	250/pkg	25689-05
Repipet Jr., 1-mL	each	21113-02
Vials, glass, 40-mL, with Septa cap	5/pkg	27940-05

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Updated November 2005 Edition 1

Volatile Acids

Method 8196

Esterification Method[1]

Powder Pillows

(27 to 2800 mg/L)

Scope and Application: For digestor sludges

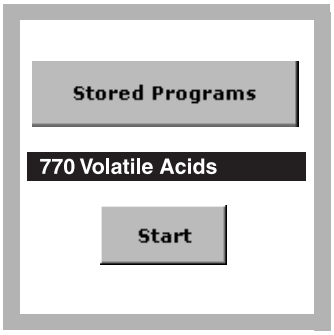
[1] Adapted from *The Analyst*, 87, 949 (1962)

Test Preparation

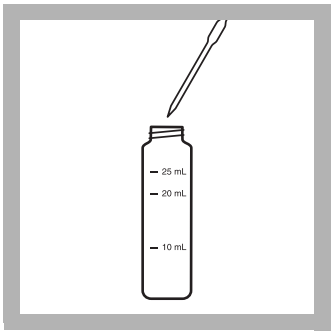
Collect the following items:	Quantity
Centrifuge	1
Centrifuge Tubes and Caps	2
Cylinder, 10-mL graduated	1
Ethylene Glycol	3 mL
Ferric Chloride-Sulfuric Acid Solution	20 mL
Funnel and Filter Paper	—
Hot Plate	1
Hydroxylamine Hydrochloride Solution, 100-g/L	1 mL
Pipet Filler	1
Pipet, 2 mL	1
Pipet, volumetric, Class A, 0.50-mL	1
Pipet, volumetric, Class A,10-mL	1
Sample Cells, 10-20-25 mL	2
Sample Cells, 1-inch square glass	2
Sodium Hydroxide Standard Solution, 4.5 N	4 mL
Sulfuric Acid Standard Solution, 19.2 N	0.4 mL
Water Bath and Rack	1
Water, deionized	20.5 mL

Note: *Reorder information for consumables and replacement items is on page 5.*

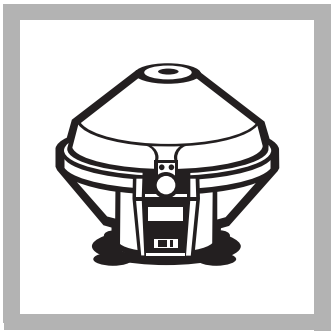
Esterification Method 8196



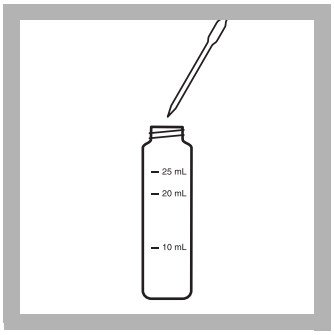
1. Select the test.



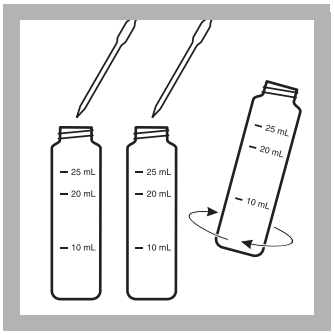
2. Blank Preparation: Pipet 0.5 mL of deionized water into a dry 25-mL sample cell.



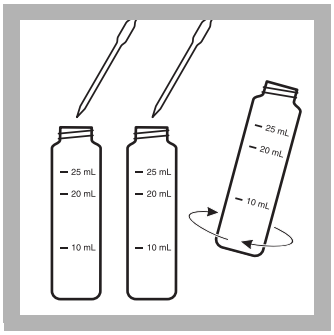
3. Filter or centrifuge 25 mL of sample. Centrifuging is faster than filtration.



4. Prepared Sample: Pipet 0.5 mL of the filtrate or supernatant into a second dry 25-mL sample cell.



5. Pipet 1.5 mL of ethylene glycol into each sample cell. Swirl to mix.



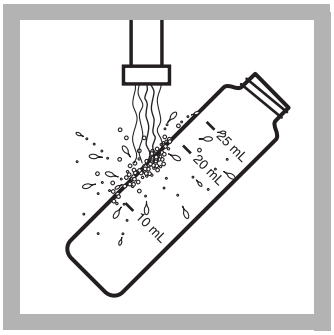
6. Pipet 0.2 mL of 19.2 N Sulfuric Acid Standard Solution into each cell. Swirl to mix.

7. Insert both cells into a boiling water bath.

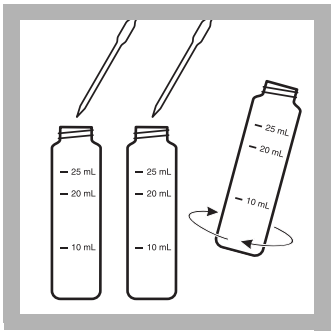
Alternatively, the cells may be boiled in a 500-mL beaker.

8. Press **TIMER>OK**.

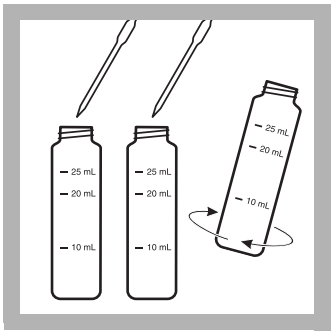
A three-minute reaction period will begin.



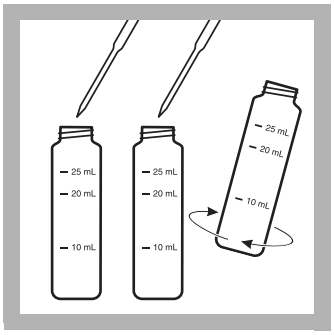
9. When the timer expires, cool the solutions to 25 °C (until the cell feels cold) with running tap water.



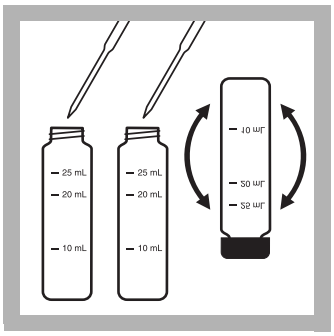
10. Using a pipet filler, pipet 0.5 mL of Hydroxylamine Hydrochloride Solution into each cell. Swirl to mix.



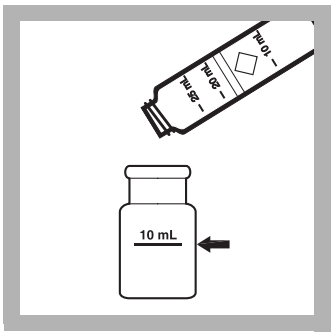
11. Using a pipet filler, pipet 2.0 mL of 4.5 N Sodium Hydroxide Standard Solution into each cell. Swirl to mix.



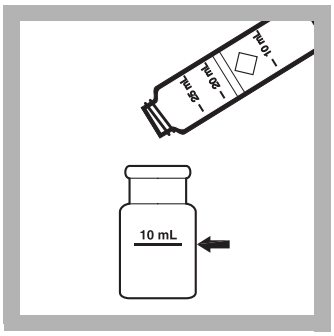
12. Add 10 mL of Ferric Chloride Sulfuric Acid Solution to each cell. Swirl to mix.



13. Add 10 mL of deionized water to each cell. Cap and invert to mix.



14. Transfer 10 mL of the blank solution from the round 25-mL cell to a clean dry square sample cell.

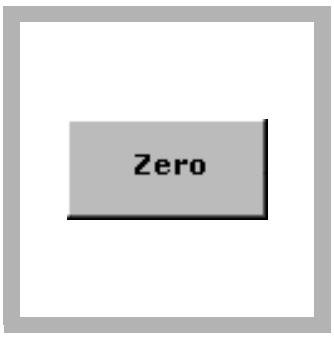


15. Transfer 10-mL of the sample solution from the round 25-mL cell to a clean dry square sample cell.

16. Immediately press **TIMER>OK**.

Another three-minute reaction period will begin. During this time, complete steps 17 and 18.

17. Blot each sample cell dry. Immediately insert the blank into the cell holder with the fill line facing right.

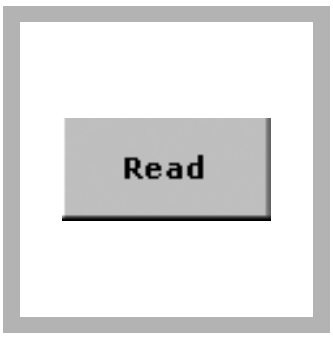


18. Press **ZERO**.

The display will show:

0 mg/L HOAC

19. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.



20. Press **READ**.

Results are in mg/L HOAC.

Sample Collection, Preservation, and Storage

Collect samples in clean plastic or glass bottles. Analyze as soon as possible after collection. Samples can be stored for up to 24 hours by cooling to 4 °C (40 °F) or below. Warm samples to room temperature before analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Volatile Acid Voluette® Ampule Standard, 62,500-mg/L as acetic acid.

5. Prepare three sample spikes. Fill three Mixing Cylinders* with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method
Prepare a 500 mg/L volatile acid standard solution as follows:

1. Pipet 4.00 mL of a 62,500-mg/L Volatile Acid Standard Solution into a 500-mL Class A volumetric flask. Dilute to volume with deionized water. Prepare this solution daily. Perform the esterification procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **OK** to accept the displayed concentration. If an alternate concentration is used, press the number in the **ADJUST TO** field and enter the actual concentration. Press **OK**. Press **ADJUST**.

Summary of Method

The volatile acid test is designed specifically for determining volatile acids in digestor sludges. The method is based on esterification of the carboxylic acids present in the sample and subsequent determination of the esters by the ferric hydroxamate reaction. All volatile acids present are reported as their equivalent mg/L as acetic acid. Test results are measured at 495 nm.

* See Optional Reagents and Apparatus on page 5.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Volatile Acid Reagent Set (90 tests), includes:			22447-00
(1) Ethylene Glycol	3 mL	1000 mL	2039-53
(2) Ferric Chloride-Sulfuric Acid Solution	20 mL	1000 mL	2042-53
(1) Hydroxylamine Hydrochloride Solution, 100-g/L	1 mL	100 mL	818-42
(1) Sodium Hydroxide Standard Solution, 4.5 N	4 mL	1000 mL	2040-53
(1) Sulfuric Acid Standard Solution, 19.2 N	0.4 mL	100 mL MDB	2038-32
Water, deionized	20.5 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Centrifuge, 115 VAC, 60 Hz.	1	each	26765-00
Centrifuge Tubes, 15-mL	2	10/pkg	22787-39
Centrifuge Tube Caps	2	20/pkg	25852-20
Cylinder, graduated, 10-mL	1	each	508-38
Filter Paper, folded, 12.5-cm	1	100/pkg	1894-57
Funnel, poly, 65-mm	1	each	1083-67
Hot Plate, 4-inch micro, 120 VAC	1	each	12067-01
Hot Plate, 4-inch micro, 240 VAC	1	each	12067-02
Pipet Filler, safety bulb	1	each	14651-00
Pipet, serological, 2-mL	1	each	532-36
Pipet, volumetric, Class A, 0.50-mL	1	each	14515-34
Pipet, volumetric, Class A, 10.00-mL	1	each	14515-38
Sample Cells, 10-20-25 mL, with cap	2	6/pkg	24019-06
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02
Water Bath and Rack	1	each	1955-55

Recommended Standards

Description	Unit	Cat. No.
Volatile Acids Standard Solution, 10-mL Voluette® Ampule, 62,500-mg/L as HOAC	16/pkg	14270-10

Optional Reagents and Apparatus

Description	Cat. No.
Cylinder, mixing	1896-40

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Updated November 2005 Edition 1

Zinc

Method 8009
Powder Pillows

Zincon Method[1]
(0.01 to 3.00 mg/L)

Scope and Application: For water and wastewater; digestion is required for determining total zinc (see Digestion on page 4); USEPA Approved for wastewater analyses[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.

[2] Federal Register, 45(105) 36166 (May 29, 1980).

Test Preparation

Before starting the test:

Use only glass-stoppered cylinders in this procedure.
Wash glassware with 1:1 HCl[1] and rinse with deionized water before use.
Use plastic droppers in this procedure. Droppers with rubber bulbs may contaminate the reagent.
ZincoVer® 5 reagent contains potassium cyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Cyanide should be collected for disposal as a reactive (D003) waste. Be sure that cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. Refer to the current MSDS for handling and disposal information.

[1] See Optional Reagents and Apparatus on page 5.

Collect the following items:	**Quantity**
Zinc Reagent Set:	
Cyclohexanone	0.5 mL
ZincoVer 5 Reagent Powder Pillow	1
Cylinder, graduated mixing, 25-mL	1
Sample Cells, 1-inch square glass, 10 mL	2

Note: *Reorder information for consumables and replacement items is on page 5.*

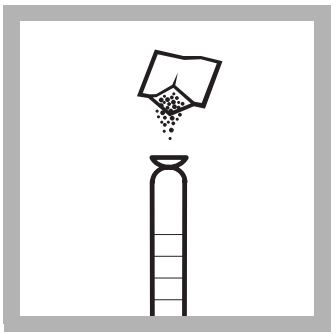
Powder Pillows — Method 8009

CAUTION
ZincoVer 5 Reagent contains cyanide and is very poisonous if taken internally or if fumes are inhaled. Do not add to an acidic sample (pH < 4).

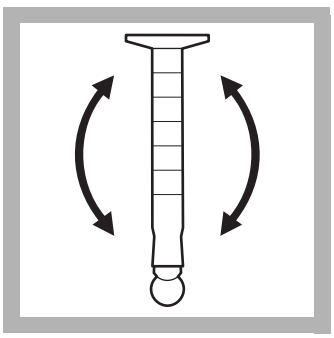
1. Press **STORED PROGRAMS**.

2. Select the test.

3. Fill a 25-mL graduated mixing cylinder with 20 mL of sample.

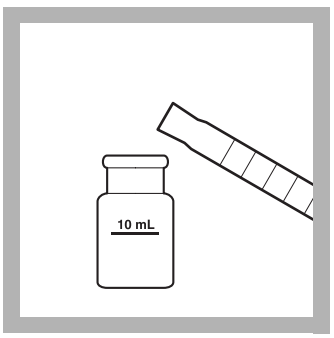


4. Add the contents of one ZincoVer 5 Reagent Powder Pillow to the cylinder. Stopper.

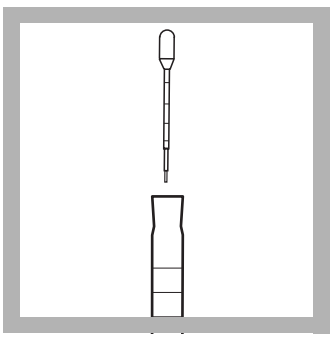


5. Invert several times to dissolve the powder completely. Inconsistent readings may result for low zinc concentrations if all the particles are not dissolved.

The sample should be orange. If the sample is brown or blue, either the zinc concentration is too high, or an interfering metal is present. Dilute the sample and repeat the test.



6. Blank Preparation: Pour 10 mL of the solution into a square sample cell.



7. Prepared Sample: Use a plastic dropper to add 0.5 mL of cyclohexanone to the remaining solution in the graduated cylinder.

8. Press **TIMER>OK**.

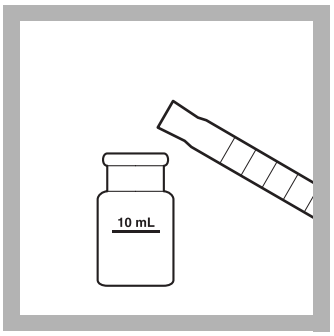
A 30-second reaction period will begin.

During the reaction period, stopper the cylinder and vigorously shake the prepared sample.

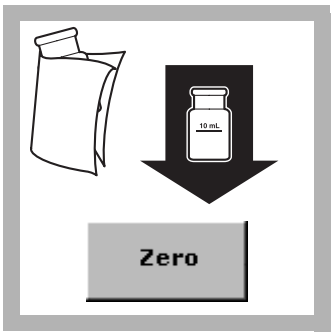
The sample will be reddish-orange, brown, or blue, depending on the zinc concentration.

9. Press **TIMER>OK**.

A three-minute reaction period will begin. During this reaction period, complete step 10.



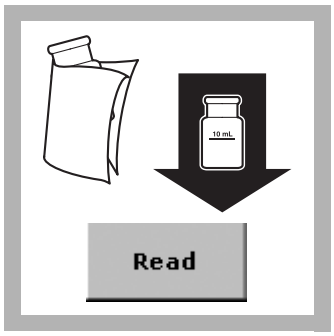
10. Pour the prepared sample solution from the cylinder into a second square sample cell.



11. When the timer expires, wipe the blank and insert it into the cell holder with the fill line facing right.

Press **ZERO**. The display will show:

0.00 mg/L Zn



12. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

Press **READ**. Results are in mg/L Zn.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 6 mg/L
Cadmium	Greater than 0.5 mg/L
Copper	Greater than 5 mg/L
Iron (ferric)	Greater than 7 mg/L
Manganese	Greater than 5 mg/L
Nickel	Greater than 5 mg/L
Organic Material	Large amounts may interfere. Pretreat the sample with a mild digestion.
Highly buffered or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment. Adjust pH to 4–5.

Samples containing amino-tri(methylene phosphonic acid) (AMP) will exhibit a negative interference. Perform a total phosphorus digestion (Method 8190) to eliminate this interference.

Important Note: *Adjust the pH of the sample after the total phosphorus digestion to 4–5 with Sodium Hydroxide before analysis with the zinc test.*

Sample Collection, Storage, and Preservation

Collect samples in acid-cleaned plastic or glass bottles. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature.

Before analysis, adjust the pH to 4–5 with 5.0 N Sodium Hydroxide. Do not exceed pH 5 as zinc may precipitate. Correct the test result for volume additions.

Accuracy Check

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Zinc Voluette® Ampule Standard, 25-mg/L Zn.

5. Prepare three sample spikes. Fill three mixing cylinders* with 20 mL of sample and use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

Standard Solution Method
Prepare a 1.00-mg/L zinc standard solution as follows:

1. Using Class A glassware, pipet 10.00 mL of Zinc Standard Solution, 100-mg/L, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the Zinc procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Digestion

Digestion is required if total zinc is being determined. The following is not the USEPA digestion.

1. If nitric acid has not been added to the sample previously, add 5 mL of Concentrated Nitric Acid* to one liter of sample (use a glass serological pipet and pipet filler). If the sample was acidified at collection, add 3 mL of nitric acid to one liter of sample.

2. Transfer 100 mL of acidified sample to a 250-mL Erlenmeyer flask.

3. Add 5 mL of 1:1 Hydrochloric Acid*.

4. Heat sample on a Hot Plate* for 15 minutes at 95 °C (203 °F). Make sure the sample does not boil.

* See Optional Reagents and Apparatus on page 5.

5. Filter cooled sample through a membrane filter and adjust the volume to 100 mL with Deionized Water.

6. Adjust the pH to 4–5 with 5.0 N Sodium Hydroxide* before analysis. See Sample Collection, Storage, and Preservation on page 3 for instructions.

Summary of Method

Zinc and other metals in the sample are complexed with cyanide. Adding cyclohexanone causes a selective release of zinc. The zinc reacts with 2-carboxy-2'-hydroxy-5'-sulfoformazyl benzene (zincon) indicator to form a blue-colored species. The blue color is masked by the brown color from the excess indicator. The intensity of the blue color is proportional to the amount of zinc present. Test results are measured at 620 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Zinc Reagent Set, 20-mL sample size (100 tests = 100 samples + 100 blanks), includes:	—	—	24293-00
Cyclohexanone	—	100 mL MDB	14033-32
ZincoVer® 5 Reagent Powder Pillows	1	100/pkg	21066-69

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, mixing, 25-mL	1	each	20886-40
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Recommended Standards

Description	Unit	Cat. No.
Water, deionized	4 L	272-56
Zinc Standard Solution, 100-mg/L	100 mL	2378-42
Zinc Standard Solution, 2-mg/L PourRite® Ampule, 25-mg/L as Zn	20/pkg	14246-20
Zinc Standard Solution, 10-mg/L Voluette® Ampule, 25-mg/L as Zn	16/pkg	14246-10

Optional Reagents and Apparatus

Description	Cat. No.
Flask, Erlenmeyer, 250 mL	505-46
Hot Plate, 115 V	12067-01
Hot Plate, 220 V	12067-02
Hydrochloric Acid 6.0 N, 1:1	884-49
Nitric Acid, concentrated	152-49
Sodium Hydroxide 5.0 N	2450-26

* See Optional Reagents and Apparatus on page 5.

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
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FAX: (970) 669-2932

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DR 2800

USER MANUAL

January 2008, Edition 2

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Section 1 Specifications

Specifications are subject to change without notice.

Performance Specifications	
Operating Mode	Transmittance (%), Absorbance and Concentration
Source Lamp	Gas-filled Tungsten (visible)
Wavelength Range	340–900 nm
Wavelength Accuracy	± 1.5 nm
Wavelength Reproducibility	< 0.1 nm
Wavelength Resolution	1 nm
Wavelength Calibration	Automatic
Wavelength Selection	Automatic, based on method selection
Spectral Bandwidth	< 8 nm
Photometric measuring range	± 3.0 Ext in Wavelength Range 340–900 nm
Photometric Accuracy	5 mAbs at 0.0–0.5 Abs 1% at 0.50–2.0 Abs
Photometric Linearity	< 0.5%–2 Abs < = 1% at > 2 Abs with neutral glass at 546 nm
Stray Light	< 0.2% T @ 340 nm with KV450/3 < 0.1% T @ 340 nm with $NaNO_2$
Data storage	500 measured values (result, date, time, sample ID, user ID)
User programs	50
Physical and Environmental Specifications	
Width	220 mm (8.6 in)
Height	135 mm (5.3 in)
Depth	330 mm (12.9 in)
Weight	4.06 kg (8.95 lbs) without battery 4.38 kg (9.66 lbs) with battery
Operating Requirements	10–40 °C (50–104 °F), max. 80% relative humidity (non-condensing)
Storage Requirements	-40–60 °C (-40–140 °F) max. 80% relative humidity (non-condensing)
Additional technical data	
Mains connection	15 V- / 30VA Plug-in power supply unit: (100–240 V/50–60 Hz)
Interfaces	Use only shielded cable with max. length of 3 m. 1 x USB type A 1 x USB type B
Enclosure Rating	IP 41 with closed lid IP 42 with Protective Cover in place
Safety class	Safety class II

Section 2 General Information

2.1 Safety information

Please read this entire manual before unpacking, setting up or operating this equipment. Pay attention to all danger, warning and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

Make sure that the protection provided by this equipment is not impaired, do not use or install this equipment in any manner other than that specified in this manual.

2.1.1 Use of hazard information

DANGER
Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

WARNING
Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION
Indicates a potentially hazardous situation that may result in minor or moderate injury.

Important Note: *Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.*

Note: *Information that supplements points in the main text.*

2.1.2 Precautionary labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed. A symbol, if noted on the instrument, will be included with a danger or caution statement in the manual.

	This symbol, if noted on the instrument, references the instruction manual for operation and/or safety information.
	Electrical equipment marked with this symbol may not be disposed of in European public disposal systems after 12 August of 2005. In conformity with European local and national regulations (EU Directive 2002/96/EC), European electrical equipment users must now return old or end-of life equipment to the Producer for disposal at no charge to the user. ***Note:*** *For return for recycling, please contact the equipment producer or supplier for instructions on how to return end-of-life equipment, producer-supplied electrical accessories and all auxiliary items for proper disposal.*

LASER CLASS 1	This symbol indicates that the instrument contains a Class 1 LASER device. Data: 0.3 mW; λ = 650 nm

2.1.3 Class 1 LASER

A Class 1 LASER is installed in this instrument. Class 1 LASERS are products where the radiant power of the LASER beam accessible (the accessible emission) is always below the Maximum

Permissible Exposure value. Therefore, for Class 1 LASERS the output power is below the level at which it is believed eye damage will occur. Exposure to the beam of a Class 1 LASER will not result in eye injury. Class 1 LASERS may therefore be considered safe. However, Class 1 LASER products may contain LASER systems of a higher Class but there are adequate engineering control measures to ensure that access to the beam is not reasonably likely. Examples of such products include LASER printers and compact disc players. CDRH assession number 0510555-02.

Data: 0.3 mW; wavelength = 650 nm

2.1.4 Chemical and Biological Safety

DANGER
Potential Chemical/ Biological Exposure Hazards. Handling chemical samples, standards and reagents can be dangerous. Users of this product are advised to familiarize themselves with safety procedures and the correct use of chemicals, and to carefully read all relevant Material Safety Data Sheets.

Normal operation of this instrument may involve the use of hazardous chemicals or biologically harmful samples.

- The user must observe all cautionary information printed on the original solution containers and safety data sheet prior to their use.
- All waste solutions must be disposed in accordance with local and national law.
- The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

2.2 Overview of product

The DR 2800 spectrophotometer is a VIS spectrophotometer with a wavelength range of 340 to 900 nm. The instrument comes with a complete set of application programs and multi-language support.

The DR 2800 spectrophotometer contains the following application modes: Stored Programs (pre-installed tests), Barcode Programs, User Programs, Favorite Programs, Single Wavelength Mode, Multi-Wavelength Mode and Time Course Mode.

The DR 2800 spectrophotometer provides digital readouts in direct concentration units, absorbance, or percent transmittance.

When a user-generated or programmed method is selected, the on-screen menus and prompts direct the user through the test.

This menu system also can be used to generate reports, statistical evaluations of generated calibration curves, and to report instrument diagnostic checks.

Section 3 Installation

WARNING
Electrical and Fire Hazards. Use only the provided power supply. Only qualified personnel should conduct the tasks described in this section of the manual.

3.1 Unpack the instrument

The DR 2800 Spectrophotometer comes packaged with the following items:

- DR 2800 spectrophotometer
- Plug-in power supply, including 4 adapter for EU, GB, USA, AUS/China
- Dust cover
- 3 different cuvette/cell adapters (A, B and C)
- Light Shield belongs to standard configuration of DR 2800
- Protective Cover
- DR 2800 User Manual
- CD-ROM containing the HACH and HACH LANGE procedures
- Quick start guide

Note: *If any of these items are missing or damaged, contact the manufacturer or a sales representative immediatly.*

3.2 Operating environment

The following conditions are necessary to ensure correct instrument operation and accurate results:

- Place the instrument firmly on an even surface. Do not push any objects under the instrument.
- Maintain an ambient temperature of 10 to 40 ºC (50 to 104 ºF) for proper instrument operation.
- The relative humidity should be less than 80%; moisture should not condense on the instrument.
- Leave at least a 15 cm (6 in.) clearance at the top and on all sides for air circulation to avoid overheating of electrical parts.
- Do not operate or store the instrument in extremely dusty, damp or wet locations.
- Keep the surface of the instrument, the cell compartment and all accessories clean and dry at all times. Splashes or spills on and in the instrument should be cleaned up immediately (see section 7.1 on page 109).

Important Note: *Protect the instrument from temperature extremes, including heaters, direct sunlight and other heat sources.*

3.3 Power connections

Install the correct adapter plug on the supplied external power supply (Figure 1) by sliding the adapter on until it "clicks" into position. Correctly mounted, both housing of power supply and plug are in line. Plug the external power supply cord into the connector on the back panel of the instrument, then plug the supply into a power outlet (100–240 V~ / 50–60 Hz). Press the power switch on the back of the instrument to initialize power (Figure 2 on page 13).

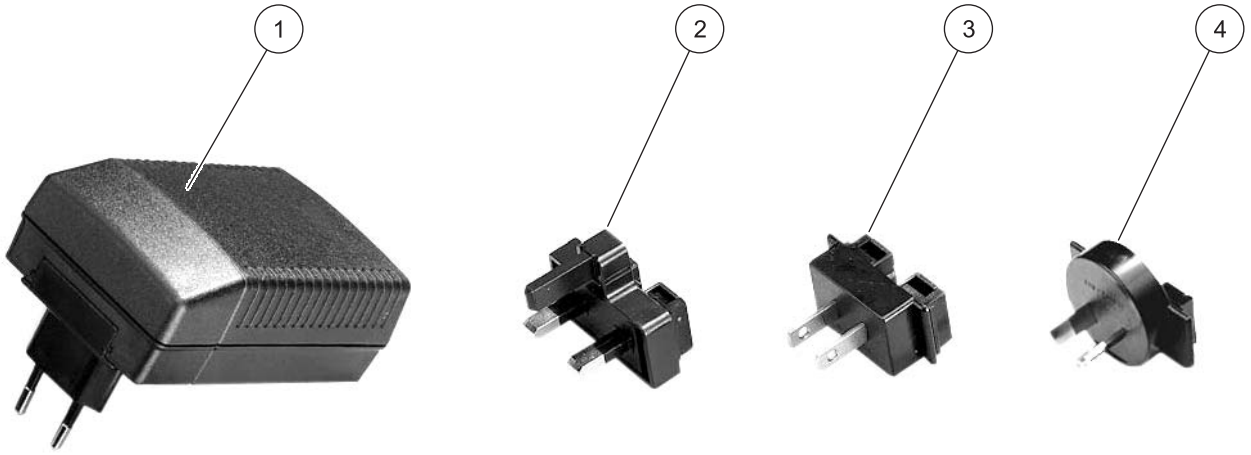


Figure 1 Power adapter

1	Power supply with EU adapter plug installed	**3**	USA adapter plug
2	UK adapter plug	**4**	AUS/China adapter plug

3.4 Connection

The DR 2800 has two USB interfaces as a standard feature, located on the back of the instrument (Figure 2).

The USB Type A interface is used for communications with a printer, USB memory stick, barcode scanner, or keyboard. A USB memory stick is used to update instrument software.

The USB Type B interface is used for communications with a PC. The optional Hach Data Trans software (see section 5.2.6.3 on page 33) must be installed on the PC for this use.

A USB hub may be used to connect several accessories at a time.

Note: *USB cables* ***must not*** *be longer than* ***3 meters*** *(10 feet).*

These USB interfaces enable data and graphics to be output to a Printer and a PC and upgrade instrument software (see section 6.7.2 on page 98).

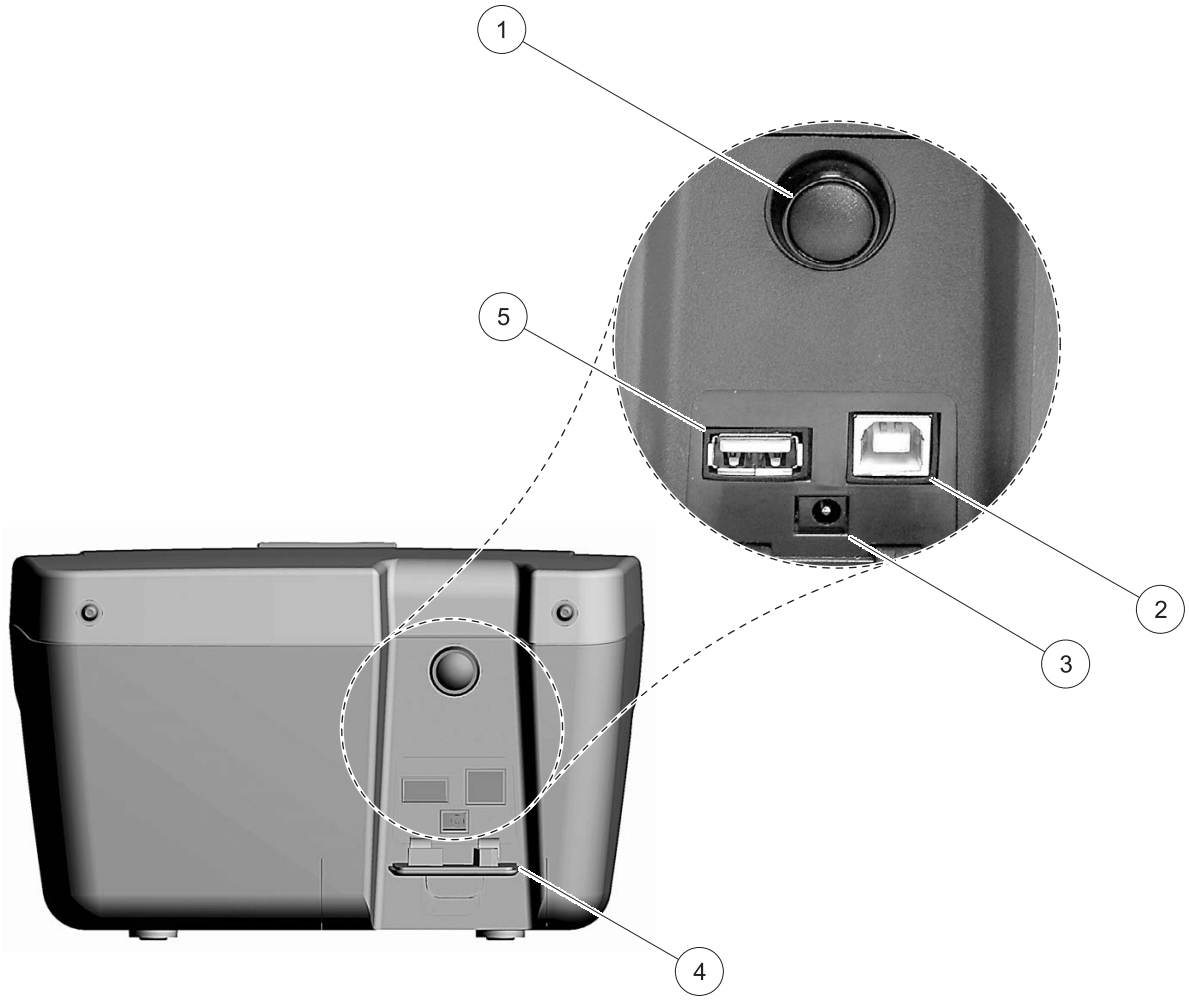


Figure 2 Interfaces

1	On/Off switch	**4**	Cover
2	USB type B	**5**	USB type A
3	Plug in power supply		

3.5 Cell compartments, Cell adapters, Light Shield and Protective Cover

3.5.1 Cell compartments and adapters

The DR 2800 has two cell compartments (Figure 3). Only one cuvette/sample cell type at a time can be used for a measurement.

Cell compartment #1

- 13-mm and 16-mm round cuvettes/vials

  Note: *Cell compartment #1 contains a barcode reader for cuvettes/vials.*

Cell compartment #2

Cell compartment #2 uses adapters to accommodate different cuvette/sample cell types.

- 1-inch square or 50-mm rectangular cuvettes/cells (can be inserted directly into the cell compartment without using an adapter).
- Adapter A: 10-mm square cuvettes/cells
- Adapter B: Pour-Thru cells (refer to the instruction sheet supplied with the Pour-Thru cell) and multi-path cuvettes/cells

  Note: *Pour-Thru Cell **must be** used with Adapter B, **not** Adapter C.*

- Adapter C: One-inch round cuvettes/cells and AccuVac® Ampules

  Note: *One-inch round cuvettes/cells and AccuVac Ampules **must be** used with Adapter C, **not** Adapter B.*

Figure 3 Cell compartments

1	Cell compartment #1	**2**	Cell compartment #2

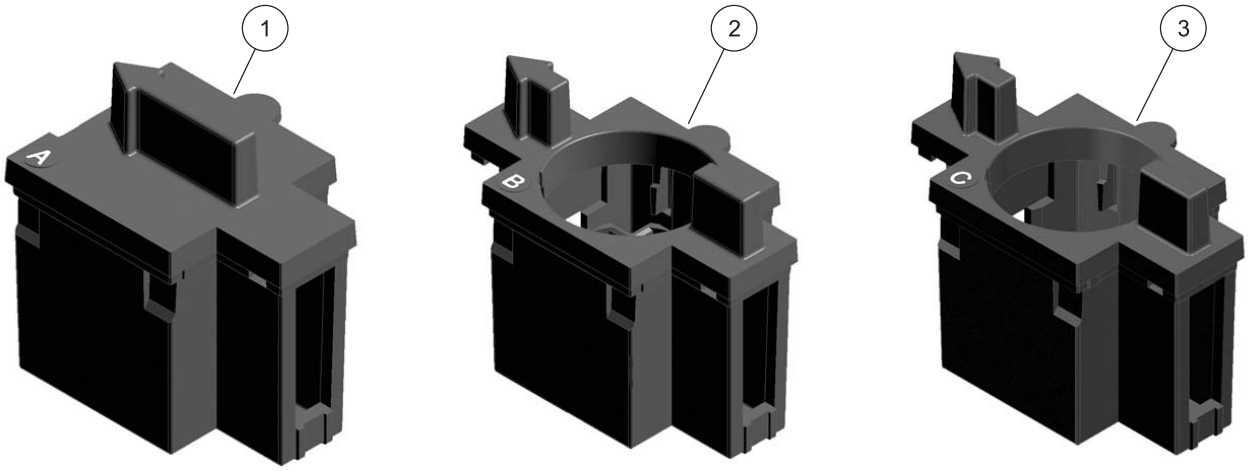


Figure 4 Cuvette/Sample cell adapters

1	10 mm square cuvette/sample cell adapter (A)	**3**	1 inch round cuvette/sample cell adapter (C)
2	1 inch Pour-Thru adapter (B)		

3.5.2 Installation of the cuvette/sample cell adapters

1. Open the cell compartment.

2. Select the correct adapter for the cuvette/sample cell type.

3. Insert the adapter so the arrow on top of the adapter points to the left (Figure 5 on page 16) and the orientation tab fits the groove in the compartment opening. The cuvette/sample cell type imprint should be legible on the adapter (Figure 4).

Note: *The arrow on top of the adapter indicates the direction of the light beam path.*

Figure 5 Installation of a cuvette/sample cell adapter

3.5.3 Use of the light shield for measurements

The light shield (Figure 6 on page 17) prevents light interference when using 13 mm and 16 mm vial tests and must be in place before measurements can be taken in cell compartment #1. The light shield is required only when using 13 mm or 16 mm vial tests.

The DR 2800 is shipped with the light shield installed. Remove the light shield before using cell compartment #2. The light shield can be stored in the Protective Cover (Figure 8 on page 18)

Figure 6 Light Shield

Installation of the light shield

1. Open the cell compartment.
2. Insert the light shield so the arrow on the light shield points to the left and the orientation tab fits the groove in the compartment opening (Figure 7).

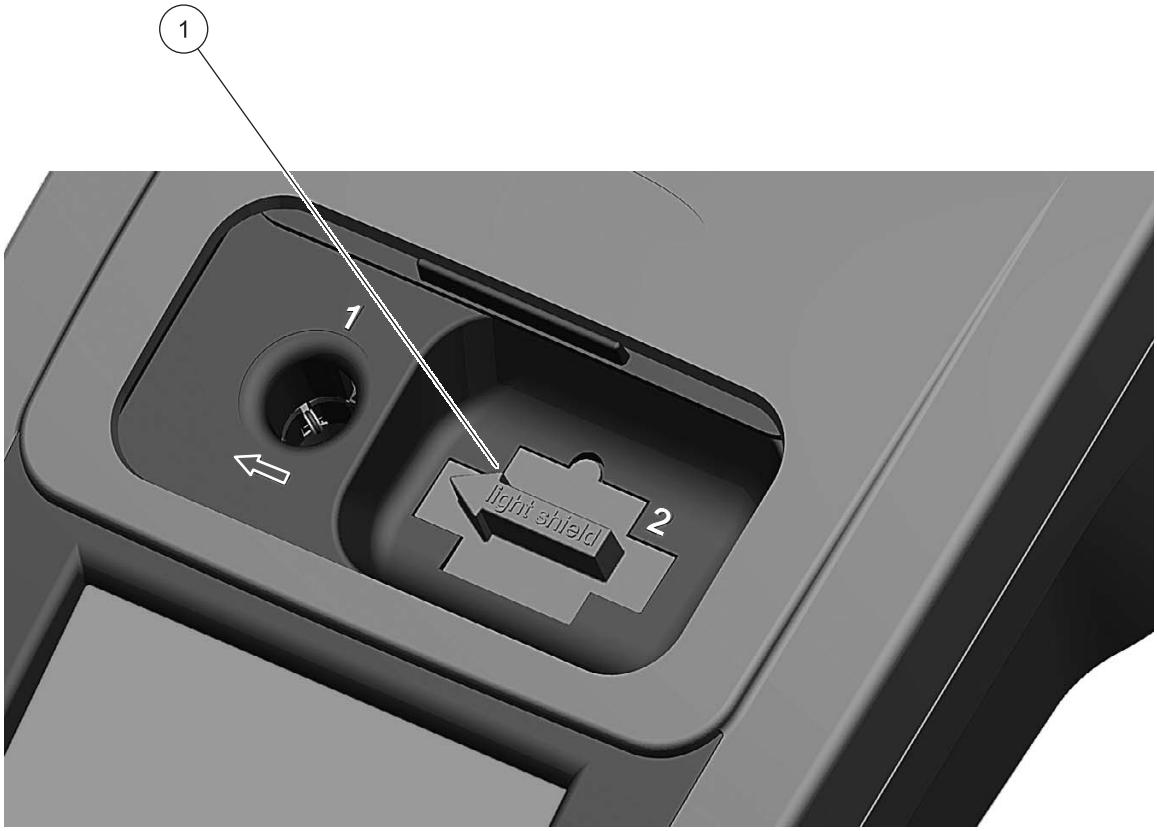


Figure 7 Light Shield in place

1	Light Shield

3.5.4 Protective Cover

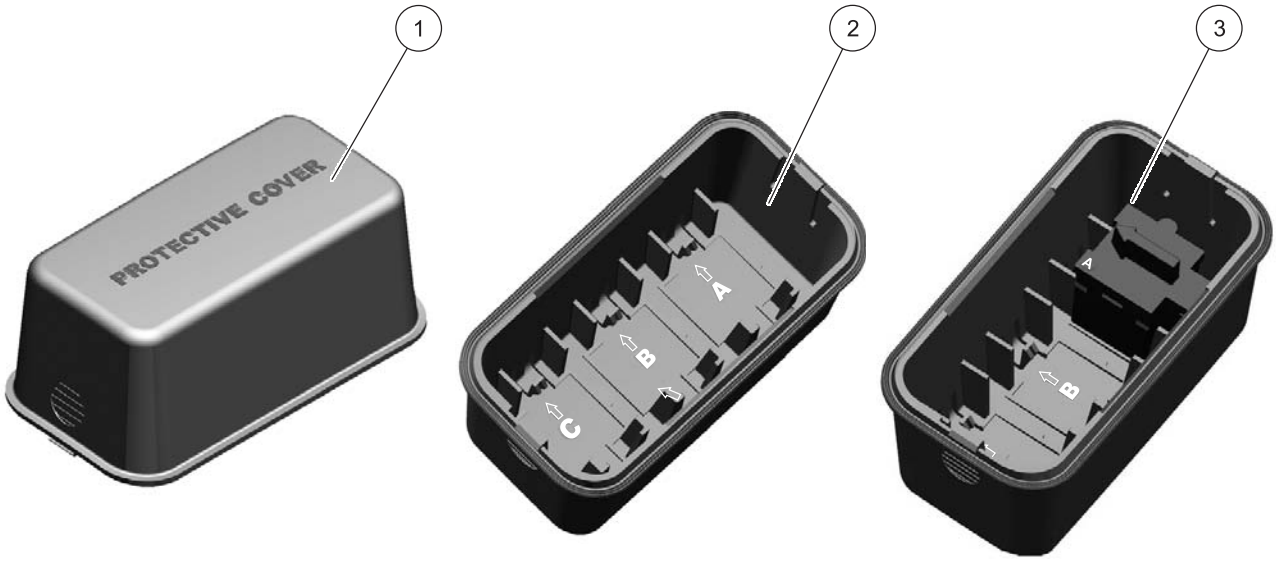


Figure 8 Protective Cover

1	Protective Cover	**3**	Protective Cover with inserted cuvette/sample cell adapter A in position A.
2	Protective Cover (inside view) The inside of the Protective Cover is intended to house the cuvette/sample cell adapters. The recesses for holding the cuvette/sample cell adapters are marked with the corresponding letters.		

3.6 Mobile use of the DR 2800 for field analysis

If the DR 2800 spectrophotometer is used to carry out field analyzes, it may be necessary to take a number of measures to compensate for the fluctuating ambient light conditions.

You should take the measures described below if the following warning is displayed after you take a measurement:
Error: Too much ambient light! Move device into shade or close the lid!

General measure:

Shield the instrument from the sun by standing so that your shadow falls on it, and carry out the measurement again. If the warning appears again, take the following measures.

Measures:

Measurements in a 10 mL round cuvette/cell, a rectangular cuvette/cell or AccuVac Ampules
Close the lid of the cuvette compartment and carry out the measurement.

Measurements in a 13 or 16 mm round cuvette/vial or a 25 mL square cuvette/cell
To obtain consistent measurement conditions in all weather conditions, place the Protective Cover on the open cuvette/cell compartment (see Figure 9 Position the Protective Cover on page 20) and carry out the measurement.

Important Note: *When you carry out measurement in 25 mL square cuvettes/cells, take care that there is no adapter in the Protective Cover.*

The enclosure rating of the photometer increases from IP41 to IP42 when the Protective Cover is in place.

3.6.1 Position the Protective Cover

Figure 9 Position the Protective Cover

1. After you have inserted the zero solution cell or the sample cell, position the Protective Cover over the open cell compartment. The lettering "Protective Cover" must be readable from the display side of the instrument.

2. Press the Protective Cover lightly into the correct position until the cell compartment is completely sealed and the Protective Cover cannot slip off.

3. Carry out the measurement.

Note: *The Protective Cover can simultaneously be used to house the different cuvette adapters. The cuvette adapter recesses in the Protective Cover are marked with the corresponding letters and arrows of the adapter. The arrows in the Protective Cover and on the adapters indicate the direction of insertion.*

3.7 Beam path

Figure 10 shows the beam path of the DR 2800:

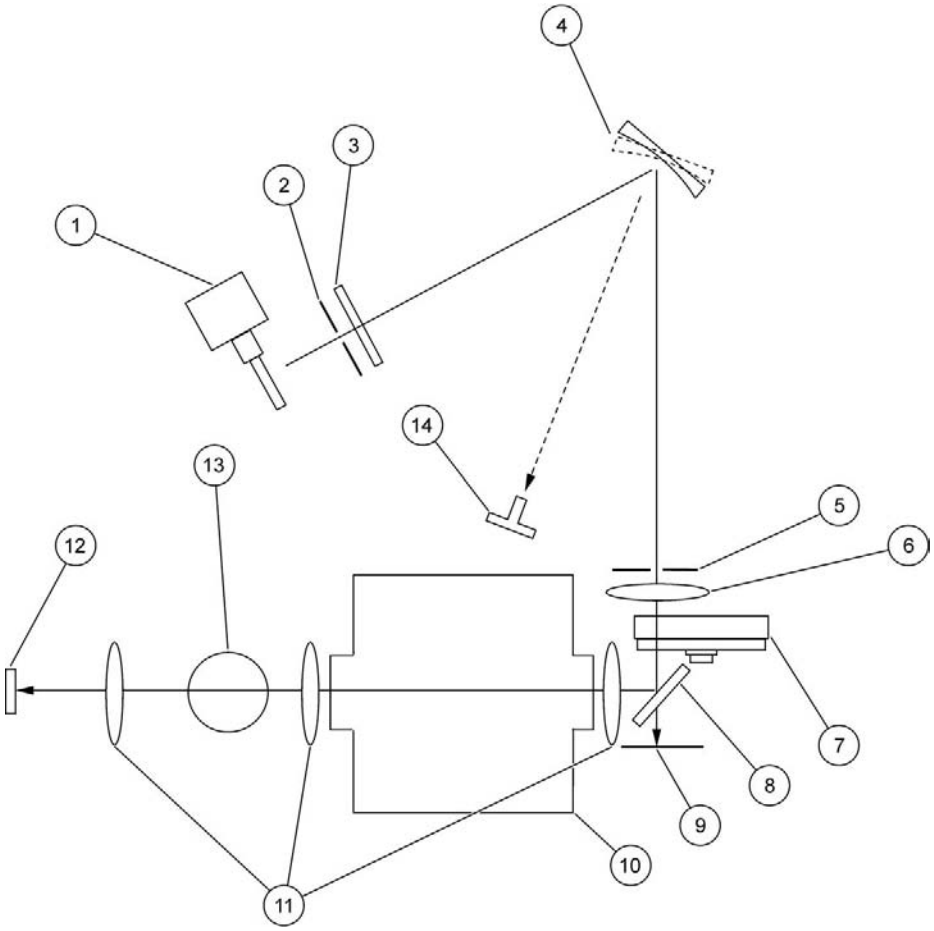


Figure 10 Beam path

1	Tungsten lamp	**8**	Splitter mirror
2	Entrance slit	**9**	Reference-element
3	Heat-protection glass	**10**	Cell compartment #2
4	Grating	**11**	Lens
5	Exit slit	**12**	Measurement element
6	Lens	**13**	Cell compartment #1
7	Filter wheel	**14**	LED

Section 4 Start Up

4.1 Power the instrument on and off

1. Plug in to the power supply (laboratory analysis) or insert the battery (field analysis).

2. The push-button switch on the back of the instrument switches the instrument on (press for about 1 second) and off (press for about 3 to 5 seconds). An acoustic signal confirms that the instrument has been switched off.

Note: *Do not turn the instrument off and on in rapid succession. Always wait about* ***20 seconds*** *before turning the instrument on again, otherwise the electronic and mechanical systems will be damaged.*

4.2 Language selection

The spectrophotometer software includes several language options. The first time the instrument is turned on, the language selection screen will appear.

1. Select the desired language.

2. Press **OK** to confirm the language selection. The self check will start automatically.

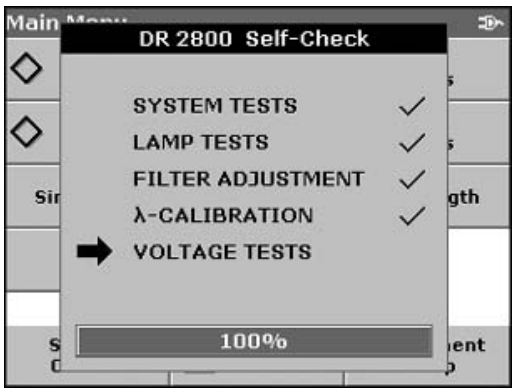
Change the language setting
The instrument functions in the selected language until the option is changed.

1. While turning the instrument on, touch the screen at any point until the list for selecting a language appears (about 30 seconds).

2. Select the required language.

3. Press OK to confirm. The test program subsequently starts automatically.

4.3 Self-Check



Each time the instrument is powered up, a series of diagnostic tests are performed automatically to ensure operation of major system components.

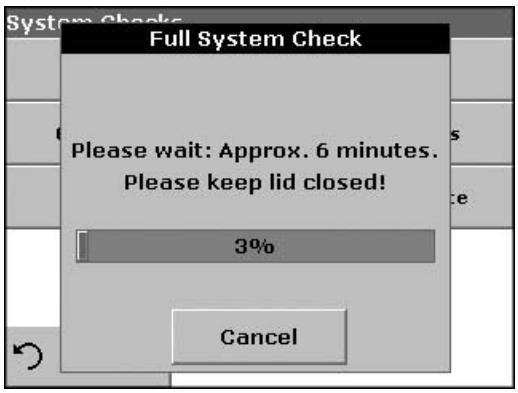
This procedure, which takes approximately two minutes, checks the system, lamp, filter adjustment, wavelength calibration and voltage. Each test which functions correctly is confirmed with a check mark.

The Main Menu is displayed when power up diagnostics are completed.

If the instrument detects any deviation relative to the last calibration, a system check has to be carried out.

4. Remove the cuvette from the cell compartment and close the lid.

5. Then press **Start**.



The system check is carried out (duration approx. 6 minutes).

Note: *Further error messages during self check, see Section 8 on page 117.*

Section 5 Standard Operations

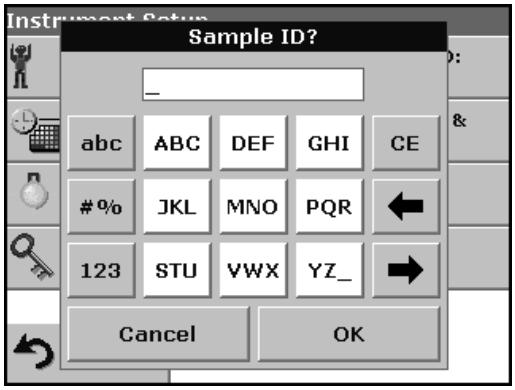
5.1 Overview

5.1.1 Tips for the use of the touch screen

The entire screen is touch-activated. To make a selection, press the screen with a fingernail, fingertip, pencil eraser or a stylus. Do not press the screen with a sharp object, such as the tip of a ball point pen.

- Do not place anything on top of the screen, to prevent damage or scratching on the screen.
- Press keys, words or icons to select them.
- Use scroll bars to move up and down long lists very quickly. Press and hold the scroll bar, then move up or down to move through the list.
- Highlight an item from a list by pressing it once. When the item has been successfully selected, it will be displayed as reversed text (light text on a dark background).

5.1.2 Use of the alphanumeric keypad



This display is used to enter letters, numbers and symbols as needed when programming the instrument. Unavailable options are disabled (grayed out). The icons on the right and left of the screen are described in Table 1.

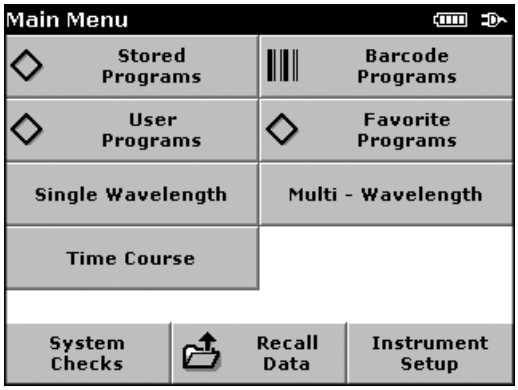
The central keypad changes to reflect the chosen entry mode. Press a key repeatedly until the desired character appears on the screen. A space can be entered by using the underscore on the **YZ_** key.

Note: *A USB keyboard (with US keyboard layout) or a USB Barcode handset scanner can be used for input (see Section 9 on page 119).*

Table 1 Alphanumeric keypad

Icon / key	Description	Function
ABC/abc	Alphabetic	When entering alphabetic characters (ex. user-entered units), this key allows to toggle between upper and lower case letters.
# %	Symbols	Punctuation, symbols and numerical sub- and superscripts may be entered.
123	Numeric	For entering regular numbers.
CE	Clear Entry	Clear the entry.
Left Arrow	Backspace	Moves back one position. This deletes the character previously entered in the new position.
Right Arrow	Advance	Moves to the next space in an entry when two adjacent characters occur on the same key.

5.1.3 Main Menu

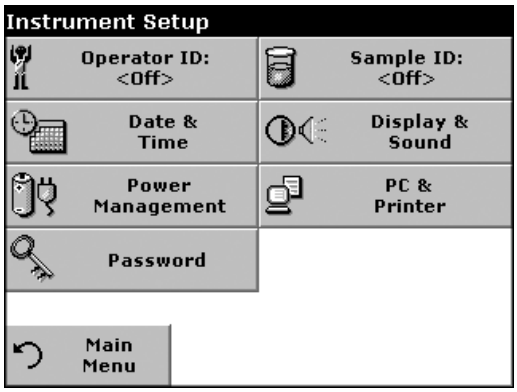


A variety of modes may be selected from the ”Main Menu“. The following table briefly describes each menu option.

Table 2 ”Main Menu“ Options DR 2800

Option	Function
Stored Programs / Barcode Programs (HACH LANGE Programs)	Stored programs are pre-programmed methods that make use of HACH reagents and LANGE cuvette tests and pipette tests. The DR 2800 Procedures Manual contains illustrated, step-by-step procedures for analyzes using HACH programs. The working procedures for LANGE tests are included in the test packs.
User Programs	User programs make "made to measure analysis" possible: - Users can program methods they have developed themselves - Existing HACH and LANGE methods can be stored as user programs. The LANGE tests can then be modified to suit the user's requirements.
Favorite Programs	List of methods/tests created by the user to suit his own requirements.
Single Wavelength	Single wavelength measurements are: **Absorbance measurements:** The light absorbed by the sample is measured in absorbance units. **Transmittance measurements (%)**: The percentage of the light that passes through the sample and reaches the detector is measured. **Concentration measurements:** A concentration factor can be entered to enable the measured absorbance values to be converted into concentration values.
Multi Wavelength	In the multi-wavelength mode, absorbance (Abs) or percentage transmittance (%T) is measured at up to four wavelengths, and absorbance differences and absorbance relationships are calculated. Simple conversions into concentrations can also be carried out.
Time Course	The time scan records the absorbance or % transmittance at a wavelength over a defined time.
System Checks	The system checks menu offers a number of options, including optical checks, output checks, lamp history, instrument update, service time and instrument backup.
Recall Data	Stored data can be called up, filtered, transmitted and deleted.
Instrument Setup	In this mode, user-specific or method-specific settings can be entered: Operator-ID, Sample-ID, Date & Time, Display & Sound, Power Management, PC & Printer and Password.

5.2 Instrument Setup mode

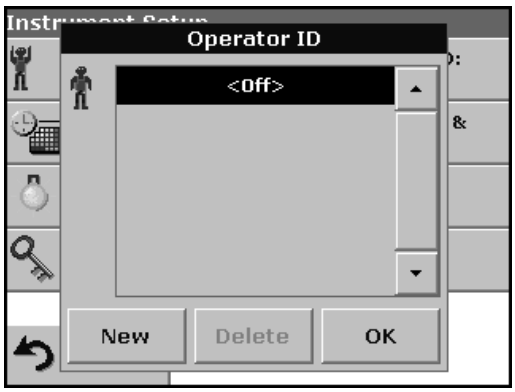


1. Select **Instrument Setup** in the ”Main Menu“.

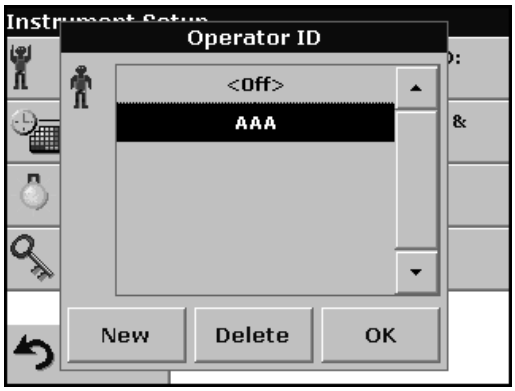
A selection of functions appears in order to configure the functions of the instrument.

5.2.1 Operator ID

Use this option to enter up to 30 sets of operator initials (up to 5 characters each) into the instrument. This feature helps record which operator measured each sample.



1. Press **Operator ID** in the Instrument Setup.
2. Press **New** to enter a new Operator ID.
3. Use the alphanumeric keypad to enter a new Operator ID.
4. Press **OK** to confirm.



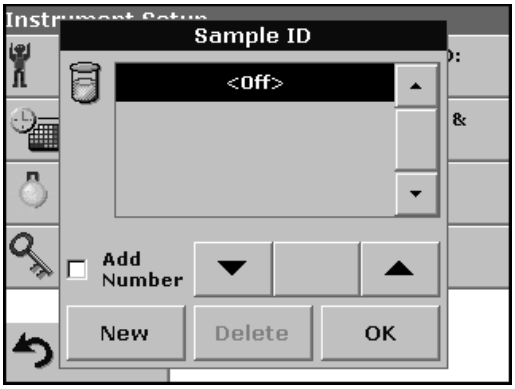
5. The display shows the chosen Operator ID.
6. Press **OK**. The instrument will return to the Instrument Setup screen and show the selected operator identifier.
7. The chosen Operator ID is activated.

Note: *Press **Delete** to remove an Operator ID from the list.*

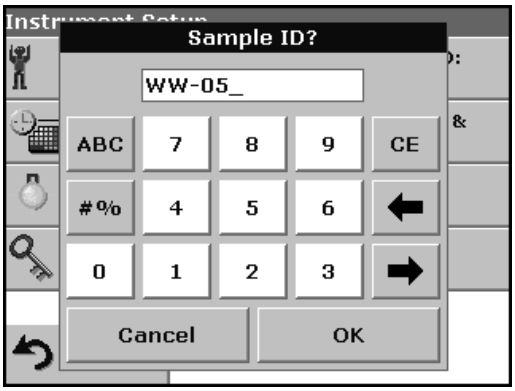
Note: *Alternatively, enter or change an Operator ID in measurement mode. In the results screen, press **Options**>**More**>**Instrument Setup** or if an Operator ID is already assigned, select the "Operator ID symbol immediately in the results screen.*

5.2.2 Sample ID

Use this option to enter up to 100 Sample Identification tags (up to 13 characters each) into the instrument. Sample IDs can be used to specify the sample location or other sample specific information.



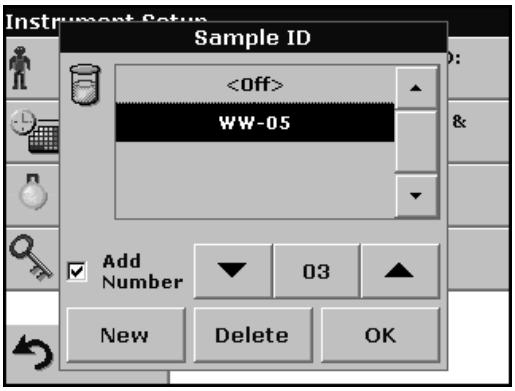
1. Press **Sample ID** in the Instrument Setup.
2. Press **New** to enter a new Sample ID.



3. Use the alphanumeric keypad to enter a new Sample ID.

   Note: *If a USB Barcode handset scanner (see Section 9 on page 119) is connected, Sample IDs can also be scanned. Sample IDs can also be entered with a USB keyboard.*

4. Press **OK** to confirm.

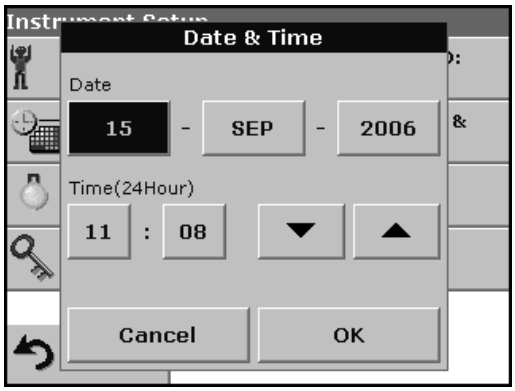


5. To number the Sample IDs sequentially (e.g. Inflow (01 etc.)), select **Add Number.**
   - Use the arrow keys to specify the first number of the sequence.
   - Use the key between the arrow keys to enter the first number of the sequence using the alphanumeric keypad.
6. Press **OK** to return to "Instrument Setup".
7. The Sample ID is activated. Each Sample ID is automatically numbered in ascending order after a measurement. The number is shown in parentheses behind the Sample ID.

Note: *To remove a Sample ID, highlight the ID and press Delete.*

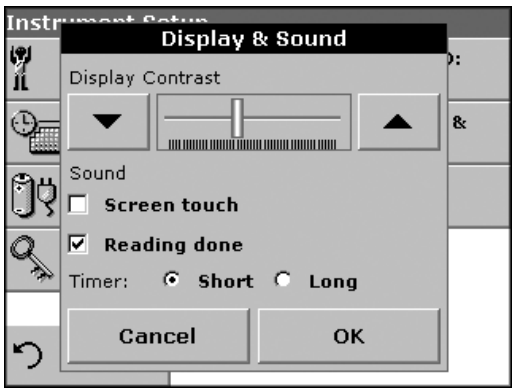
Note: *A Sample ID can be entered or changed in measurement mode. In the results screen, press* ***Options***>***More***>***Instrument Setup****. If a Sample ID is already assigned, select the Sample ID symbol in the results screen.*

5.2.3 Date and time



1. Press **Date & Time** in the Instrument Setup.
2. The date and time are subdivided over a number of fields. Press the appropriate field and use the arrow keys to change the value.
3. Press **OK** to confirm. The instrument will return to Instrument Setup.

5.2.4 Display and sound preferences



1. Press **Display & Sound** in the Instrument Setup.

The following options will be displayed:

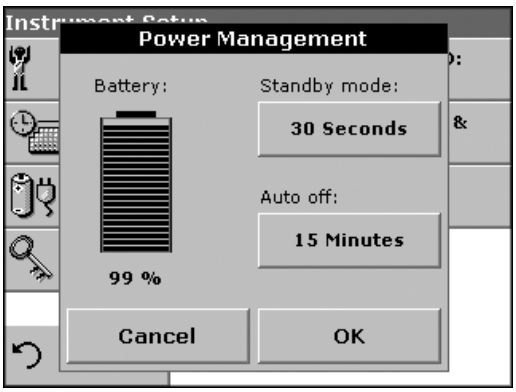
- **Display Contrast**—Adjusts the display brightness to suit lighting conditions.
- **Screen touch**—Activates//Deactivates a short beep each time the screen is pressed (Default:off).
- **Reading done**—Activates/Deactivates a sound when a reading is complete (Default: short beep every time a reading is complete).
- **Timer**—Adjusts the length of the timer sound. Select Short or Long. Long beeps are recommended for noisy environments.

2. Press **OK** to confirm. The instrument will return to Instrument Setup.

5.2.5 Power Management

The DR 2800 can run on mains or battery power.

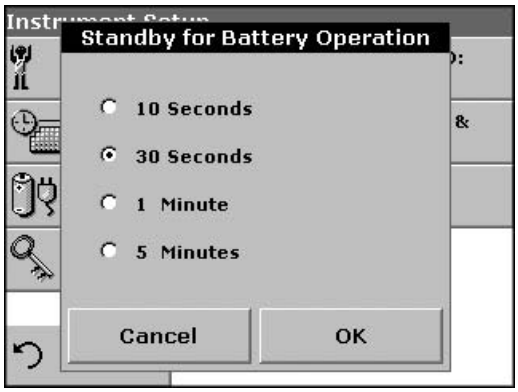
Note: *The battery is* ***not*** *part of the standard scope of delivery.*



1. Select **Power Management** in the ”Instrument Setup“ menu.

   The battery symbol indicates the charge status of the battery in %.

   Note: *The timer settings in the* ***Power Management*** *menu are only active when the instrument is running on battery power.*



2. Select one of the options under **Standby mode** to set the length of the idle period that can elapse before the instrument switches to the Standby mode when running on battery power.

Note: *In standby mode, the backlighting of the screen is switched off. Touching the display causes the lighting to switch on again.*

3. Select one of the options under **Auto off** to set the length of the idle period that can elapse before the instrument automatically switches off when running on battery power.

Note: *After the instrument switches itself off automatically, you have to press the push-button switch on the back of the instrument to start it again.*

5.2.6 PC and printer

The instrument is provided with 2 USB interfaces, which are located on the back of the instrument (see Figure 2 on page 13). These interfaces can be used for exporting data and graphics to a printer, updating data and for data communication to a personal computer. These interfaces can be used for the connection of a USB stick, an external USB keyboard or a USB Barcode handset scanner.

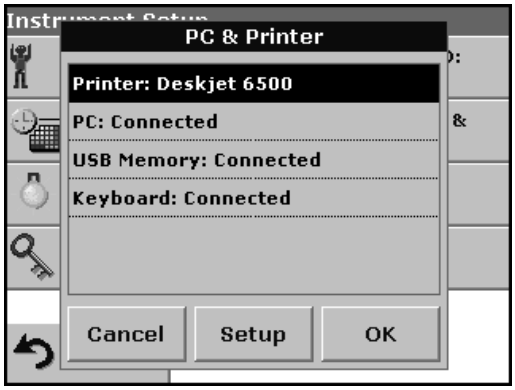
Note: *A USB hub may be used to connect several accessories at a time.*

A USB memory stick is used to upgrade data and software, see section 6.7.2 on page 98.

Important Note: *A screened* ***USB cable*** *must not be longer than* ***3 m****!*

Table 3 USB connector

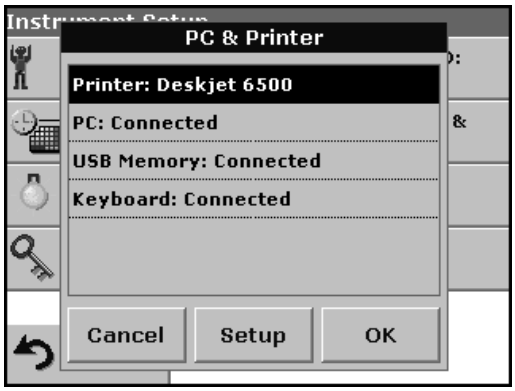
USB Interfaces	Description
USB (Type B)	This USB interface is only intended to connect the instrument to a PC (with installation of the HACH Data Trans Software).
USB (Type A)	This USB port can be used to connect a printer, a USB memory stick , a barcode scanner or a keyboard.



1. Press **PC & Printer** in the Instrument Setup.

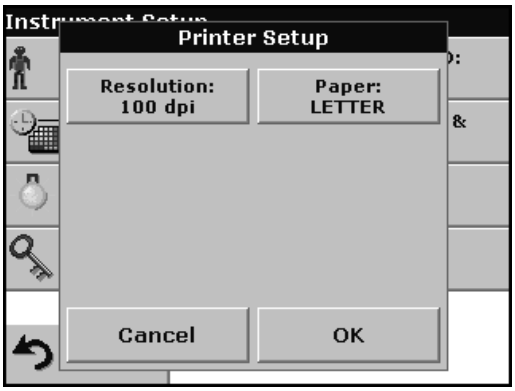
   A list with information about the connections opens.

5.2.6.1 Printer setup



For reasons of compatibility, the printer language must be **HP PCL 3**.

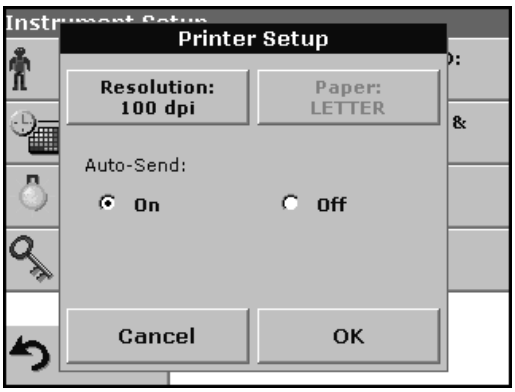
2. Press **Printer**.
3. Press **Setup** to display the Printer Setup screen.



Printer Setup:

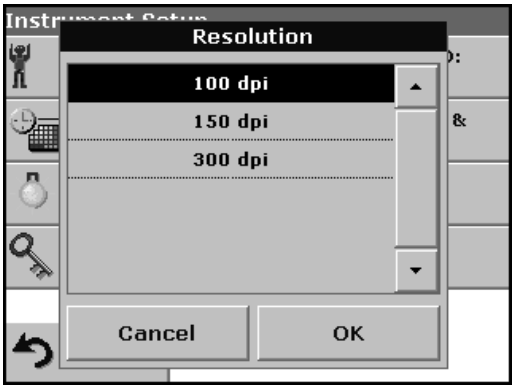
- Resolution: Print quality
- Paper: Paper size

Note: *If an optional Thermal Printer is connected, the function "Auto Send" on/off is available.*



4. Select **Auto-Send: On** to send all measured data automatically to the Thermal printer.

Note: *The option Auto-Send is **not** available for any other printer (e.g. ink jet printer).*



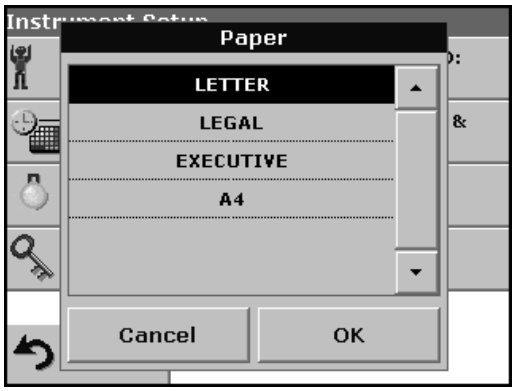
5. Press **Resolution** to select the print quality.

Select between

- 100 dpi
- 150 dpi and
- 300 dpi

6. Press **OK** to confirm.

Note: *Press **OK** again to return to the **PC & Printer** menu.*



7. Press **Paper** to select the paper size.

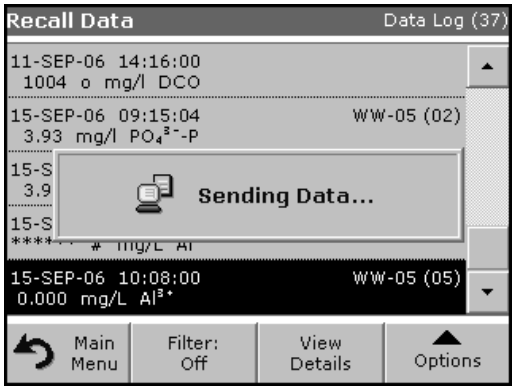
   Select between

   - Letter
   - Legal
   - Executive
   - A4

8. Press **OK** to confirm.

Note: *Press **OK** again to return to the **PC & Printer** menu.*

5.2.6.2 Print data

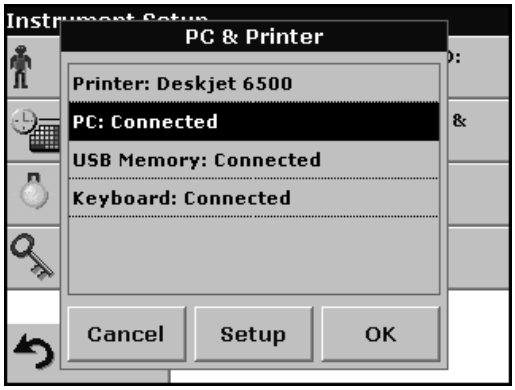


1. Press **Recall Data** in the Main Menu.
2. Select the data source, where the data to be printed are stored.
3. A list is displayed. Data can be filtered. For more information see section 5.3.1.2 on page 37.
4. Press the **PC & Printer** icon to send the data (table, curve) immediately to the printer.
5. Highlight **Single point** or **Filtered data** or **All data** and press **OK** to confirm.

Sending Data... is displayed until the data have been printed.

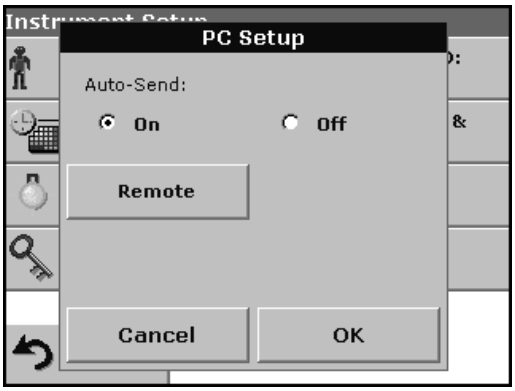
5.2.6.3 HACH Data Trans

The optional HACH Data Trans software must be installed on the PC for the subsequent to process for measurement data.



1. Press **PC & Printer** in the Instrument Setup.
2. Select **PC**.
3. Press **Setup** to display the PC Setup screen.

   For further installation instructions, refer to the HACH Data Trans user manual.



4. Select **Auto-Send: On** to send all measured data automatically to the PC.

   Note: *If* ***Auto-Send: Off*** *is selected, the* ***PC & Printer*** *icon must be pressed, in order to send data to the PC.*

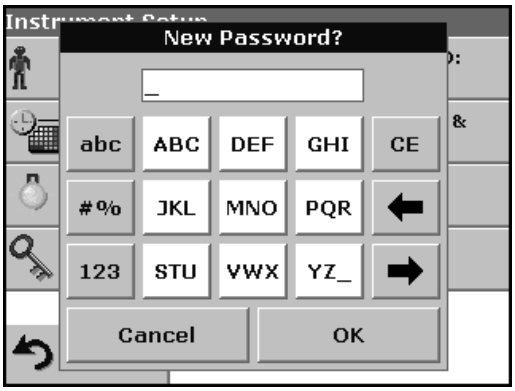
Note: *The remote function is only for monitoring the data transfer.*

5.2.7 Password

The Password menu contains a variety of security settings to control access to various functions. For example, prevent unauthorized changes to stored programs or instrument configurations.

1. Press **Password** in the Instrument Setup menu.

2. In order to highlight the **Security List** assign a password. Press **Set Password**.



3. Use the alphanumeric keypad to enter a new Password (up to 10 characters each) and press **OK** to confirm.

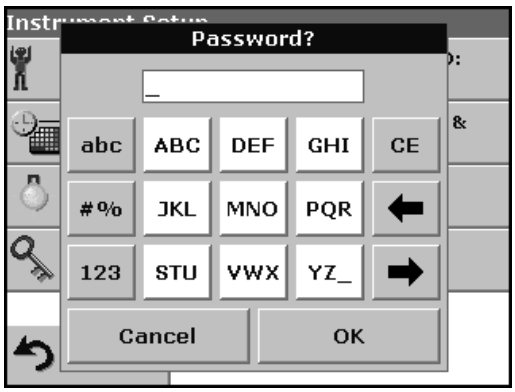
   The access to the Security List is activated.

4. Press **Security List** to lock various functions for unauthorized users.

5. Highlight the desired functions to control.
6. Confirm the **Security List** with **OK** to return to the Password menu.
7. Press **On** to highlight the new settings of the Security List.
8. Enter the new Password again to confirm.
9. Press **OK** to return to Instrument Setup.

Note: *The alphanumeric keypad to the Password inquiry appears when a user tries to reach a locked setting.*

5.2.7.1 Password deactivation



1. Press **Password** in the Instrument Setup.
2. Use the alphanumeric keypad to enter the former Password and press **OK** to confirm.

3. Press **Off** to deactivate the settings of the Security List.

4. Press **OK** to return to Instrument Setup.

Note: *Use this function to delete the former Password or to enter a new one.*

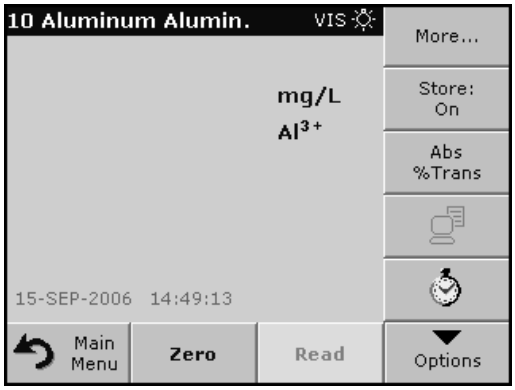
5.3 Store, recall, send and delete data

5.3.1 The data log

The Data Log will store up to 500 readings taken in the modes: Stored Programs, Barcode Programs, User Programs, Favorite Programs, Single Wavelength and Multi Wavelength. A complete record of the analysis is stored, including the Date, Time, Results, Sample ID, and Operator ID.

5.3.1.1 Auto/manual data storage

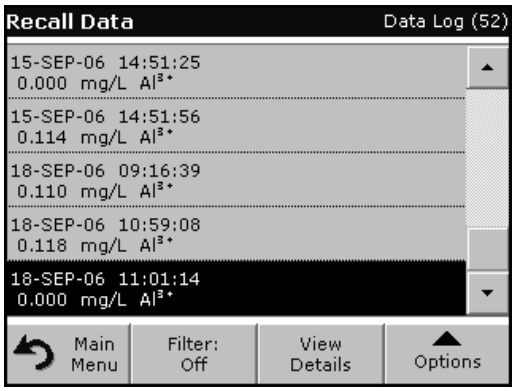
The data storage parameter indicates whether data are to be stored automatically or manually (in which case the user has to decide which data to store).



1. Press **Store: On/Off** in the Options menu.
   - With the **Store On** setting, all measurement data are stored automatically.
   - With the **Store Off** setting, no measurement data are stored. However, this setting can be changed to **Store On** in the result display through configuration. The reading currently shown in the display is then stored.

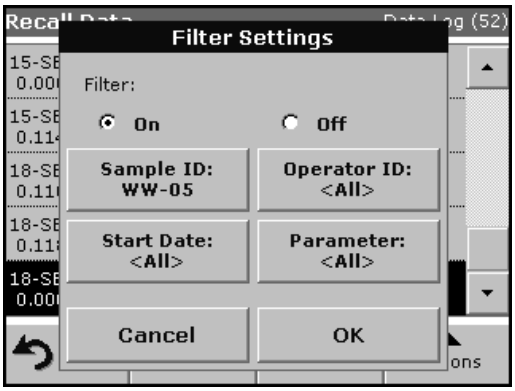
Note: *When the instrument's memory (data log) is full, the oldest data are automatically deleted allowing the new data to be stored.*

5.3.1.2 Recall stored data from the data log



1. Press **Recall Data** in the Main Menu.
2. Press **Data Log**.

   A listing of the stored data is displayed.
3. Press **Filter: On/Off**.

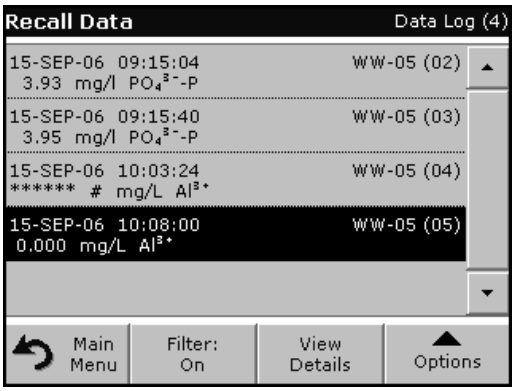


4. The function **Filter Settings** is used to search for specific items.

5. Highlight **On** to turn on the filters to select data by

   - Sample ID
   - Operator ID
   - Start Date
   - Parameter

   or any combination of the four.



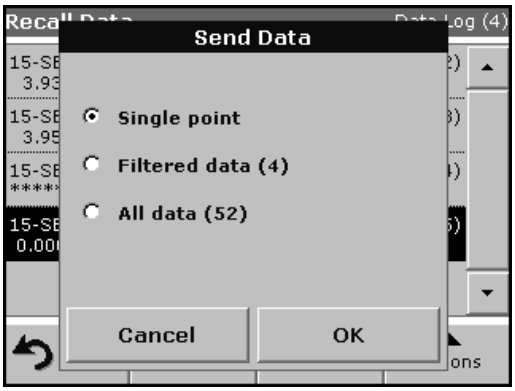
6. Press **OK** to confirm the selection. The chosen items are listed.

7. Press **View Details** to get more information.

5.3.1.3 Send data from the data log

Data is sent from the data log as CSV (Comma Separated Value) files through a USB memory stick to a file named DATALOG. The file can then be processed using a spreadsheet program. The file name will be formatted as: DLYear_Month_Day_Hour_Minute_Second. CSV.

To send data to a Printer, see section 5.2.6.2 on page 33.



1. Plug in the USB device (Figure 2 on page 13).

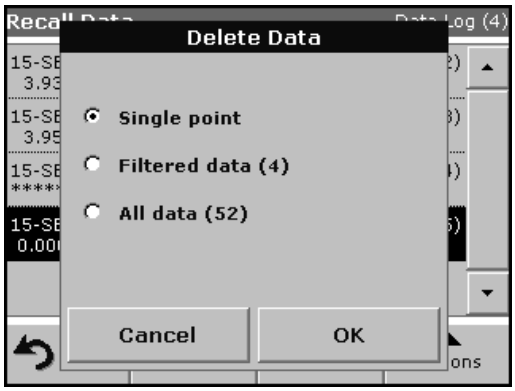
2. Press **Recall Data** from the Main Menu. Press **Options** and then the **PC & Printer** icon.

3. Select the data to send to the memory stick and press **OK**.

   Note: *The number in parenthesis is the total number of data sets assigned to this selection.*

To send measurement data to a PC:
The optional HACH Data Trans software must be installed on the PC, see section 5.2.6.3 on page 33.

5.3.1.4 Delete stored data from the data log



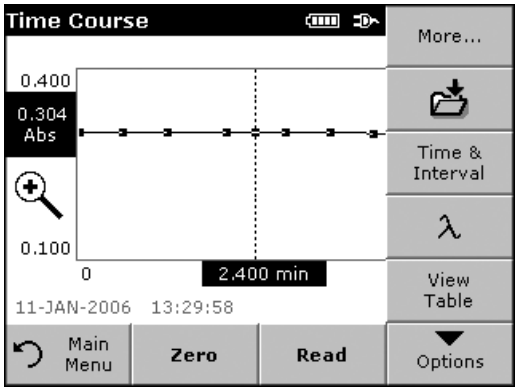
1. Press **Recall Data** in the Main Menu.
2. Press **Data Log>Options>Delete**.
3. Highlight **Single Point** or **Filtered data** or **All data** and press **OK** to confirm.

   Note: *The number in parentheses is the total number of data sets assigned to this selection.*

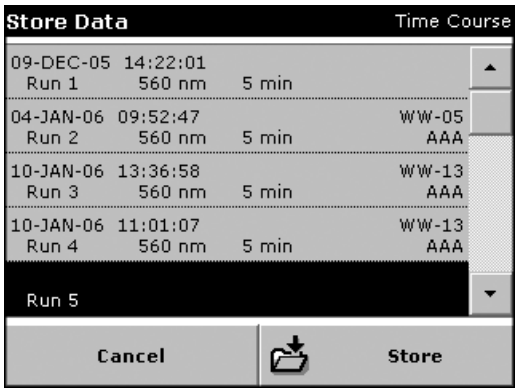
5.3.2 Time Course

The instrument can store 20 Time Course Data sets. The data can be stored manually at the user's discretion after viewing the data.

5.3.2.1 Data Storage from Time Course



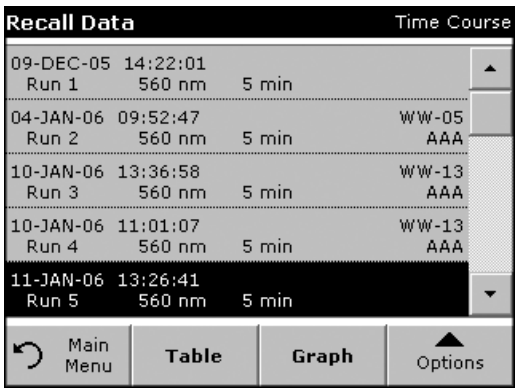
1. Press the **Store icon** in the ”Options“ menu from Time Course mode after a reading is taken.



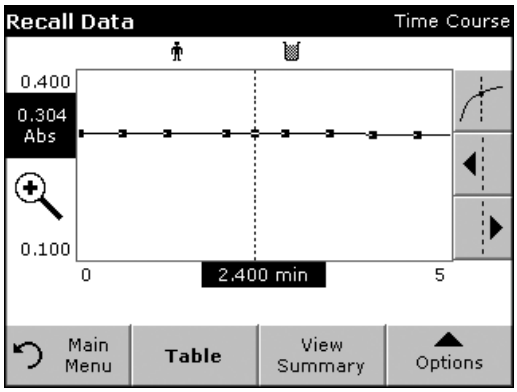
The Store Data list opens.

2. Press **Store** to save the current scan to the highlighted numbered line. A run can also be overwritten.

5.3.2.2 Recall Stored Data from Time Course

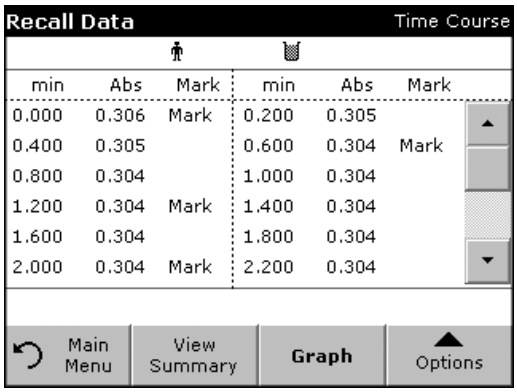


1. Press **Recall Data** in the ”Main Menu“.
   a. Select **Time Course** to call up the required data.
   b. If you are already working in this program, press **Options**, **More...** and then **Recall Data**.



2. Press **Graph** to look at details.

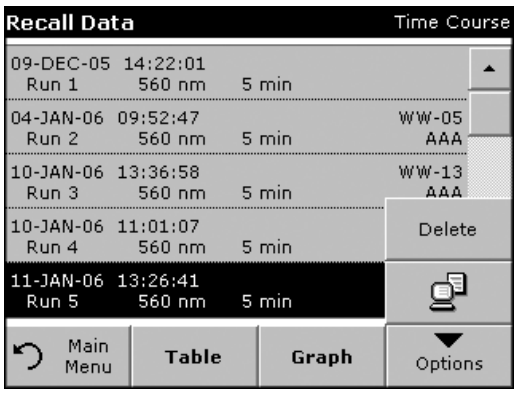
Note: *Press* ***View Summary*** *to return to the Recall Data list.*



3. Press **Table** to look at details.

Note: *Press* ***View Summary*** *to return to the Recall Data list.*

5.3.2.3 Send Data from Time Course



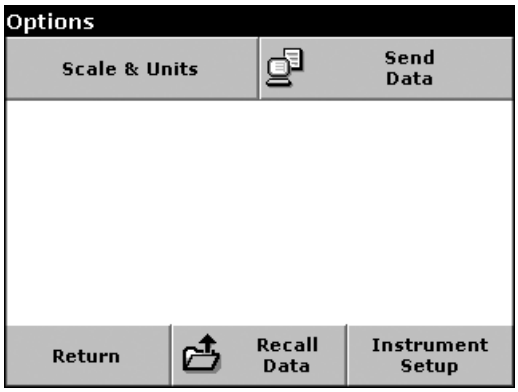
Option 1:

4. Press **Recall Data** in the ”Main Menu“ and then **Time Course** to send the data to a USB memory stick or to a printer.

The files will be automatically send as CSV files (Comma Separated Value) to a file ”TCData“ (Time Course Data).

The file name will be formatted as: ”TCData_X.csv”
X = number of scans (1–20)

For further processing use a spreadsheet program.



Option 2:

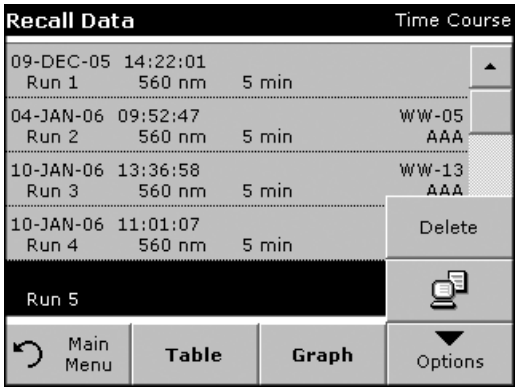
5. Press **Time Course** and then **Options**, **More...** and **Send Data** to send the data to a USB memory stick or to a printer.

   The files will be automatically send as CSV files (Comma Separated Value) to a file ”TCData“ (Time Course Data).

   The file name will be formatted as: ”TCYear_Month_Day_Hour_Minute_Second.CSV“ (Time Course Data)

   For further processing use a spreadsheet program.

5.3.2.4 Delete Stored Data from Time Course



1. Press **Recall Data** from the ”Main Menu“ and then **Time Course** or ”Options“ menu, **More...**, **Recall Data**:

   A listing of the stored data is displayed.

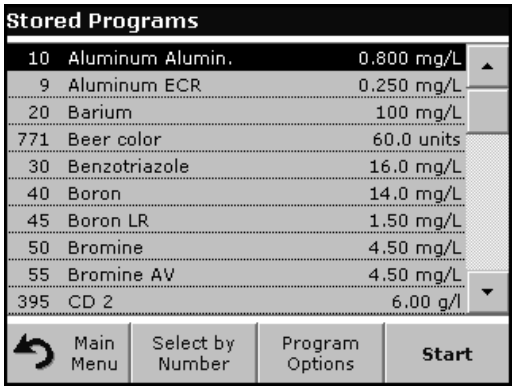
2. Highlight any data you want to delete.

3. Press **Delete** in the ”Options“ menu and confirm with **OK**.

5.4 Stored Programs

The instrument contains more than 200 programmed procedures. They can be accessed through the **Stored Programs** menu.

5.4.1 Select a saved test/method; enter user-specific basic data



1. Press **Stored Programs** in the Main Menu to view an alphabetical list of stored programs with program numbers.

   The Stored Programs list will appear.

2. Highlight the required test.

   Note: *Select the program number by name or use the arrow keys to scroll through the list quickly and highlight the program or press* ***Select by number*** *to search for a specific program number. Use the alphanumeric keypad to enter the test number and press* ***OK****.*

3. Press **Start** to run the program. After a program is selected, the screen for that parameter will appear.

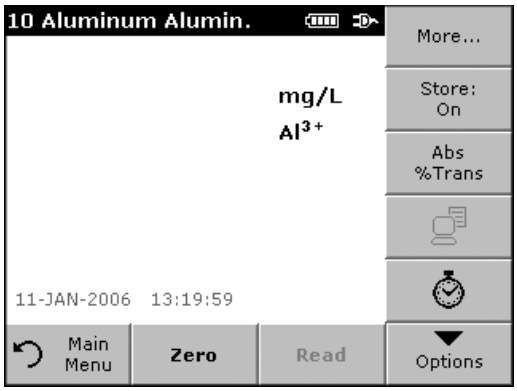
   Note: *All corresponding data (wavelength, factors and constants) are already preset.*

4. Follow the chemical procedures described in the corresponding Procedures Manual.

5.4.2 Stored program options

1. From the Main Menu, select **Stored Programs**. Select the necessary method and press **Start**.

2. Press **Options** for Parameter Setup. Refer to Table 4 for stored program descriptions.



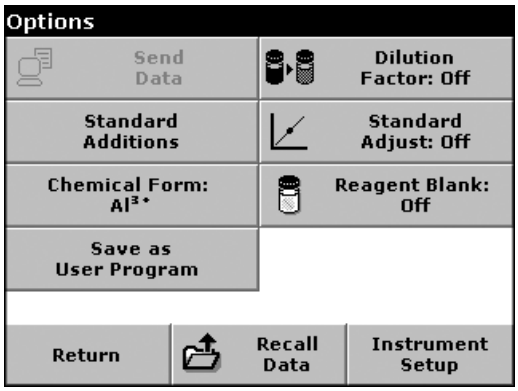
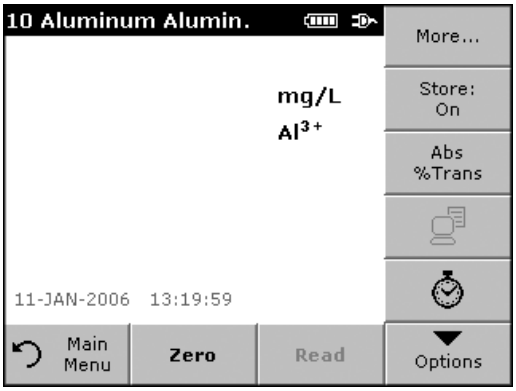


Table 4 Stored programs options

Options	Description
More	For further Options



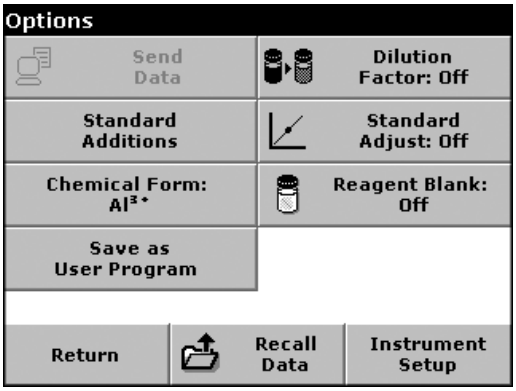
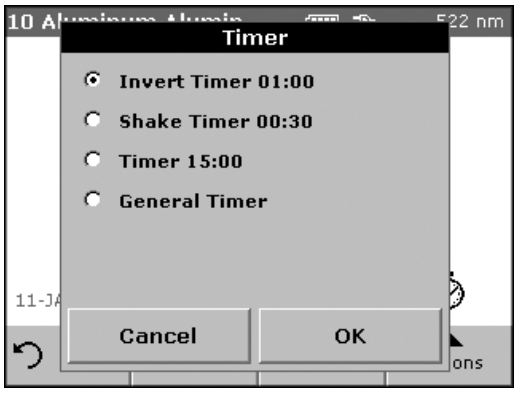


Table 4 Stored programs options (continued)

Options	Description
Store Off/On	With the **Store On** setting, all measurement data are stored automatically. With the **Store Off** setting, no measurement data are stored.
% Trans/Conc/Abs	To switch to % transmittance, concentration or absorbance readings
Send Data icon / Send Data	To send Data to a printer, computer or USB memory stick (Type A)
Timer icon	This functions as a stopwatch. It helps to ensure that the steps of an analysis are correctly timed (e.g. reaction times, wait times, etc., can be exactly specified). When the specified time has elapsed, an acoustic signal is emitted. The use of the timer has no influence on the measurement program.
Dilution Factor Off/On	A corrective dilution factor can be entered in order to take account of certain properties. The number entered at the dilution factor prompt will be multiplied by the result to compensate for the adjustment. For example, if the sample has been diluted by a factor of 2, enter 2. The default setting of the dilution factor is turned off. ***Note:*** *When a dilution is in effect, the dilution icon will appear on the display.*
Standard Addition	This enables the accuracy of the measurements to be checked. The (working) procedure for a test parameter contains a detailed explanation of how to use this function.
Standard Adjust	The (working) procedure for a test parameter indicates whether a standard adjustment is necessary and, if so, how to proceed.
Chemical Form	Some of the stored tests/methods allow to select the chemical form and the measuring range.
Reagent Blank	Some of the stored tests/methods include the "Reagent Blank" function. This enables the reagent blank value to be added to or subtracted from, the subsequent readings. The reagent blank value shifts the calibration curve along the y-axis, without changing the shape or gradient of the curve. The effect corresponds to a y-axis intercept of the calibration straight line. This is made clear by the following equation: Concentration = [(Conc. factor) * Abs] – (reagent blank value).
Save as User Program	To store the selected parameters as a User Program, see section 6.1 on page 61.
Recall Data	Call up saved measurement data or time courses, see section 5.3 on page 37.
Instrument Setup	Basic data of the instrument, see section 5.2 on page 27.

5.4.3 Use of program timers

Some procedures do not require the use of timers. Other procedures require several timers. These timers are pre-programmed into each **Stored Program**, along with a description of the activity to be performed during the timed period.



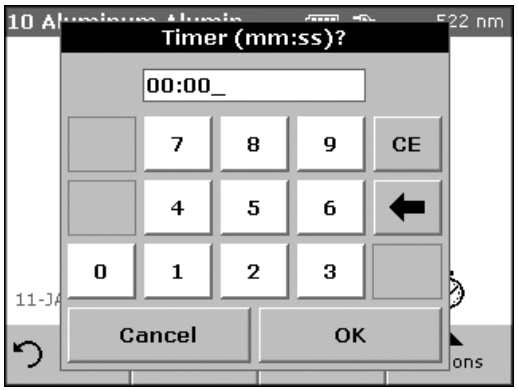
1. Press the **Timer** icon on the display.
2. Press **OK** to start the first timer.

   The timer will count down on the screen.
3. To start the next timed activity for the Stored Program, press the Timer icon and **OK**.

   Note: *Press **Close** to view the measurement screen while the timer is running. The time will be shown left side bottom instead of the date.*

   Note: *Press **Cancel** to stop the timer midway through the countdown.*

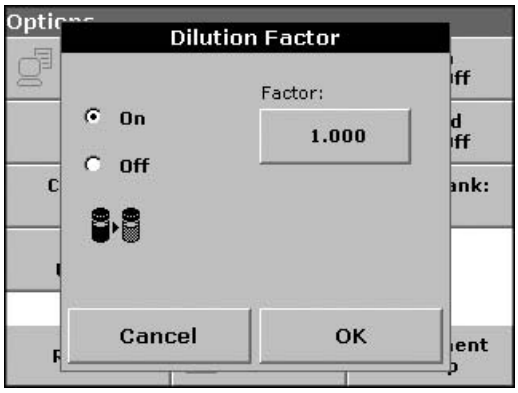
   Note: *The Timer will beep when the end of the running time is reached.*



A general purpose timer is also available in many programs. When the timer icon is visible, press the icon and select General Timer. A new screen will appear. Enter the length of the timed interval and press OK to start the timer. The timer will beep when the timed interval ends.

5.4.4 Set the dilution factor

The Dilution Factor function is used to calculate the original concentration of a sample that has been diluted by a known ratio. For example, dilution factor can bring the analyzed concentration within the test range.



1. Press **Options**>**More…**>**Dilution Factor**.

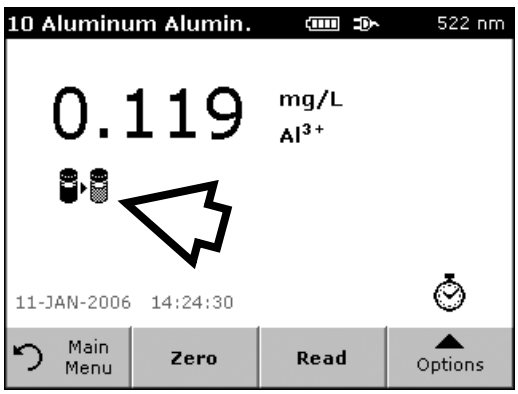
   The number entered at the dilution factor prompt will be multiplied by the result to compensate for the adjustment.

   For example, if the sample has been diluted by a factor of 2, enter 2. The default setting of the dilution factor is turned off.

2. Press **OK** to confirm. Press again **OK**.

3. Press **Return** to return to the result display.

4. Confirm 'Store present reading again?' to save the current measured value if necessary.



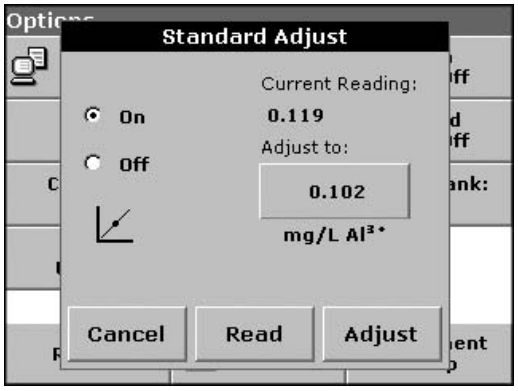
Note: *When a dilution is in effect, the dilution icon will appear on the display.*

Note: *If using undiluted samples, set the dilution factor off again.*

5.4.5 Run a standard adjust

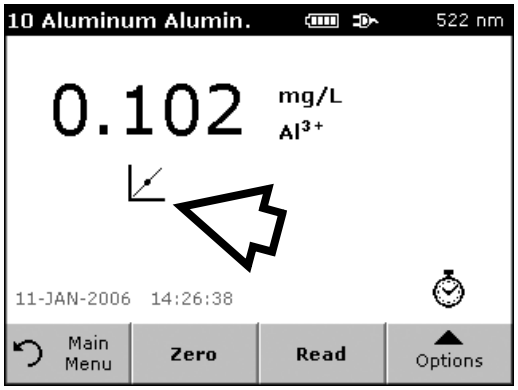
The Standard Adjust function allows the calibration curve for a stored program to be adjusted based on analysis of a known standard solution. The Accuracy Check section of written procedures often suggests a standard solution concentration for this purpose.

Read a standard before setting Standard Adjust to On.



1. Follow the entire procedure, using a known standard for the sample.
2. After reading the concentration, press **Options>More>Standard Adjust**.
3. If Standard Adjust is set to **Off**, turn it **On**.

   The Current Reading will show the concentration. The box on the right will show the default standard value for the test, as mentioned in the procedure.
4. If the measurement used a standard concentration that is different from the one displayed in the box, press the box on the right to enter a different standard value and enter the new value. Press **OK** to confirm.



5. Press **Adjust** to enable the Standard Adjust. The Standard Adjust icon will appear.

   Note: *The adjustment must be within certain limits, which vary with each program. The allowable percentage is shown after "Adjustment".*

   Note: *When a Standard Adjust is in effect, the Standard Adjust icon will appear on the display.*

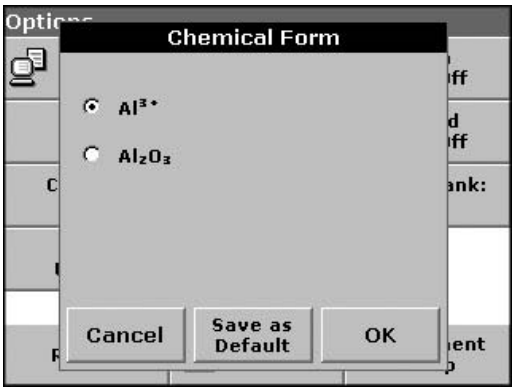
5.4.6 Set the chemical form

Some Stored Programs allow a variety of chemical forms to be selected.

Press the unit (e.g. mg/L) or the chemical representation of the evaluation form (e.g. Al^{3+}). A list of available evaluation forms is displayed. Select the required form by pressing the corresponding entry in the list.

Note: *When the program is exited, the evaluation form reverts to the standard setting.*

An alternative way of changing the standard setting:



1. Press **Options>More>Chemical Form**.
2. Select the Chemical Form.

   Note: *The stoichiometric conversion of the measurement result is carried out automatically.*

   Note: *The selected Chemical Form will appear on the display. Test results will be calculated and displayed in this chemical form.*

5.4.6.1 Change of the default setting of the chemical form

1. Insert the sample cuvette or blank (depending on the working procedure) into the cell compartment.
2. In the result display, press **Options>More>Chemical Form**.
3. A list of available evaluation forms appears. Select the new default setting.
4. Press **Save as Default.**

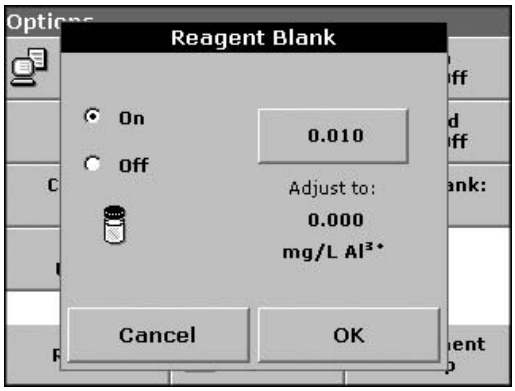
The current result and all further measurements will be displayed in the new chemical form.

5.4.7 Run a reagent blank

Some of the stored tests/methods include the "Reagent Blank" function. This enables the reagent blank value to be measured and then taken into account in calculating the measurement result.

Measurement/analysis of a reagent blank:

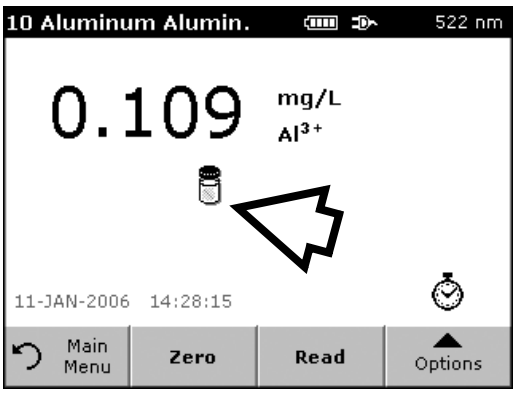
1. Prepare the test/method in accordance with the (working) procedure. Instead of a sample, deionized water is used to determine the reagent blank value.
2. Select the test. If required by the (working) procedure, insert the cell with deionized water into the cell compartment. Press **Zero**.
3. Insert the prepared sample cell into the cell compartment. Press **Read**. The result is displayed.



4. Press **Options>More>Reagent Blank**.
5. Press **On** to highlight the Reagent Blank function.
6. The concentration shown on the key is the measured value of the reagent blank. To use this value for more analyses of this parameter, press **OK**.
7. To enter a previously recorded reagent blank value, press the key and use the alphanumeric keypad.
8. Press **OK**.

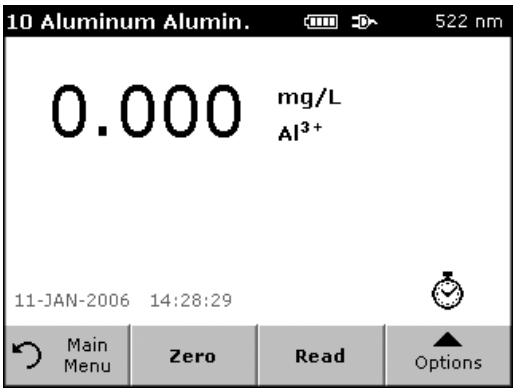
Note: *The Reagent Blank function is deactivated when the measurement program is left. To use the same blank value later for other tests using the same reagent lot, enter the value per step 7.*

Note: *The results calculated using the reagent blank value must lie within the limits of the measuring range of the test/method.*

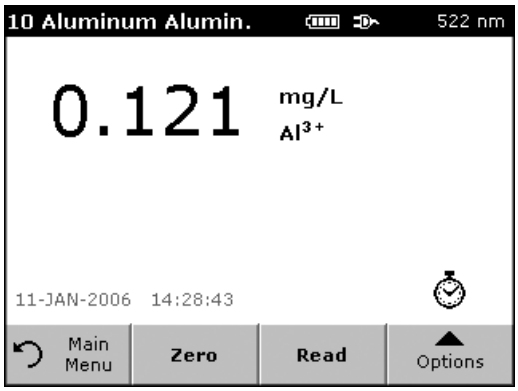


Note: *The reagent blank icon is shown in the result display (see arrow) when the function is active.*

5.4.8 Analysis of samples



1. Press **Stored Programs** and select a program.
2. Insert the blank cuvette/cell into the cuvette/sample cell holder.
3. Press **Zero**.



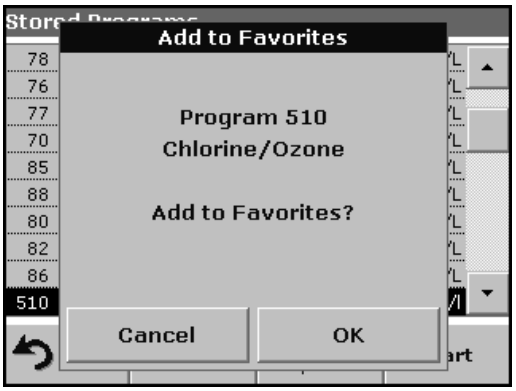
4. Remove zero solution and insert sample cuvette/cell into the cell compartment.

5. Press **Read**. The result will be displayed.

6. For data storage, see section 5.3.1 on page 37.

5.4.9 Add stored programs to the favorite programs list

The Favorites menu simplifies test selection by creating a list of the most frequently used tests from the Stored Programs and User Programs.



1. Press **Stored Programs** in the Main Menu. The Stored Programs list will appear.

2. Highlight the selection by pressing it or **Select by Number** to search for the program by number.

3. Press **Add to Favorites** and press **OK** to confirm.

   The program can now be selected from **Favorite Programs** menu in the Main Menu.

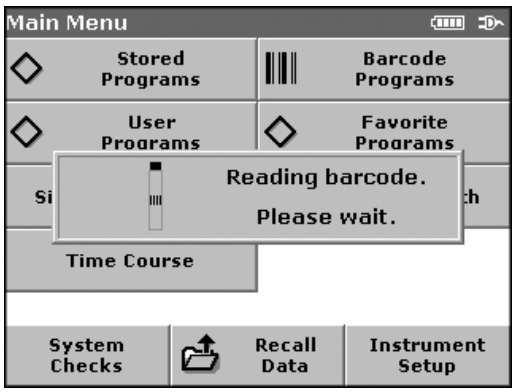
5.5 Barcode Programs

A special barcode reader in cell compartment #1 automatically reads the barcode on the 13 mm cuvette/vial as the cuvette/vial completes a single rotation. The instrument uses the barcode identification to automatically set the correct wavelength for the analysis and calculates the result immediately with the help of the stored factors.

In addition, measured values are recorded at 10 different positions during a rotation. A special outlier-elimination program is run and then the average of the measured values is calculated. Cuvette/vial errors and soiling are recognized and highly precise results are obtained.

Figure 11 Cell compartment #1 for barcode 13 mm cuvettes/vials

5.5.1 Complete a barcode 13 mm test/vial



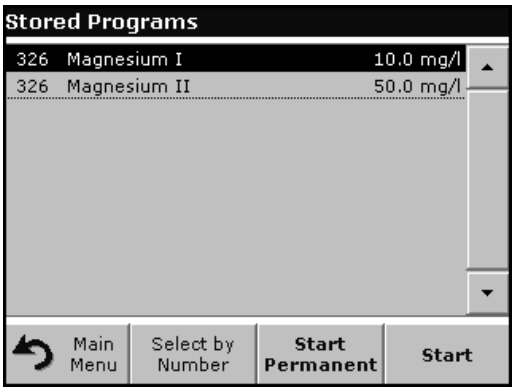
1. Insert the light shield in cell compartment #2.
2. Prepare the barcode 13 mm cuvette/vial in accordance with the working procedure and insert the cuvette/vial in cell compartment #1.
   - When a coded cuvette/vial is placed in cell compartment #1 (Figure 11), the corresponding measurement program is automatically activated in the Main Menu.

- Otherwise, press **Barcode Programs** in the Main Menu. and insert the blank or sample cuvette/vial (depending on the working procedure) in cell compartment #1.

The measurement is started automatically and the results are displayed.

To evaluate other cuvette/vial tests and other parameters, insert the prepared cuvette/vial into the cell compartment and read the result.

5.5.2 Select the measuring range



Some tests can be used for different measuring ranges. After the sample cuvette/vial has been inserted, a list of the different measuring ranges is displayed.

Select the required measuring range by highlighting the appropriate line.

Press **Start Permanent** if this measuring range is to apply to all subsequent measurements.

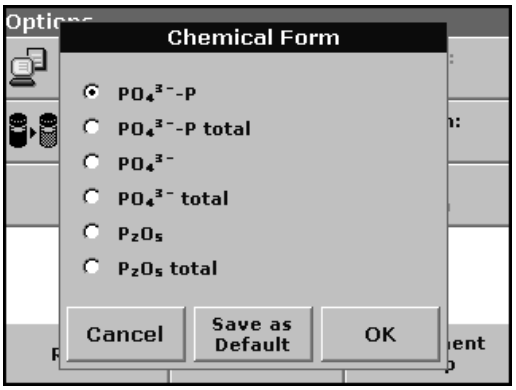
Changing the standard setting
In the result display, press **Options>More>Permanent: On**. The key will change to **Permanent: Off.**

5.5.3 Select the chemical evaluation form

The chemical form of the test result of a number of parameters can be selected individually.

In the result display, press the unit (e.g. mg/L) or the chemical representation of the evaluation form (e.g. PO_4^{3-}–P). A list of possible evaluation forms is displayed, from which the required form can be selected. Press **OK** to confirm.

Another way of changing the standard setting is:



1. In the result display, press **Options>More>Chemical Form**.

   A list of available evaluation forms appears.

2. Select the required chemical form and press **OK** to confirm.

   Note: *The selected chemical form is displayed, but does not become the default. To change the default, see section 5.5.3.1.*

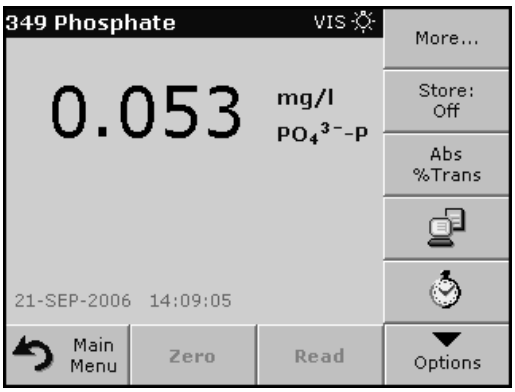
5.5.3.1 Change of the default setting of the chemical form

1. Insert the blank or sample cuvette/cell (depending on the working procedure) into the cell compartment.
2. In the result display, press **Options>More>Chemical Form**.
3. A list of available evaluation forms appear. Select the new default setting.
4. Press **Save as Default**.

The current result and all further measurements will be displayed in the new chemical form.

5.5.4 Basic test-specific and sample-specific data settings

Press **Options** to change test or sample specific settings.



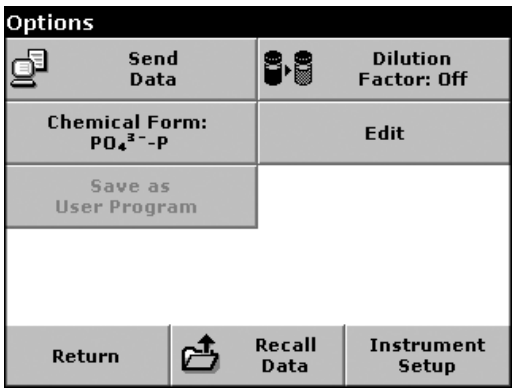
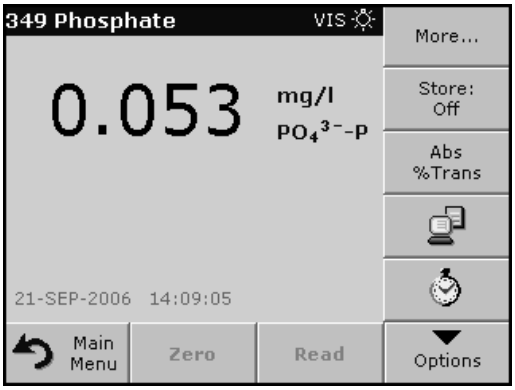


Table 5 Barcode program options

Options	Description
More	For further Options
Store Off/On	With the **Store On** setting, all measurement data are stored automatically. With the **Store Off** setting, no measurement data are stored.
Abs % Trans	To switch to % transmittance or absorbance readings
Send Data icon / Send Data	To send data to a printer, computer or USB memory stick (Type A)
Timer icon	This functions as a stopwatch. It helps to ensure that the steps of an analysis are correctly timed (e.g. reaction times, wait times, etc., can be exactly specified). When the specified time has elapsed, an acoustic signal is emitted. The use of the timer has no influence on the measurement program.
Dilution Factor Off/On	A corrective dilution factor can be entered in order to take account of certain properties. The number entered at the dilution factor prompt will be multiplied by the result to compensate for the adjustment. For example, if the sample has been diluted by a factor of 2, enter 2. The default setting of the dilution factor is turned off. ***Note:*** *When a dilution is in effect, the dilution icon will appear on the display.* ***Note:*** *If undiluted samples are used, set the dilution factor off.*
Chemical Form	Some of the stored tests/methods allow to select the chemical form and the measuring range.
Edit	To modify an existing program



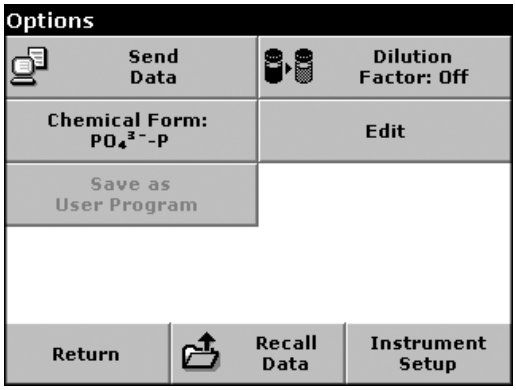
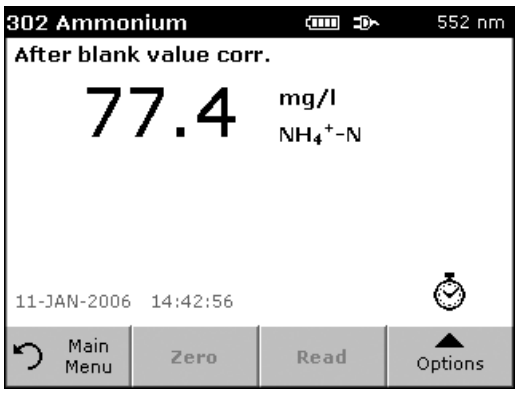


Table 5 Barcode program options (continued)

Options	Description
Save as User Program	To store the selected parameters as a User Program, see section 6.1 on page 61.
Recall Data	Call up saved measurement data or time courses, see section 5.3 on page 37.
Instrument Setup	Basic data of the instrument, see section 5.2 on page 27.

5.5.5 Sample blank



Turbidity and color in the sample matrix can falsify the results of a photometric analysis. The interference factors come from the sample or are created by reactions with the reagents.

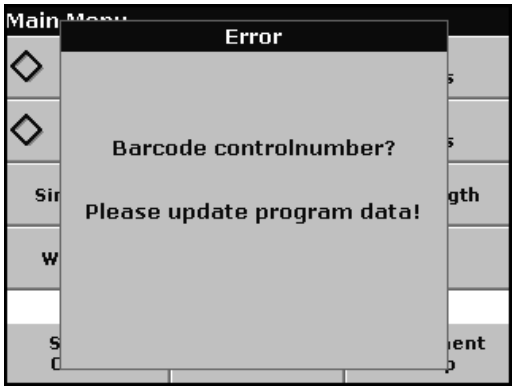
The influence of turbidity and/or color can be eliminated or reduced by taking a sample blank reading.

In the barcode mode, a special cuvette/vial (TNT919) containing the sample blank is placed in cell compartment #1 after the sample reading has been taken and is automatically measured. The sample reading is then corrected by adding or subtracting the blank value. The final result is displayed, with the message "After blank value corr.".

Some barcode tests do not require a sample blank value to be determined, as turbidity and color are dealt with during the test procedure. Other barcode tests use the reagent test vials to determine the sample blank. Please refer to the Hach procedure for method-specific instructions.

5.5.6 Update/edit barcode tests

5.5.6.1 Manual update of a barcode test

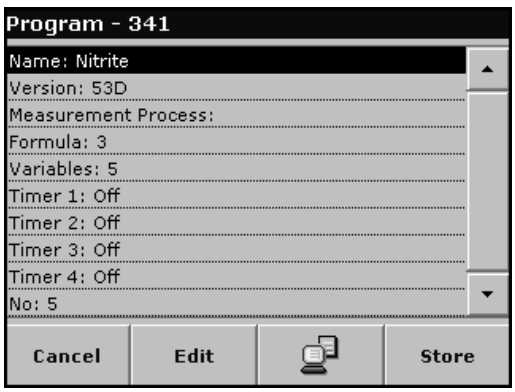


Using the data provided in the barcode, the instrument automatically sets the measurement wavelength and factors. If a discrepancy is detected between the barcode data and the stored data or a new test is identified, the instrument requests an update.

Data types are described in Table 6.

Table 6 Program update options

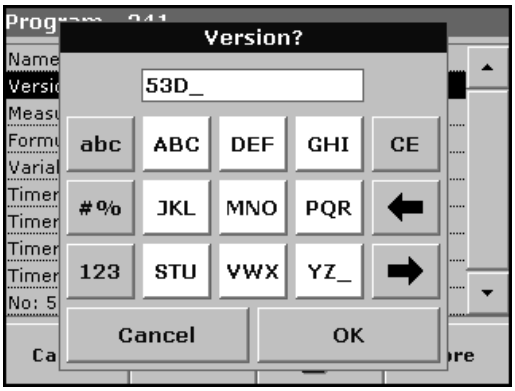
Program point	Description
Name	Name of the analysis parameter
Version	An abbreviation assigned by the user or the version number, etc. is entered here
Measurement process	Exact definition of the test: the number of wavelengths at which measurements are carried out, the number of absorbance measurements needed, the keys to be used, any waiting periods between measurements, etc.
Formula	Definition of the evaluation formulas with which the test result is calculated
Variables	The number of variables shown in the display depends on the definition of the measurement process and the formulas. Input of the numerical values of the wavelengths, factors, constants, etc.
Timer 1, Timer 2, Timer 3, Timer 4	This function can be used to enter abbreviations and defined times for up to four timers. Highlight the appropriate line and press **Edit**. The timers are activated or deactivated with the control boxes on the left of the display. In the next column, a selection can be made from a list of names that describe the corresponding work step. In the third column, the times for each active timer are entered.



Enter the Name
Check the working procedure in advance to determine whether this point needs to be changed.

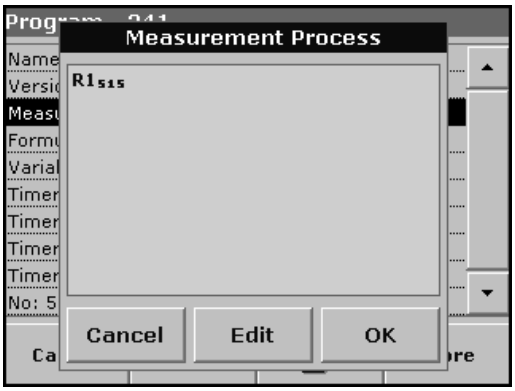
1. Highlight the line containing **Name** and press **Edit**. Use the alphanumeric keypad to enter the name indicated in the working procedure.

2. Press **OK** to confirm.



Enter the Version

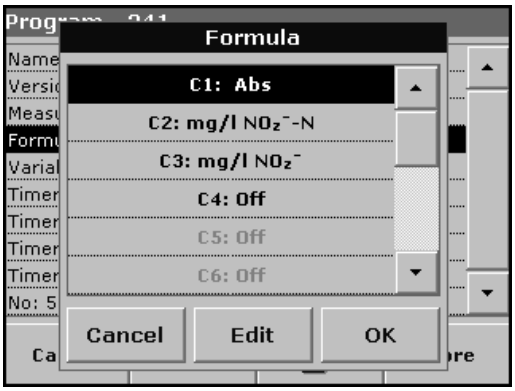
1. Highlight the line containing **Version** and press **Edit**. Use the alphanumeric keypad to enter the name indicated in the working procedure.
2. Press **OK** to confirm.



Enter the Measurement Process
For detailed information on entering the measurement process, see section 6.1.2 on page 72.

Check the working procedure in advance to determine whether this point needs to be changed.

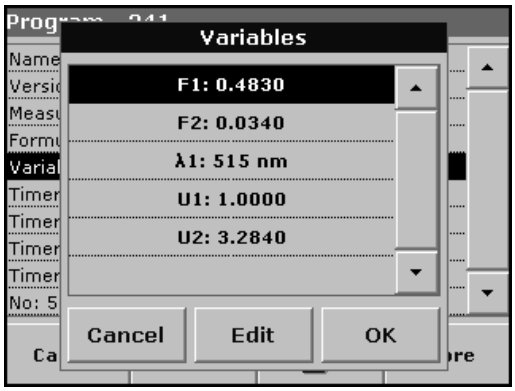
1. Highlight the **Measurement Process** line in the data overview and press **Edit**.
2. Press **Edit**, select the sequence to be edited and press **Delete**.
3. Press **New** and use the alphanumeric keypad to enter the process indicated in the working procedure.



Enter the Formula, Concentration Units, Designation, Measuring Ranges
For detailed information on entering the Formula, see section 6.1.2 on page 72.

Check the working procedure in advance to determine whether this information needs to be changed.

1. Highlight the **Formula** line in the data overview and press **Edit**.
2. Select the formula, press **Edit** and use the alphanumeric keypad to enter the data specified in the working procedure (for C1=, C2=, units, name, measuring range limits, etc.).



Enter the Variables (Factors, Wavelength and Coversion Factors)
For detailed information on entering the Variables, see section 6.1.2 on page 72.

Check the working procedure in advance to determine whether this point needs to be changed.

1. Highlight the **Variables** line in the data overview and press **Edit**.

2. Select the variable, press **Edit** and use the alphanumeric keypad to enter the data specified in the working procedure (for F1, F2, λ1, U1 etc.). Confirm each entry with **OK**.

Abbreviation of Variables:

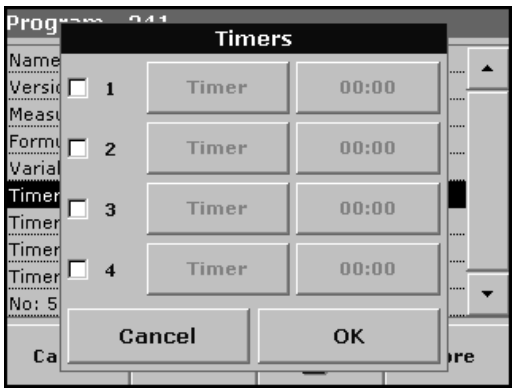
F1: Factor 1

F2: Factor 2

λ1: Wavelength 1

U1: Conversion Factor 1 for the first chemical form

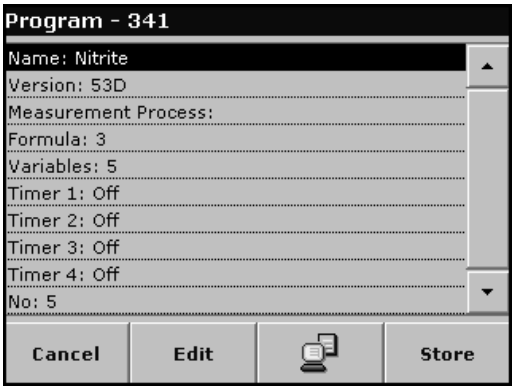
U2: Conversion Factor 2 for additional chemical forms etc.



Timer 1, Timer 2, Timer 3 and Timer 4:
The timers are activated or deactivated with the control boxes on the left of the display. In the next column, a selection can be made from a list of names that describe the corresponding work step. In the third column, the times for each active timer are entered.

5.5.6.2 Update an existing barcode test

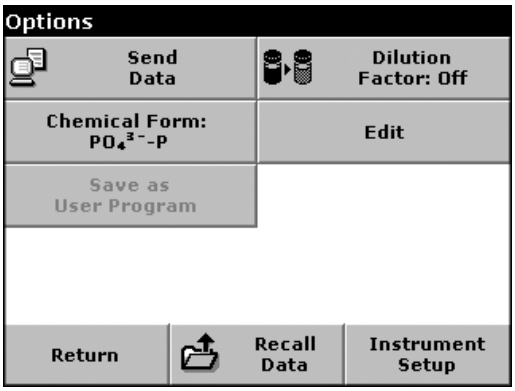
Note: *Only in very few cases does the revision of a test require all test specifications to be updated.*



Option 1: Manual update of test data

1. Insert the light shield in cell compartment #2.
2. The instrument needs to be at the Main Menu or press **Barcode Programs** in the Main Menu.
3. Insert the blank or sample cuvette/vial (depending on the working procedure) in cell compartment #1.
4. After "Barcode controlnumber?", a data overview is displayed, including the specifications of the test that is to be revised.

   Note: *The working procedures of the corresponding test contains the new data (Wavelength, Factors, Measurement Ranges, Conversion Factor for chemical Form, etc.).*
5. Highlight the corresponding line and press **Edit**.
6. When finished programming, press **Store** and then **Cancel** to return to the User Programs menu.
7. Press **Start to** begin the test with the new data.

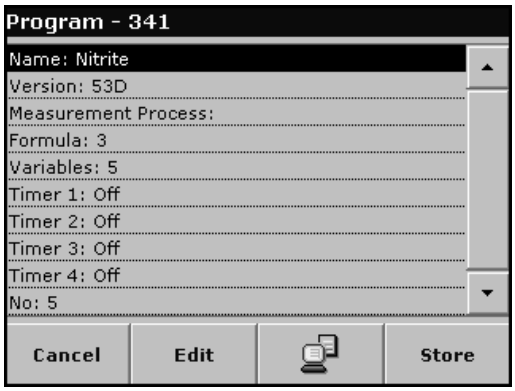


Option 2: Manual check/revision of test data
If an incorrect entry was made during the data update and is not recognized via the barcode of the cuvette/vial (e.g. an incorrect measuring range), another option is available for checking and if necessary revising, the test data.

1. Insert the light shield in cell compartment #2.
2. The instrument need to be at the Main Menu or press **Barcode Programs** in the Main Menu.
3. Insert the blank or sample cuvette/vial (depending on the working procedure) in cell compartment #1.
4. Press **Options>More>Edit**.
5. A data overview is displayed, including the specifications of the test that is to be revised. Compare the displayed data with the data in the working procedure and edit the displayed data in accordance with the working procedure.

5.5.6.3 Program a new test

1. The instrument needs to be at the Main Menu or press **Barcode Programs** in the Main Menu.
2. Insert the sample cuvette/vial or blank (depending on the working procedure) into the cell compartment.



3. After "Program not available", a data overview is displayed, including the specifications of the test that is to be revised.

   The working procedures of the corresponding test contains the new data (Wavelength, Factors, Measurement Ranges, Conversion Factor for chemical Form etc.).
4. Highlight the corresponding line including the specifications of the test that is to be programmed and press **Edit**.
5. To finish programming, press **Store** and then **Cancel** to return to the Main Menu.
6. Press **Start** to begin the test with the new data.

5.5.7 Upgrade of the instrument software

To obtain the software for the update from the Internet at **www.hach.com**:

1. Go to **http://www.hach.com**.
2. On the DR 2800 product page, click **Lab System Software**/**Software Update Downloads** under **Downloads**.
3. Locate the appropriate download and follow the prompts for saving the file(s) to the USB memory stick.
4. On the DR 2800, press **Instrument Update** in the System Checks menu.
5. Connect the USB stick to the USB interface on the DR 2800 (section 3.4 on page 13). Press **OK**.The link is established automatically and the software is updated.

6. Press **OK** to return to the System Checks menu.

Note: *When the instrument software has been updated, a prompt to restart the instrument is displayed.*

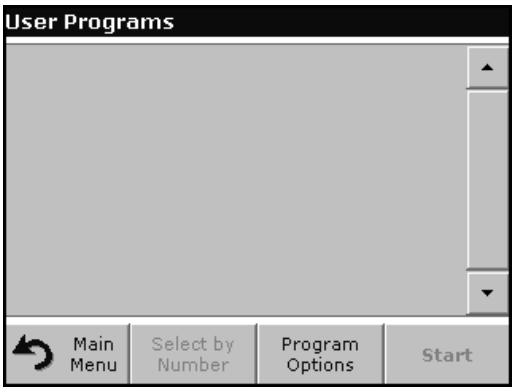
6.1 User Programs

User programs provide the opportunity to complete "made to measure" analysis.

The User Programs database is empty when the instrument leaves the factory and is used to accommodate programs created by users specific needs. Here are a few examples of entries:

- Programming of user-created procedures. The analysis procedure must be developed first, before it can be programmed. The user must define or determine the program sequences, calculation formulas, measurement wavelengths, factors, measuring range limits, etc.
- Modified tests
- Assignment of user programs to the favorites menu for frequently used tests.
- Creation of a specific selection of methods and tests.

Press **User Programs** in the Main Menu and then **Program Options**. The **Program Options** menu contains several input and editing options (Table 7):



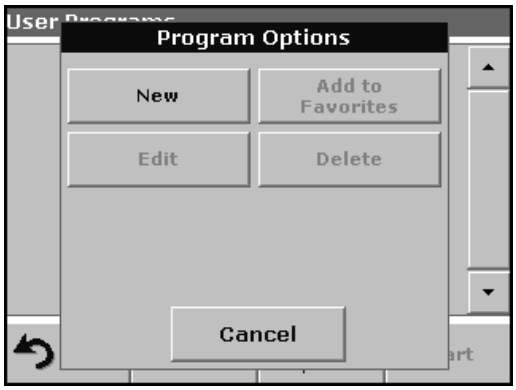


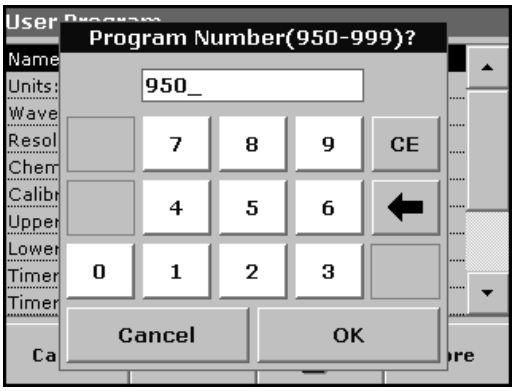
Table 7 Program options user programs

Options	Description
New	Select New to program a new user program. ***Note:*** *The first time* ***Program Options*** *is selected, only the* ***New*** *option is available. The other options remain inactive (grey) until the first program has been created.*
Add to favorites	Select **Add to favorites** to add an existing user program to the list of frequently used programs.
Edit	Select **Edit** to modify an existing program
Delete	Select **Delete** to remove a program from the list of user programs. The program will be simultaneously deleted from the Favorites list.

6.1.1 Program a user method

All input steps and their significance and options are explained in the following sections.

1. Select **New** in the Program Options menu.

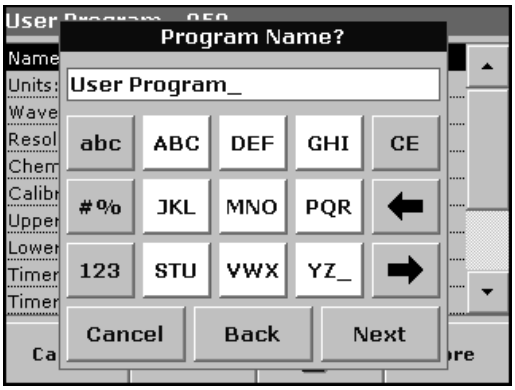


Program Number:
Specific test number, with which the program can subsequently be called up from the selection list in the **User Programs** menu or the **Favorites** menu.

2. Use the alphanumeric keypad to enter a program number between 950 and 999. The lowest available number appears automatically.

3. Press **OK**.

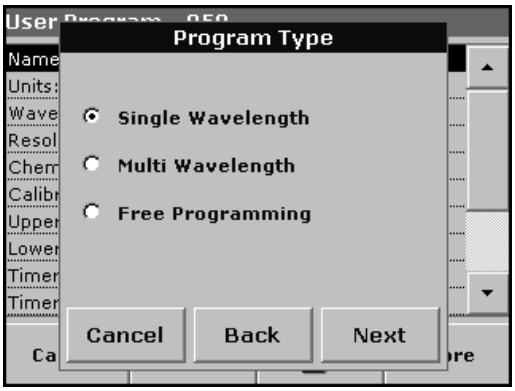
Note: *If the program number is already assigned to another user program, a message appears, asking whether the existing program should be replaced. Press* ***OK*** *to overwrite the existing program.*



Program Name:

4. Use the alphanumeric keypad to enter a program name. The name can be a maximum of 28 characters long.

5. Press **Back** to go back to the previous program point or press **Next** to continue with the input of the program data.



Program Type:

6. Select the required option (Table 8) and press **Next**.

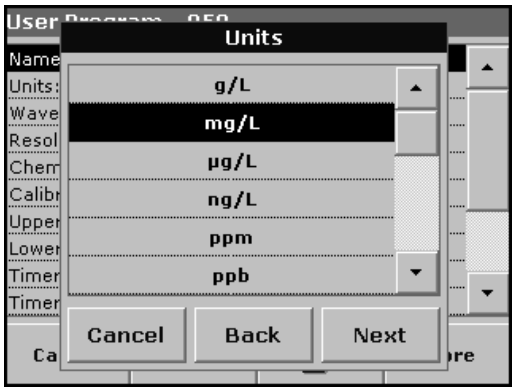
7. If the Single Wavelength (section 6.1.1.1 on page 63) or Multi Wavelength (section 6.1.1.2 on page 64) is selected, define the unit, wavelength, absorbance formula, wavelength λx, concentration factor Kx, concentration resolution, chemical form and calibration equation.
More information on Free Programming parameters see section 6.1.2 on page 72.

Table 8 Program descriptions

Program Type	Description
Single Wavelength	Measurements at a defined wavelength
Multi Wavelength	In the Multi Wavelength mode, absorbance values can be measured at up to four wavelengths and the results can be mathematically processed to obtain sums, differences and relationships.
Free Programming	This is an advanced form of programming for original user-developed methods. In Free Programming, the user defines the measurement process, variables and calculations involved in obtaining a reading.

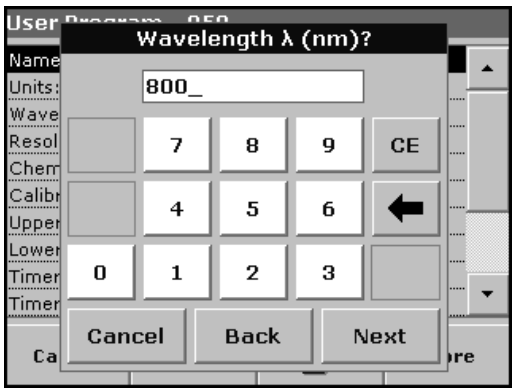
6.1.1.1 Single wavelength settings

If the Single Wavelength mode is selected, the following parameters can be defined:



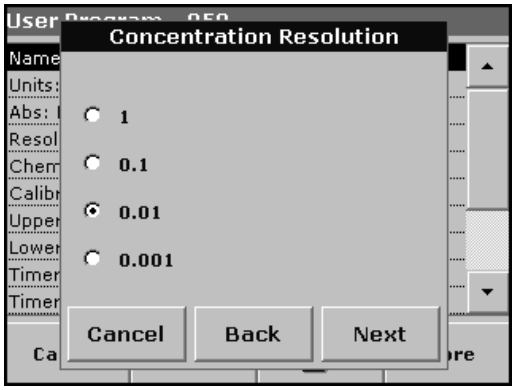
Units:
Select the required unit from the list and press **Next**.

Note: *Units of measure not included in this list can be added in the edit program under* ***Program Options***, ***Edit***. *Select* ***Units***, ***Edit*** *and then* ***New***.

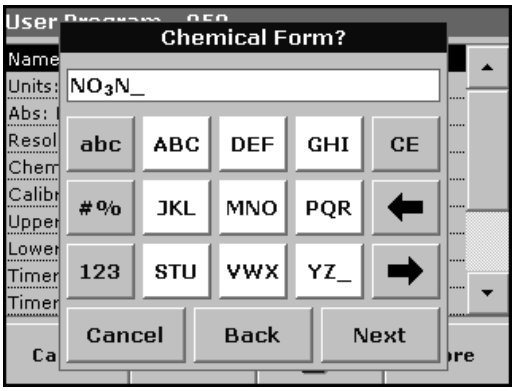


Wavelength (single wavelength program type):
Use the alphanumeric keypad to enter the measurement wavelength. The entered wavelength must be in the range from 340–900 nm.

Press **Next** to proceed.



Concentration resolution (number of decimal places)
Select the required number of decimal places from the displayed list and press **Next**.

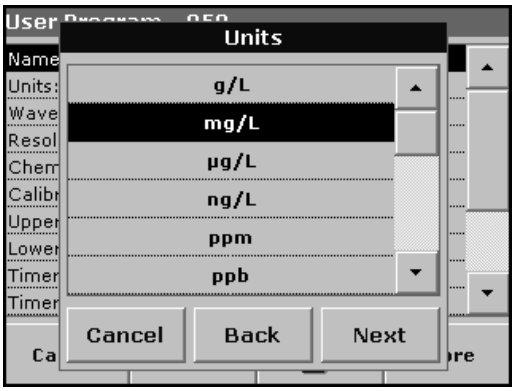


Chemical form:
Enter the chemical formula used in the display to represent the analysis parameter.

Use the alphanumeric keypad to enter the chemical form and press **Next** to enter Calibration settings.

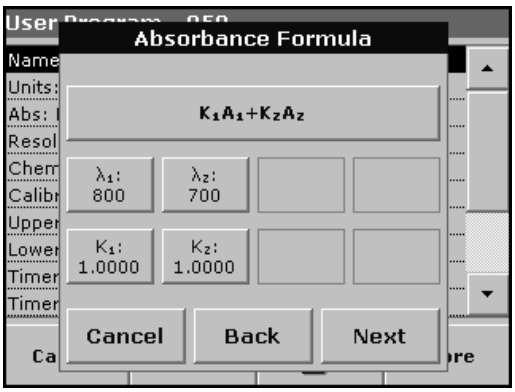
6.1.1.2 Multi wavelength settings

If the Multi Wavelength mode is selected, the following parameters can be defined:



Units:
Select the required unit from the list and press **Next**.

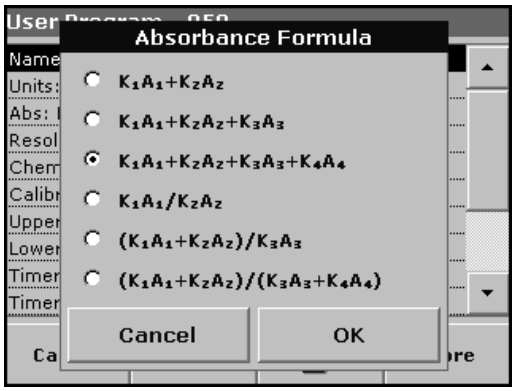
Note: *Units of measure not included in this list can be added in the edit program under* ***Program Options***, ***Edit***. *Select* ***Units***, ***Edit*** *and then* ***New***.



Absorbance formula (multi-wavelength program type):
The Absorbance Formula menu is used to define the wavelengths and the coefficients used in the formula. The absorbance formula defines the calculation for the multi-wavelength measurement. Press the appropriate key to edit the input.

Press the **Formula key**.

In the displayed list, select the formula for the program and press **OK** to confirm.



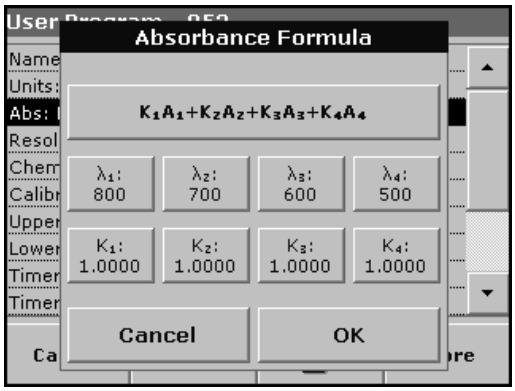
List of available absorbance formulas
A_1 is the absorbance at wavelength 1,

A_2 is the absorbance at wavelength 2 and so on

K_1 is the factor at wavelength 1,

K_2 is the factor at wavelength 2 and so on

If a subtraction has to be completed, the factors can be entered with a minus sign.

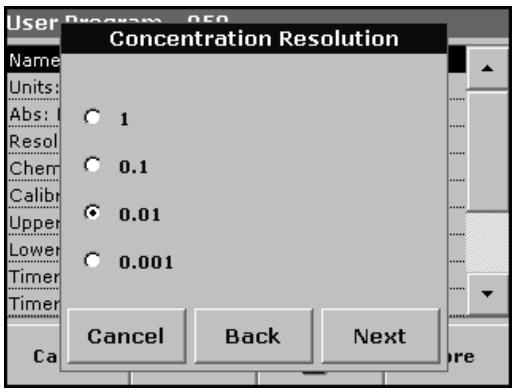


Wavelength λ_x:
Press a λ_x key and use the alphanumeric keypad to enter a wavelength. Press another λ_x key and enter the next wavelength. If necessary, repeat until all the wavelengths for the formula have been entered. The wavelengths must be in the range from 340–900 nm. Press **OK**.

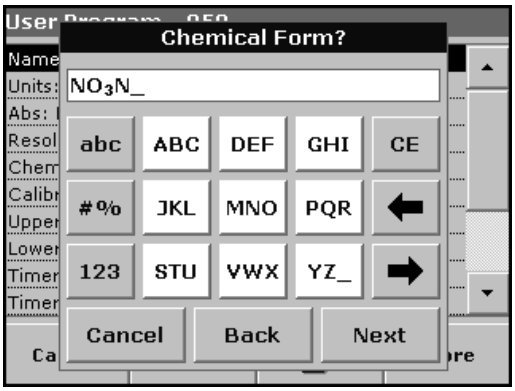
Concentration Factor Kx
Multiplication factor for converting absorbance values into concentration values.

Press a factor key and use the alphanumeric keypad to enter a factor. If the formula includes more than one factor, press another factor key and enter another factor and repeat until all the factors have been entered. Press **OK**. When all the relevant data have been entered, press **Next**.

Note: *Up to 5 digits can be entered, including a maximum of 4 decimal places to the right of the decimal.*



Concentration resolution (number of decimal places)
Select the required number of decimal places from the displayed list and press **Next**.



Chemical form:
Enter the chemical formula used in the display to represent the analysis parameter.

Use the alphanumeric keypad to enter the chemical form and press **Next** to enter Calibration settings.

6.1.1.3 Calibration settings for single and multi wavelength mode

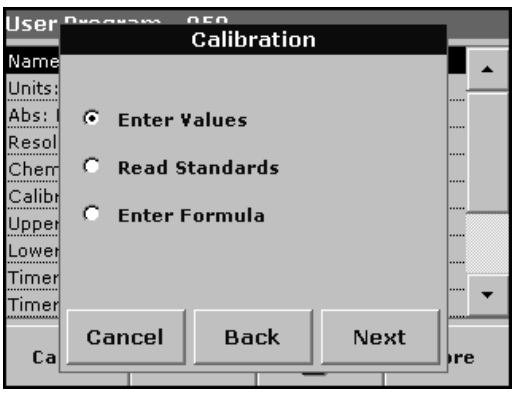
A method is calibrated by determining the absorbance values of several standard solutions of known concentration.

There are three ways to create and store a calibration curve. Instructions for each method follows (Table 9).

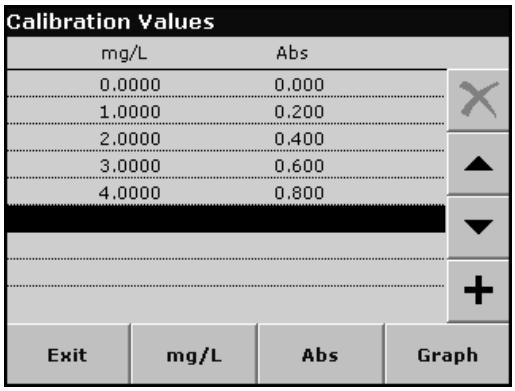
Table 9 Calibration settings

Mode	Descriptions
Enter values	A calibration table is created by entering the concentration values and the absorbance values of the analyte solution. The absorbance values are plotted versus standard concentrations and the calibration curve is displayed as a graph (page 66).
Read Standards	A calibration table is created by entering the concentration values of the standard solutions and then measuring the absorbance of the analyte solutions. The absorbance values are plotted versus standard concentrations and the calibration curve is displayed as a graph (page 68).
Enter Formula	If the calibration curve can be determined from the mathematical relationship between concentration and absorbance by linear regression, etc., the corresponding formula can be selected (linear, 2nd or 3rd order polynomial) from a list and the appropriate factors can be entered (page 69).

Calibration by entering calibration values
Enter concentration/absorbance



1. Select the **Enter Values** and press **Next**.

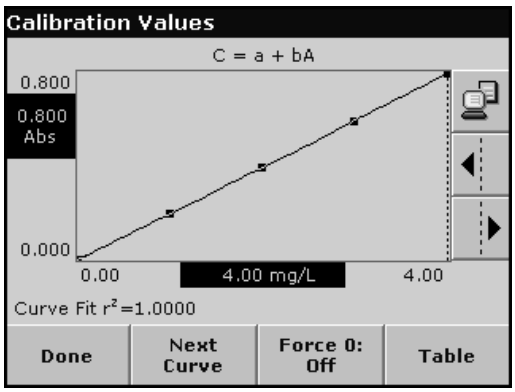


2. To enter the standard concentrations and corresponding absorbance values in the displayed table, press the "**+**" symbol. Use the alphanumeric keypad to enter the values.

Press **OK** and enter the corresponding absorbance value. Press **OK**.

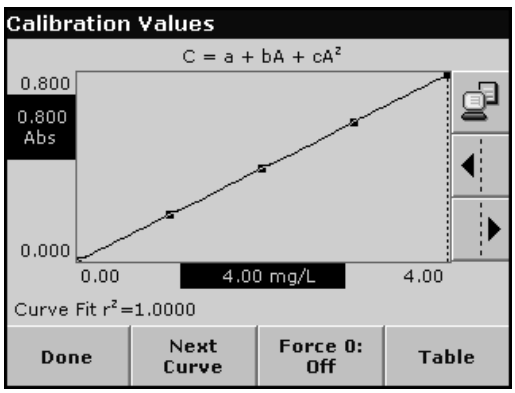
The entered data are displayed in the table. Repeat the sequence for each data point to enter it.

3. To change a value in the table, highlight the appropriate line, press the unit key (e.g. **mg/L**) or **Abs** and enter the changed value via the alphanumeric keypad.



4. When the data have all been entered, press **Graph**.

Note: *The correlation coefficient (r^2) is shown on the left below the axes.*



5. The linear equation corresponds to the standard setting. Press **Next Curve** to display the polynomial 2nd order curve. Press **Next Curve** again, to display the polynomial 3rd order curve.

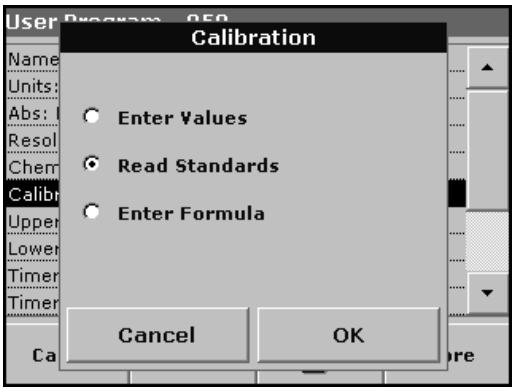
6. Press **Force 0** to change the setting from **Off** to **On**. The curve now passes through the origin of the coordinate system.

Note: *This may have an adverse effect on the correlation coefficient (r^2).*

7. Press **Table** to display the table again.

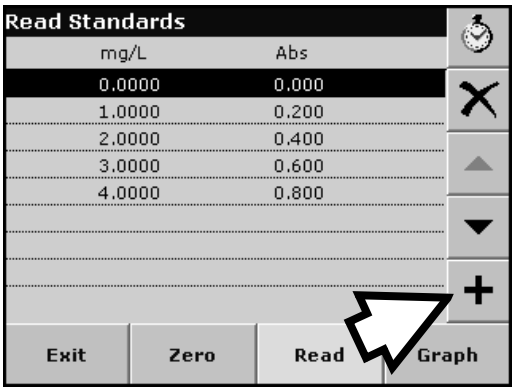
8. When the table has been completed and the curve type has been chosen, press **Done** when the graph is displayed or Exit when the table is displayed. Go to section 6.1.1.4 on page 70.

Calibration by reading standards



1. Press **Read Standards** and press **Next**.

2. To enter the standard concentrations in the displayed table, press the "**+**" symbol. Use the alphanumeric keypad to enter the standard concentration. Press **OK**.



3. Press the "+" symbol again (see arrow) and enter the next standard concentration. Repeat this sequence until all standard conentrations (maximum of 24 solutions) have been entered.

4. Highlight the line with the appropriate concentration and insert the cuvette with the corresponding standard solution.

5. Insert the zero solution into the cell compartment. Press **Zero**.

6. Insert the **first** standard solution into the cell compartment. Press **Read**.

   Insert the **second** standard solution into the cell compartment. Press **Read**.

   Repeat this sequence until all the standard solutions have been measured (maximum of 24 solutions).

The entered and measured data are displayed in the table.

Note: *To delete a standard concentration, highlight the appropriate line and press the* ***Delete*** *icon.*

The timer icon shown in the display helps to ensure, when necessary, that the steps of an analysis are correctly timed (e.g. reaction times, wait times, etc., can be exactly specified). When the specified time has elapsed, an acoustic signal is emitted. The use of the timer has no influence on the measurement program.

7. When the data have all been entered and the measurements have all been completed, press **Graph**.

8. The linear curve corresponds to the standard setting. Press **Next Curve** to display the polynomial 2nd order curve.Press **Next Curve** again to display the polynomial 3rd order curve.

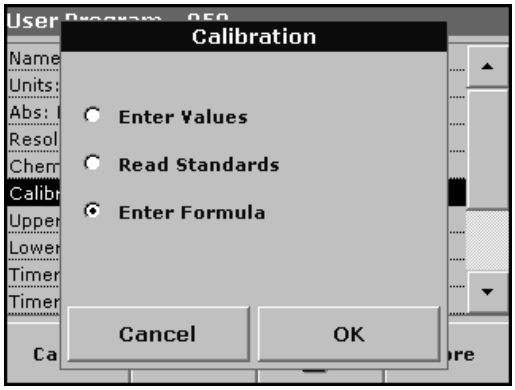
9. Press **Force 0** to change the setting from **Off** to **On**. The curve then passes through the origin of the coordinate system.

   Note: *This may have an adverse effect on the correlation coefficient (r^2).*

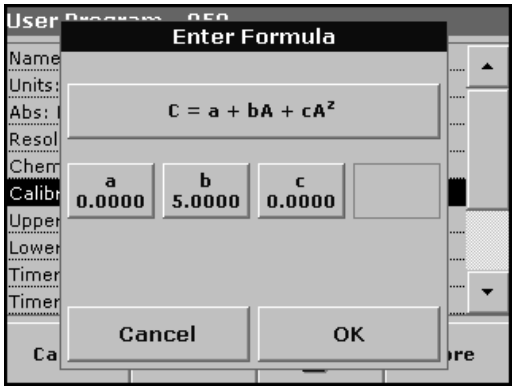
10. Press **Table** to display the table again.

11. When the table has been completed and the curve type has been chosen, press **Done** when the graph is displayed or **Exit** when the table is displayed. Go to section 6.1.1.4 on page 70.

Calibration by entering the formula



1. Press **Enter Formula** and press **Next**.



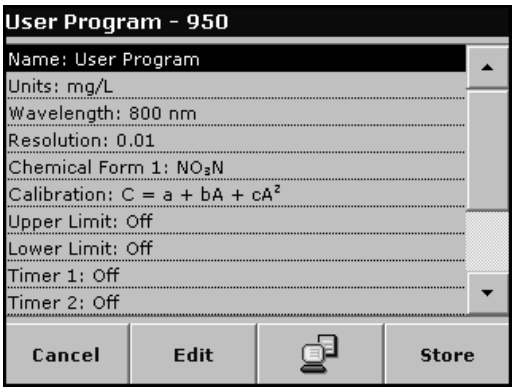
2. Press the formula key.

   A list of available formulas (linear and 2nd and 3rd order polynomial) is displayed. Up to 4 coefficients can be entered, depending on the selected formula. Press the required formula.

3. Depending on the selected formula, the required coefficients (a, b, c...) are displayed. Press the coefficient keys and enter the corresponding values via the alphanumeric keypad. After each entry, press **OK** to confirm.

   Note: *The coefficients can have 5 digits and can have a positive or a negative sign.*

6.1.1.4 Store a user program



The input of the basic data is complete. An overview of the variable program data is displayed.

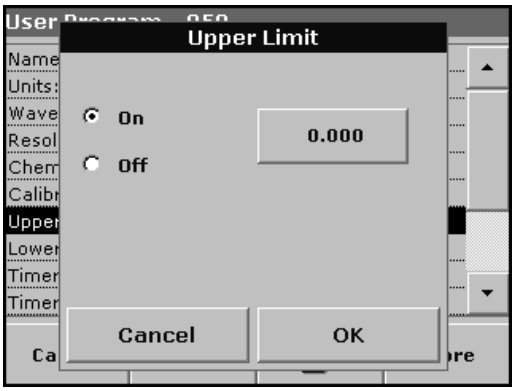
1. To enter more specifications or change existing ones, highlight the appropriate line and press **Edit**.
2. Select **Store** to save the user program.
3. Press the **PC & Printer** icon to send the program data to a printer or to a USB memory stick (connect the USB memory stick to the USB interface first).

   Note: *The test data will be formatted in the preinstalled PrgData folder as a* ***.csv*** *and as a* ***.lst*** *file.*
4. To transfer the program data from one instrument to another create a new folder on the USB memory stick named **dbhlc** and copy the **.lst** files into this folder. (see section 6.7.2 on page 98).

6.1.1.5 Additional user-defined parameters and functions

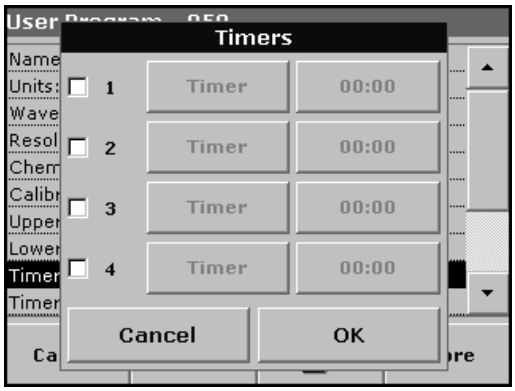
In addition to the previously defined basic data, additional parameters and functions can be defined for user programs:

- measuring range upper and lower limits
- timer functions
- chemical forms



Upper and lower limits of the measuring range
It is possible to enter a maximum (upper) and minimum (lower) measurement value. An error message is displayed if a reading is above the upper limit or below the lower limit.

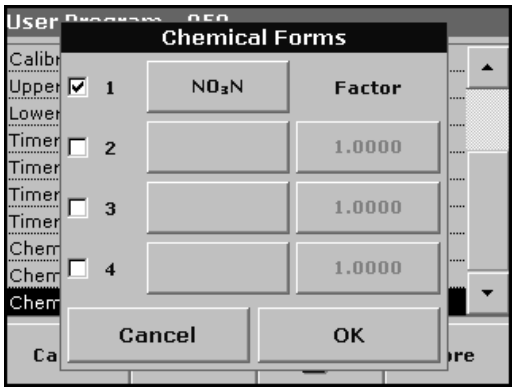
1. Highlight the appropriate line in the overview of the program data and press **Edit**.
2. Set **On** and press the **0.000** key to enter the measuring range limit. Confirm the entry by pressing **OK**.



Timer 1 / Timer 2 / Timer 3 / Timer 4:
This function can define time intervals for up to four timers. Timer designations such as Shake, Wait and Swirl can be assigned.

1. Highlight the appropriate line in the overview of the program data and press **Edit**.

2. The timers are activated or deactivated with the check boxes in the left part of the display. In the next column, a selection can be made from a list of names that designate the corresponding work step. In the third column, the times for each timer are entered (in mm:ss).



Chemical form 2 / chemical form 3 / chemical form 4:
If a **Chemical Form 1** has been defined, up to three additional alternative forms can be entered here.

1. Highlight the appropriate line in the overview of the program data and press **Edit**.

2. The chemical forms are activated or deactivated with the check boxes in the left part of the display.

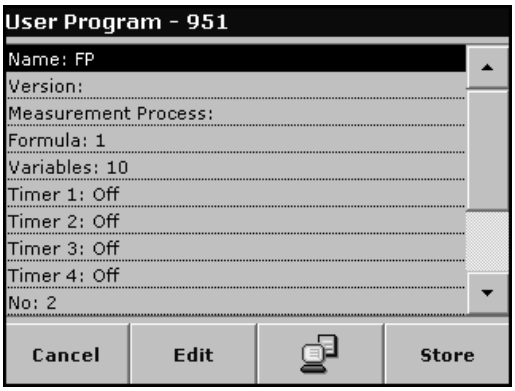
3. Press the left key to enter another chemical form with the alphanumeric keypad and press **OK** to confirm. Press the right key to enter the conversion factor to calculate the concentration of the additional chemical form from the concentration of Chemical Form 1 and press **OK** to confirm.

4. Press Store to save the program data. Press **Cancel** to return to the Main Menu.

6.1.2 Free programming program type

Free Programming is an advanced option for entering original user-developed methods. When the Free Programming option is selected, an overview of the specifications of the programmed test is displayed. Each input option can be modified to develop the user method. Refer to Table 10 for more information. To modify an input option, select the appropriate line and press **Edit**.

Important Note: *Perform steps 1–6 of Programming a New User Method (section 6.1.1 on page 61) before proceeding with Free Programming.*



Highlight the line containing the program point that is to be edited or defined and press **Edit**.

Table 10 Definitions of the program points

Program point	Description
Name	Name of the analysis parameter
Version	An abbreviation or version number assigned by the user is entered here.
Measurement Process	Exact definition of the test: the number of wavelengths at which measurements are made, the number of absorbance measurements needed, the keys to be used, any waiting periods between measurements, etc.
Formula	Definition of the formulas with which the test result is calculated.
Variables	The number of variables shown in the display depends on the definition of the measurement process and the formulas. Input of the numerical values of the wavelengths, factors, constants, etc.
Timer 1, Timer 2, Timer 3, Timer 4	Used to enter abbreviations and defined times for up to four timers. Highlight the appropriate line and press **Edit**. The timers are activated or deactivated with the control boxes on the left of the display. In the next column, a selection can be made from a list of names that describe the corresponding work step. In the third column, the times for each active timer are entered.

6.1.2.1 Measurement process

The measurement process defines the handling and the measurements of the test:

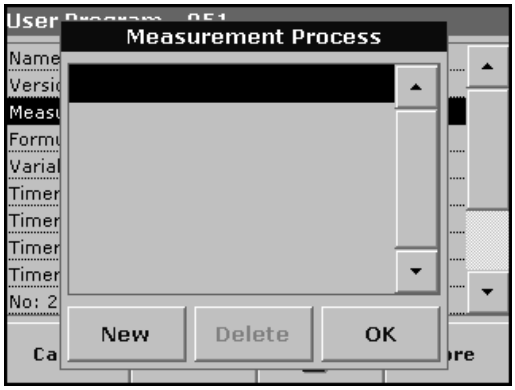
- At which and how many wavelengths should measurements be completed
- How many absorbance measurements must be completed?
- When should the zero measurement and the sample measurement be completed?
- Are waiting times necessary between measurements?

- Should individual program sequences be repeated?

The elements of a measuring sequence, such as zero and sample measurements and the timer(s) (reaction times, waiting times, etc.) are individually defined.

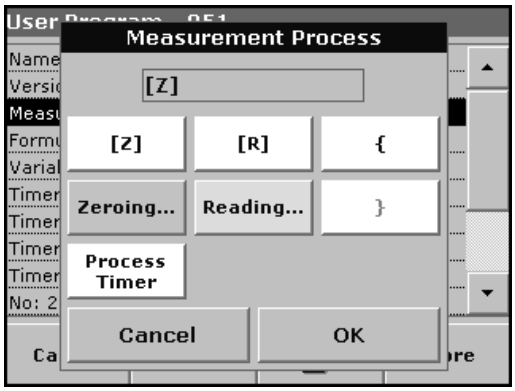
6.1.2.2 Enter a new element of a measuring sequence

Important Note: *Each component of the measurement process* ***must*** *be entered in the order in which it will be completed.*



1. Highlight the **Measurement Process** line in the data overview and press **Edit**.
2. Press **Edit** again and then **New**.

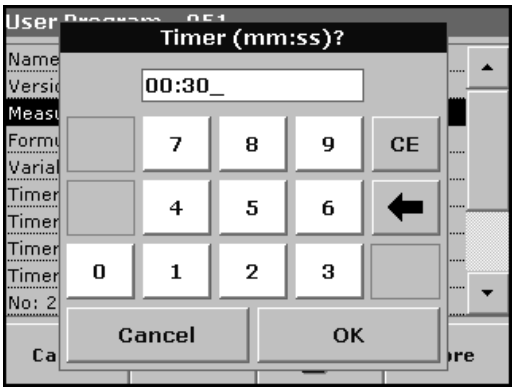
Content and definition of the keys



[Z] key / **Zeroing**

1. Press the **[Z]** key to program a zero measurement. Confirm with **OK**.
2. Press **New** and then **Zeroing...** and use the alphanumeric keypad to enter the wavelength at which the zero measurement is to be completed. Press **OK** and confirm the input by pressing **OK** again.
3. If zero measurements are to be carried out at a number of wavelengths, repeat the above two steps for each wavelength.

Note: *The entered measurement sequence is displayed.*

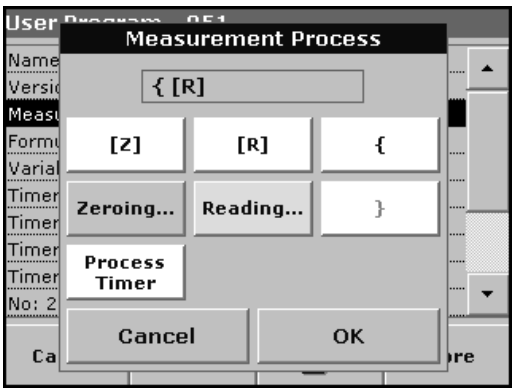


Process Timer key

1. Press the **Process Timer** key to enter any waiting, reaction or handling times that have to be taken into account. Use the alphanumeric keypad to enter the time. Press **OK** and confirm the input by pressing **OK** again.

   Note: *This time is integrated into the measurement process.*

   Note: *The entered measurement sequence is displayed.*



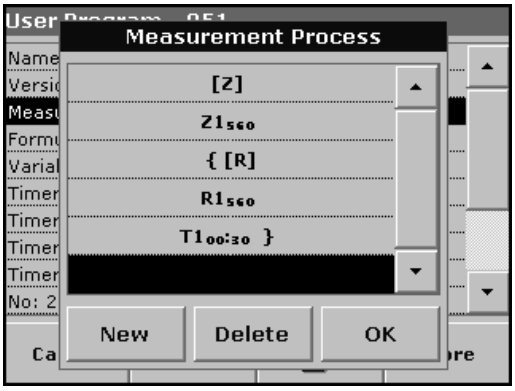
[R] key **/ Reading...**

1. Press the **[R]** key to program a measurement of the substance that is to be analyzed. Confirm with **OK**.

2. Press **New** and then **Reading...** and use the alphanumeric keypad to enter the wavelength at which the measurement is to be completed. Press **OK** and confirm the input by pressing **OK** again.

3. If measurements are to be completed at a number of wavelengths, repeat the above two steps for each wavelength.

   Note: *The entered measurement sequence is displayed.*



{ } key
Elements of the measurement sequence that are to be repeated are placed in brackets.

The left bracket "{"marks the start of the sequence that is to be repeated and the right bracket "}" marks the end.

Note: *The key showing the right bracket remains inactive until a left bracket is entered.*

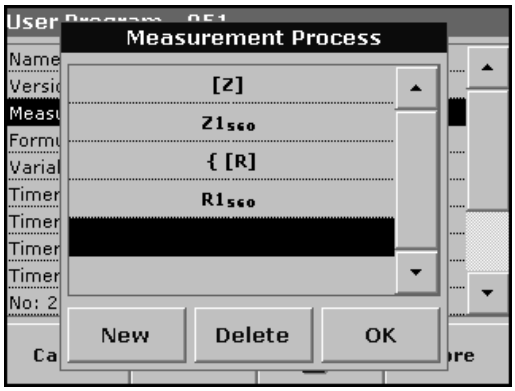
1. Press **{**.

2. Press the key that defines the sequence that is to be repeated: **[Z]** or **[R]**. Confirm with **OK**.

3. Press **New** and then press **Zeroing...** or **Reading...** and use the alphanumeric keypad to enter the wavelength at which the measurement should be completed. Press **OK** and confirm the input by pressing **OK** again.

4. Press **}** to end the sequence.

   Note: *If an action such as a zero measurement recurs at different stages of a measurement sequence, the series of actions is numbered sequentially (e.g. Z1, Z2, etc.).*



Deleting an element of a measuring sequence
Select the appropriate line and press **Delete**. The element is deleted.

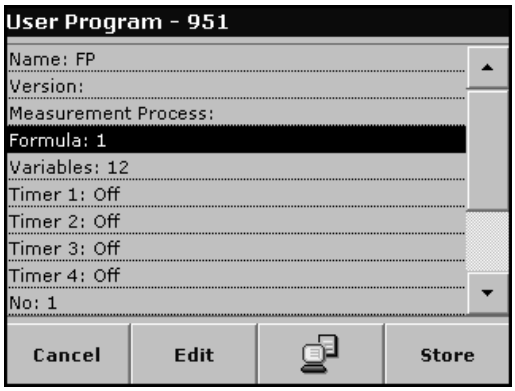
Inserting an element of a measuring sequence
Select the line in the measuring sequence where the insertion is be made and press **New**.

A new element can be entered at the selected position.

When the input is complete, press **OK** in the "Measurement Process" display. The data overview is then displayed.

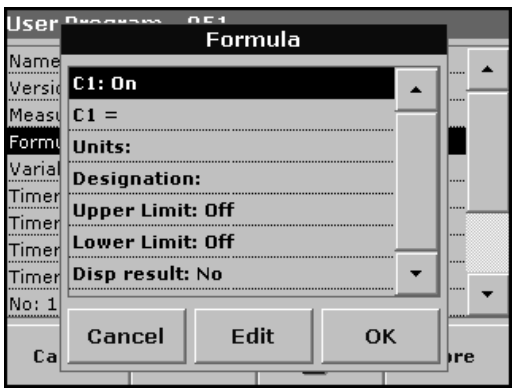
6.1.2.3 Enter the calibration formula (evaluation formula)

The calibration formula (evaluation formula) defines the calculation and display of intermediate and final results. The previously defined elements of the measuring sequence are the basis for calculating the concentrations.

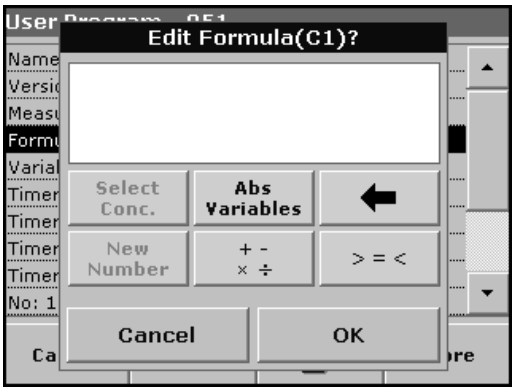


Enter calibration formula C1

1. Highlight the **Formula** line in the data overview and press **Edit**.



2. Highlight the line **C1: Off** and press **Edit**.
3. Select **C1: Off** again and press **Edit**. The display changes to **C1: On**.
4. Highlight the next line **C1 =** to define the formula and press **Edit**.



Refer to Table 11 for detailed information on the Edit formula keys.

Note: *The evaluation formula is built up successively in the display in accordance with the input.*

Note: *The* ***arrow*** *key deletes the most recently entered element of the formula.*

Table 11 Edit formula key descriptions

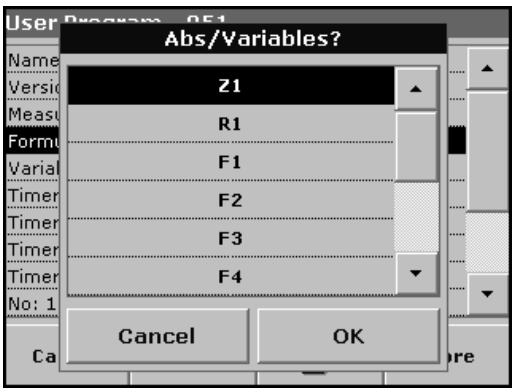
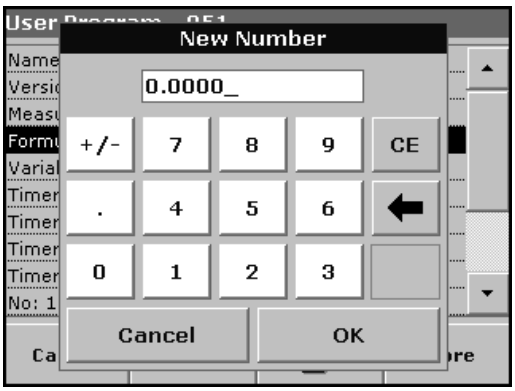
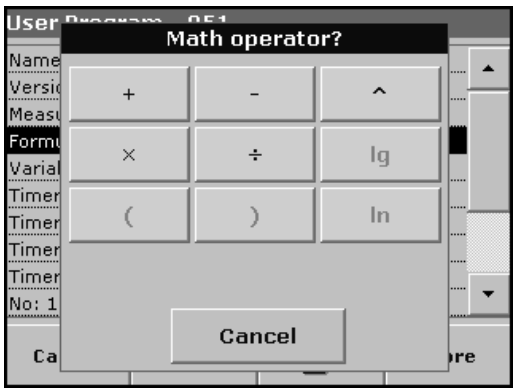
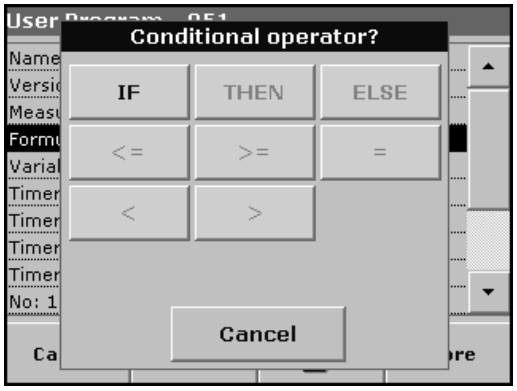
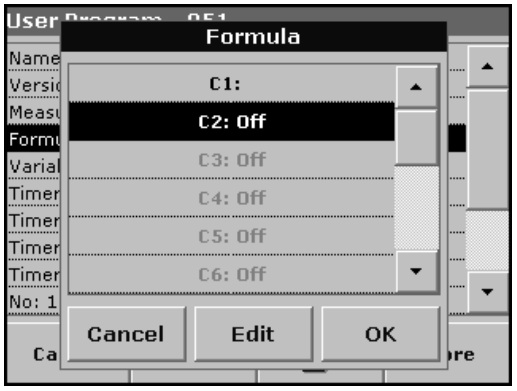
Screen	Key	Description
	**Abs/Variables**	Press the **Abs/Variables** key to select, from the displayed list, the required element of the defined measuring sequence and therefore the corresponding measurement wavelength, so that this can be taken into account in the formula.
	**New Number**	Press **New Number** to enter a new factor or constant.

Table 11 Edit formula key descriptions (continued)

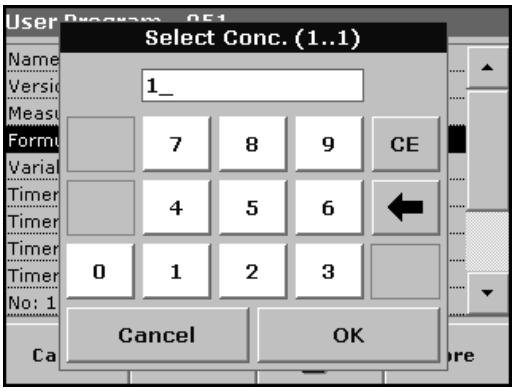
Screen	Key	Description
	**+– ÷x**	Press **+– ÷x**, to enter a mathematical operation. Select the operation and press **OK** to confirm. The available choice of mathematical operations depends on the defined formula. This means that functions such as "**( )**" or "**ln**"/"**log**" etc. are only active if a term in parentheses or the calculation of a logarithm is mathematically permissible in the defined formula (this also applies to the basic mathematical operations). The following mathematical operations are available: • + (Addition) • – (Subtraction) • ÷ (Division) • x (Multiplication) • ^ (Exponent) • Ln (Natural logarithm) • Log (Common logarithm)
	**>=<**	Press >=< to include logic statements/links/conditions in the formula. The following functions are available: = (Equal to), < (Less than), > (Greater than), <= (Less than or equal to), >= (Greater than or equal to), IF, THEN, ELSE When the evaluation formula C1 has been entered completely, press **OK** to confirm. Press **OK** again to return to the Formula display. When the formula C1 has been entered and confirmed, the parameter name, upper and lower limits of the measuring range and display result (yes, no) can be entered.



Enter the next calibration formula (C2 or Cn)

1. Highlight **C2: Off** and press **Edit**.
2. Select **C2: Off** again and press **Edit**. The display switches to **C2: On**.
3. Highlight the next line **C2 =** to define the formula and press **Edit**.

In addition to the keys described in Table 11, only one other function is available:



Select Conc. key
If an already defined formula, in this case C1, is to be taken into account in the formula for C2, press **Select conc.**

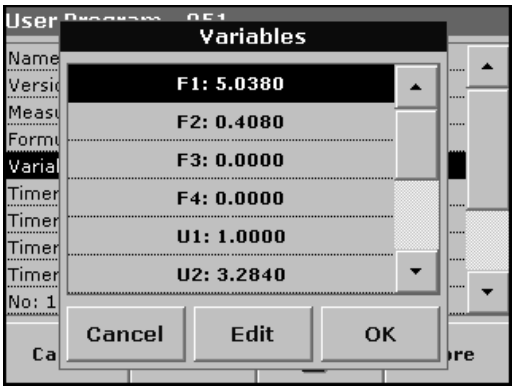
Enter the number of the formula (e.g. 1 for C1) and press OK to confirm.

Cn can now be linked with a mathematical operation.

Note: *The Cn concentrations that are to be calculated are numbered in sequence: C1, C2, C3, etc.*

Note: *When the first Cn formula has been defined, the Formula list is automatically extended by Cn+1.*

6.1.2.4 Enter variables



1. Highlight the **Variables** line in the data overview and press **Edit**.

2. Select the variable to be edited, press **Edit** and use the alphanumeric keypad to enter the data specified in the working procedure (for F1, F2, λ1, U1 etc.). Press **OK** to confirm each entry.

Abbreviation of Variables:

F1: Factor 1

F2: Factor 2

λ1: Wavelength 1

U1: Conversion Factor 1 for the first chemical form

U2: Conversion Factor 2 for the further chemical form etc.

6.1.2.5 Save a free programming user program

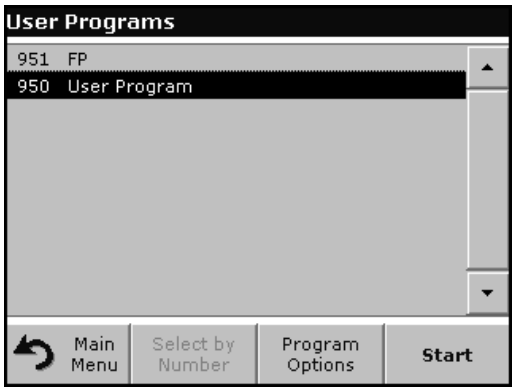
1. Press **Store** to save the entered data. The data can be stored under any data point (Measurement sequence, Formula, Timer, etc.).

2. Press the **PC & Printer** icon to send the program data to a printer or to a USB memory stick (connect the USB memory stick to the USB interface first).

   Note: *The test data will be formatted in the preinstalled PrgData folder as a* ***.csv*** *and as a* ***.lst*** *file.*

3. To transfer the program data from one instrument to another create a new folder on the USB memory stick named **dbhlc** and copy the **.lst** files into this folder (see section 6.7.2 on page 98).

6.1.3 Select a user program



1. Press **User Programs** in the Main Menu to view an alphabetical list of user programs with program numbers.

   The User Programs list will appear.

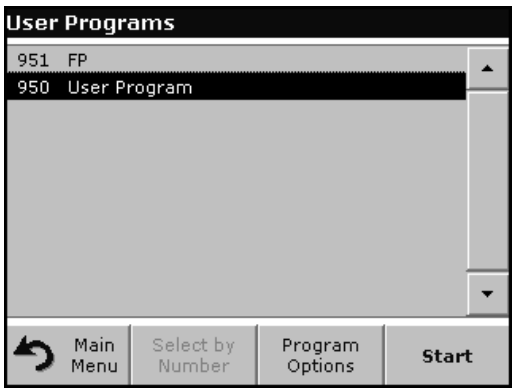
   Note: *Use the scroll bar to scroll through the list quickly.*

   Note: *Highlight the selection by pressing it or press* ***Select by Number*** *to search for the program by number.*

2. Press **Start** to run the program.

6.1.4 Add, edit and delete user programs from the favorites list

The most frequently used tests/methods in the User Program menu can also be added to the list of favorites to simplify their selection.

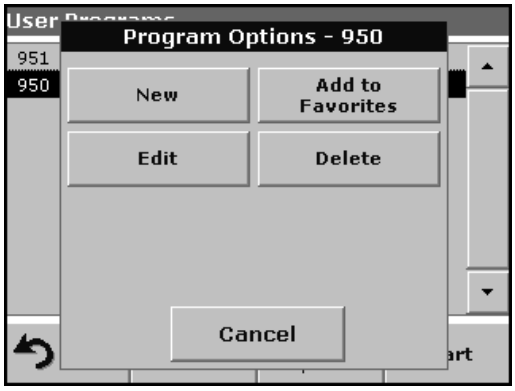


1. Press **User Programs** in the Main Menu.

   The User Programs list will appear.

2. Highlight the selection by pressing it or press **Select by Number** to search for the program by number.

   Note: *Use the scroll bar to scroll through the list quickly. Use the alphanumeric keypad to enter the test number (program number) and press OK to confirm.*



3. Press **Program Options**.

4. Press **Add to Favorites**, **Edit** or **Delete** and press **OK** to confirm.

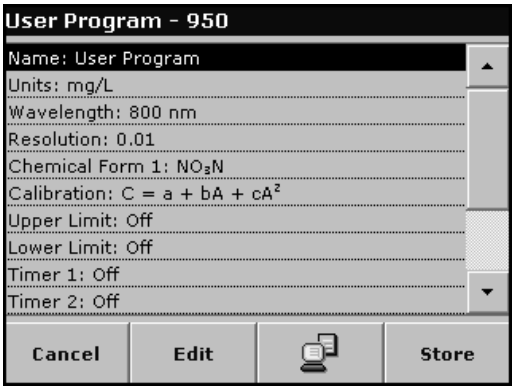
   Note: *If the stored program is deleted in User Programs, it will also be deleted in Favorites Programs.*

6.1.4.1 Add to Favorites

1. Press **Add to Favorites** and press **OK** to confirm.

   The program is added to the Favorites.

6.1.4.2 Edit



1. Press **Edit** and press **OK** to confirm.

   An overview of the specifications of the programmed test is displayed. More information about the input options is provided in the section 6.1.2 on page 72.

6.1.4.3 Delete

1. Press **Delete** and press **OK** to confirm.

   The program is deleted from the list of User Programs.

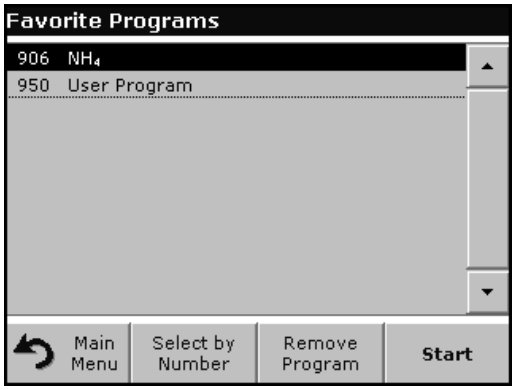
   Note: *If the stored program is deleted in User Programs, it will also be deleted in Favorites Programs.*

6.2 Favorite Programs

The most frequently used tests/methods in the **Stored Programs** menu and the **User Programs** menu can also be added to the list of favorites to simplify their selection.

To add **Stored Programs** and/or **User Programs** to the favorites list or the favorite programs, see section 6.1.4 on page 79.

6.2.1 Recall a favorite program



1. Press **Favorite Programs** in the Main Menu.

   The Favorite Programs list will appear.

2. Highlight the selection by pressing it.

   Note: *Use the scroll bar to scroll through the list quickly.*

   Note: *Press **Select by Number** to search for the program by number. Use the alphanumeric keypad to enter the test number (program number) and confirm the input by pressing **OK**.*

3. Press **Start**.

6.2.2 Delete a favorite program

1. Press **Favorite Programs** in the Main Menu.

   The Favorite Programs list will appear.

2. Highlight the selection by pressing it.

   Note: *Use the scroll bar to scroll through the list quickly.*

   Note: *Press **Select by Number** to search for the program by number. Use the alphanumeric keypad to enter the test number (program number) and confirm the input by pressing **OK**.*

3. Press **Remove Program** and press **OK** to confirm.

   Note: *If a **Favorite Programs** is deleted, it will stay in the **User Programs** or **Stored Programs**.*

   Note: *If the stored program is deleted in User Programs, it will also be deleted in Favorites Programs.*

6.3 Standard Addition – monitoring/checking results

The accuracy of measured values (their correspondence with the actual concentration of the analyte in the sample) and their precision (correspondence of the measurement results obtained from several samples containing the same concentration of the test analyte) can be determined or improved using the standard addition method.

This method (also referred to as spiking) serves to identify sample-specific interference factors, e.g. substances in the sample that falsify the analysis (sample matrix effect), a defective measuring instrument or contaminated reagents.

Method:
A defined amount (concentration) of a standard solution of the test substance is added to the sample. The detection rate should be close to 100%.

$$\text{Detection rate} = \frac{\text{Measured value after a standard addition}}{\text{Expected value after a standard addition}}$$

<table>
<tr><th>Detection rate</th><th colspan="2">Conclusion</th></tr>
<tr><td>100%</td><td colspan="2">Probability that the measurement results are correct is high.</td></tr>
<tr><td rowspan="4">< 100%</td><td colspan="2">Assumption: The analysis was falsified by substances in the sample (sample matrix effect)
Test to determine whether a sample matrix effect is present:
Use distilled water instead of the sample. Add standard solution as described in the procedure.</td></tr>
<tr><td>Detection rate</td><td>Conclusion</td></tr>
<tr><td>100%</td><td>Ions in the sample are interfering with the analysis, causing false results to be obtained.</td></tr>
<tr><td>≠ 100%</td><td>No interfering ions - consider other interference factors.</td></tr>
</table>

Measures to identify other interference factors:

Checklist:

1. Check if the procedure is completed correctly:
   - **a.** Are the reagents added in the correct order?
   - **b.** Is enough time allowed for color development?
   - **c.** Is the correct glassware in use?
   - **d.** Is the glassware clean?

e. Does the test require the sample to be at a certain temperature?

f. Was the pH of the sample in the correct range?

g. Is the pipette volume correct?

2. Check the used reagents by repeating the standard addition procedure with freshly prepared reagents.

<table>
<tr><th>Detection rate</th><th colspan="2">Conclusion</th></tr>
<tr><td rowspan="3">100%</td><td colspan="2">The originally used reagents were defective.
Check the standard solution:
Repeat the standard addition procedure with a freshly prepared standard solution.</td></tr>
<tr><td>Detection rate</td><td>Conclusion</td></tr>
<tr><td>100%</td><td>The originally used standard solution was defective.</td></tr>
</table>

If none of these measures resolves the problem, please contact the manufacturer or a sales representative.

6.3.1 Complete a standard addition

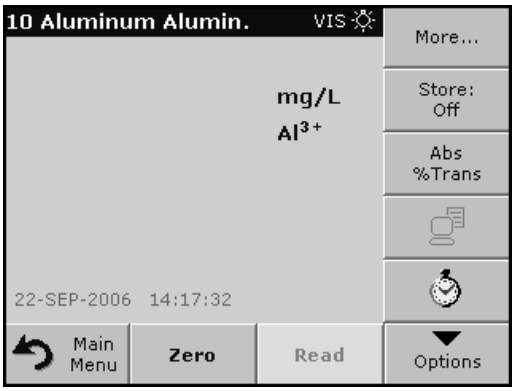
Carry out the standard addition in accordance with the corresponding procedure.

There are two different methods:

Peak volume (Standard addition):
Defined volumes of a standard solution are added step by step to an already analyzed sample. The sample is measured again after each addition.

Sample volume:
A defined volume of a standard solution of known concentration is added to the volume of sample specified in the procedure and the sample is measured after each addition. In most cases three different standard solutions are prepared and the procedure is repeated for each of them.

Note: *The units and chemical forms used for the sample are used for the standard solutions. Be careful to ensure that to use the correct units for subsequent entries.*

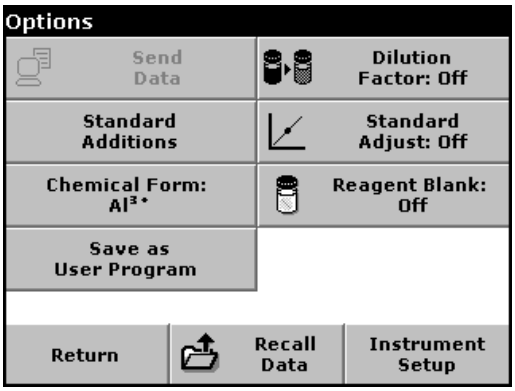


Peak volume/sample volume methods

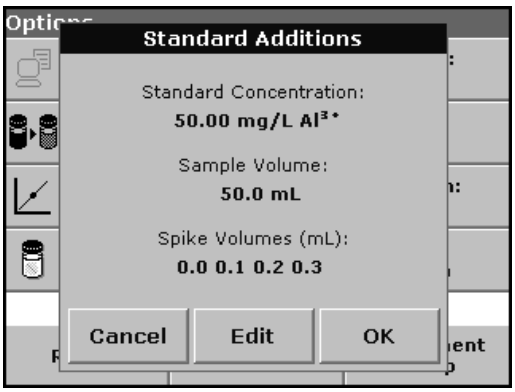
1. Select **Stored Programs** in the main menu. Select the required program.

2. Press **Start**.

3. Analyze a sample without added standard solution in accordance with the instructions in the Procedures Manual. When the measurement is complete, leave the sample cuvette/cell in the cuvette/sample cell holder.

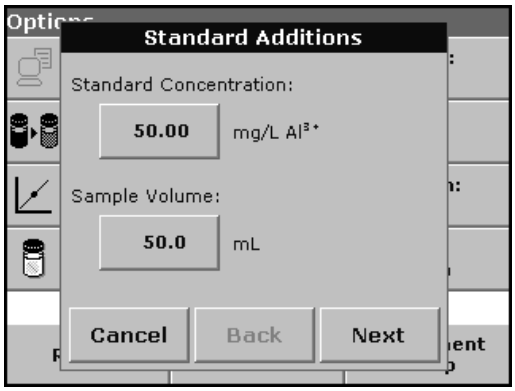


4. Press **Options>More>Standard Addition**.

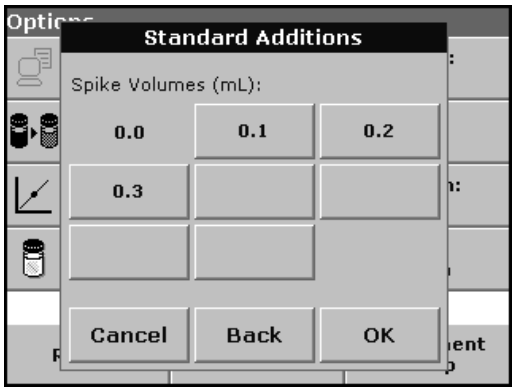


An overview of the data of the standard addition procedure is displayed.

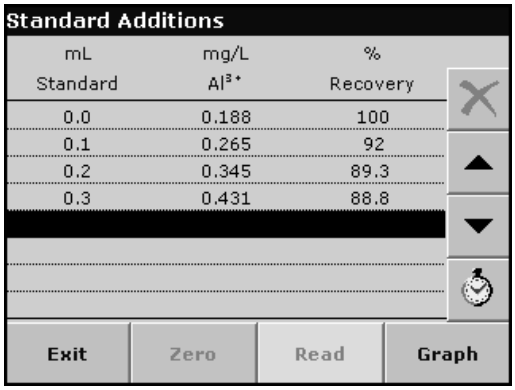
5. Press **OK** to accept the standard values for standard concentration, sample volume (total) and standard addition volume. Press **Edit** to change any of these values.



6. Press the key of the value to change it. Use the alphanumeric keypad to change the value. Press **OK** to confirm.



7. Press the keys to enter the standard addition volumes. Use the alphanumeric keypad to enter the new data and press **OK**.



Description of the table of readings

- The **first** column shows the standard addition volume. 0 mL stands for a sample containing no added standard.
- The **second** column shows the reading of the samples with and without added standard.
- The **third** column shows the detection rate of the added standard.

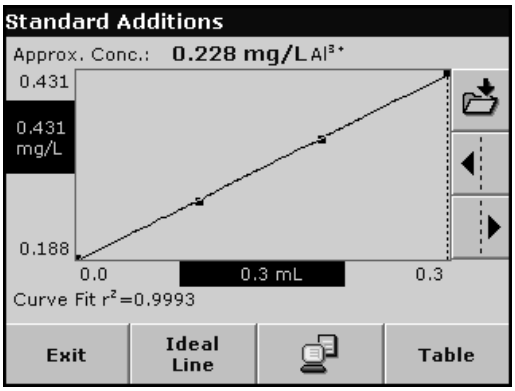
Note: *The black highlighted line is active.*

8. The reading of the sample in the instrument, without added standard, is automatically shown under 0 mL.

9. Prepare the standard addition solution as described in the procedure.

10. Use the arrow keys to select the first standard addition volume in the table and insert the cuvette with the corresponding volume of added standard into the cell compartment. Press **Read**.

Repeat the procedure from point 8 with all the other standard addition solutions.



11. After all the standard addition solutions have been measured, press **Graph**.

The regression line through the standard addition data points is displayed.

The correlation coefficient r^2 indicates how close the data points are to the line.

If the correlation coefficient = 1, the curve is linear.

The concentration shown above the curve is the estimated concentration of the sample without the added standard.

Note: *In the curve menu, the name on the* ***Curve*** *key switches to Table. Press* ***Table*** *to display all the data in the table again.*

12. Press **Ideal line** to display the relationship between the added standard solutions and the ideal line (detection rate 100%).

6.4 Single Wavelength (absorbance, concentration and transmittance measurements)

The Single Wavelength mode can be used in three ways. For sample measurements at a single wavelength, the instrument can be programmed to measure the absorbance, % transmittance or concentration of the analyte.

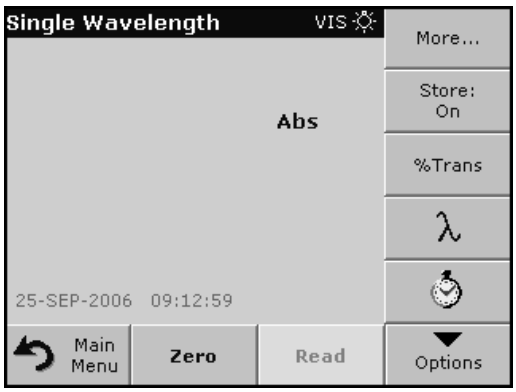
Absorbance measures the amount of light absorbed by the sample, in units of absorbance.

% transmittance measures the percent of the original light that passes through the sample and reaches the detector.

Turn the concentration factor on to select a specific multiplier for converting absorbance readings to concentration. In a graph of concentration versus the absorbance, the concentration factor is the slope of the line.

6.4.1 Set up single wavelength mode

Press **Single Wavelength** in the Main Menu. Press **Options** for Parameter Setup.



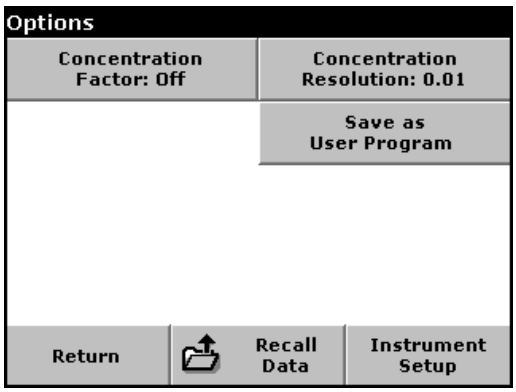
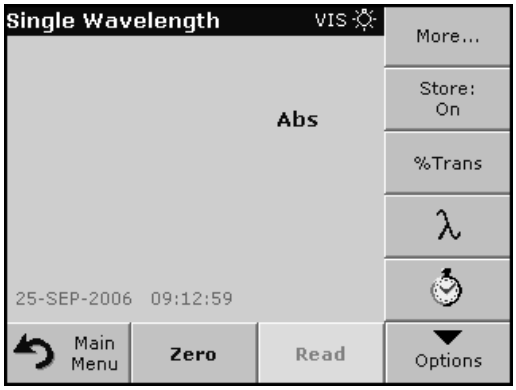


Table 12 Single wavelength setup options

Options	Description
More	For further Options
Store Off/On	With the **Store On** setting, all measurement data are stored automatically. With the **Store Off** setting, no measurement data are stored.
% Trans/Abs	To switch to % transmittance, concentration or absorbance readings
λ **Wavelength**	To enter the measurement wavelength. Use the alphanumeric keypad to enter the measurement wavelength. The entered wavelength must be in the range from 340–900 nm.
Timer icon	This functions as a stopwatch. It helps to ensure that the steps of an analysis are correctly timed (e.g. reaction times, wait times, etc., can be exactly specified). When the specified time has elapsed, an acoustic signal is emitted. The use of the timer has no influence on the measurement program.
Concentration Factor	Multiplication factor for converting absorbance values into concentration values.
Concentration Resolution	To select the position of the decimal point in the calculated concentration readings.
Save as User Program	To store the selected parameters as a User Program, see section 6.1 on page 61
Recall Data	Call up saved measurement data or time courses, see section 5.3 on page 37.



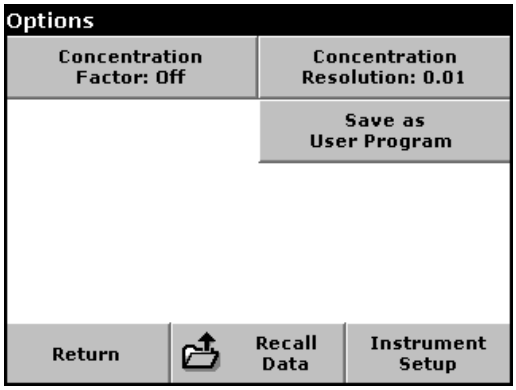
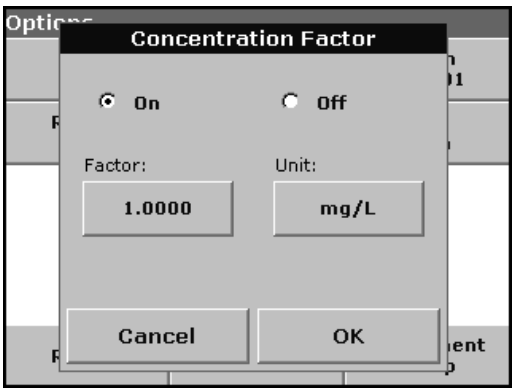


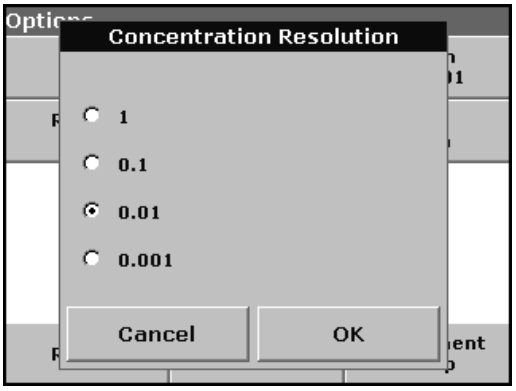
Table 12 Single wavelength setup options (continued)

Options	Description
Instrument Setup	Basic data of the instrument, see section 5.2 on page 27.



Concentration factor:

1. Press **Concentration Factor: Off** in the Options menu. Press **On** to highlight this feature.
2. Press the ”Factor“ key and use the alphanumeric keypad to enter the factor by which absorbance readings are to be multiplied. Press the ”Unit“ key to select the units for concentration measurements or to create a new unit.
3. Press **OK** to confirm.



Concentration resolution:

1. Press **Concentration Resolution** in the Options menu.
2. Select the resolution and press **OK** to confirm.

6.4.2 Take single wavelength measurements

1. Insert the blank cuvette/cell into the cuvette/sample cell holder. Press **Zero**.

   Note: *The **Read** key is only active after the zero measurement has been completed.*

2. Insert the sample cuvette/cell into the cuvette/sample cell holder. Press **Read**.

3. For data storage, see section 5.3.1 on page 37.

6.5 Multi-Wavelength mode – measurements at more than one wavelength

In the multi-wavelength mode, absorbance values can be measured at up to four wavelengths and the results can be mathematically processed to obtain sums, differences and relationships.

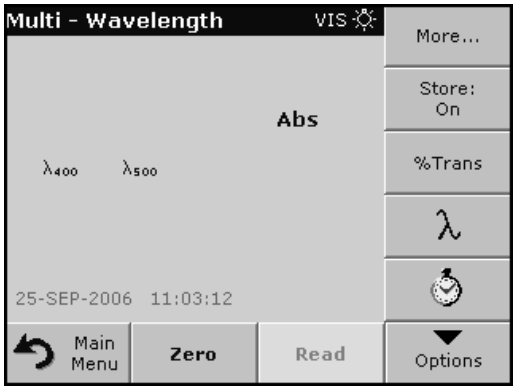
Absorbance measures the amount of light absorbed by the sample, in units of absorbance.

% Transmittance measures the percent of the original light that passes through the sample and reaches the detector.

Turning on the concentration factor allows selection of a specific multiplier for converting absorbance readings to concentration. In a graph of concentration versus the absorbance, the concentration factor is the slope of the line. Concentration is calculated using a single factor for each wavelength, which is input by the user.

6.5.1 Set the reading mode at different wavelengths

Press **Multi Wavelength** in the Main Menu. Press **Options** for Parameter Setup.



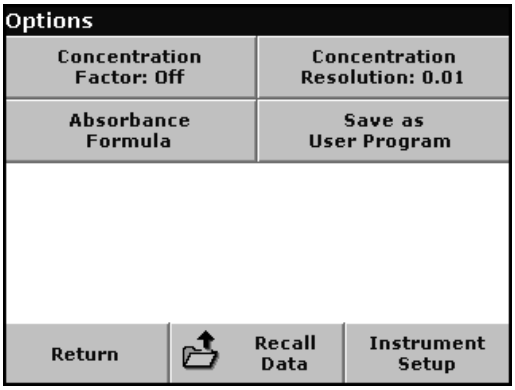
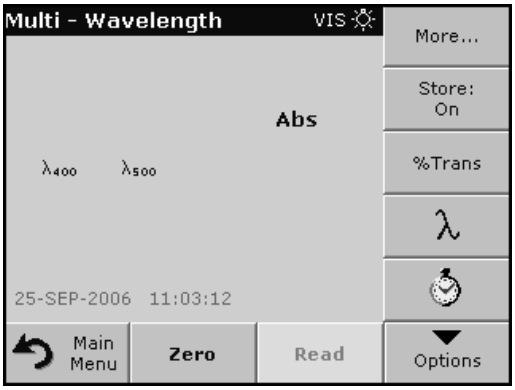


Table 13 Multi-wavelength setup options

Options	Description
More	For further Options
Store Off/On	With the **Store On** setting, all measurement data are stored automatically. With the **Store Off** setting, no measurement data are stored.
% Trans/Abs	To switch to % transmittance, concentration or absorbance readings
λ **Wavelength**	To enter the measurement wavelength. Use the alphanumeric keypad to enter the measurement wavelength. The entered wavelength must be in the range from 340–900 nm.
Timer icon	This functions as a stopwatch. It helps to ensure that the steps of an analysis are correctly timed (e.g. reaction times, wait times, etc., can be exactly specified). When the specified time has elapsed, an acoustic signal is emitted. The use of the timer has no influence on the measurement program.
Concentration Factor	Multiplication factor for converting absorbance values into concentration values.
Concentration Resolution	To select the position of the decimal point in the calculated concentration readings.
Absorbance Formula	Calculation basis for evaluating samples
Save as User Program	To store the selected parameters as a User Program, see section 6.1 on page 61



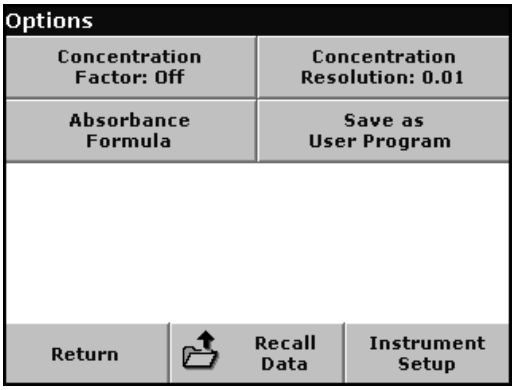
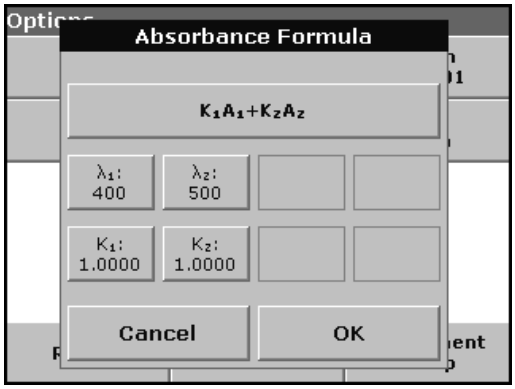


Table 13 Multi-wavelength setup options (continued)

Options	Description
Recall Data	Call up saved measurement data or time courses, see section 5.3 on page 37.
Instrument Setup	Basic data of the instrument, see section 5.2 on page 27.



λ / Absorbance formula:

1. Press **Absorbance Formula**.
2. The formula selected in the top key determines the number of wavelength and coefficent keys that will appear below. To change the absorbance formula, press the top key, select a formula from the displayed list and press **OK**. When a new formula is selected, the number of variables below changes to match.

The following formulas are available:

$K_1 A_1 + K_2 A_2$
$K_1 A_1 + K_2 A_2 + K_3 A_3$
$K_1 A_1 + K_2 A_2 + K_3 A_3 + K_4 A_4$
$K_1 A_1 / K_2 A_2$
$(K_1 A_1 + K_2 A_2) / K_3 A_3$
$(K_1 A_1 + K_2 A_2) / (K_3 A_3 + K_4 A_4)$

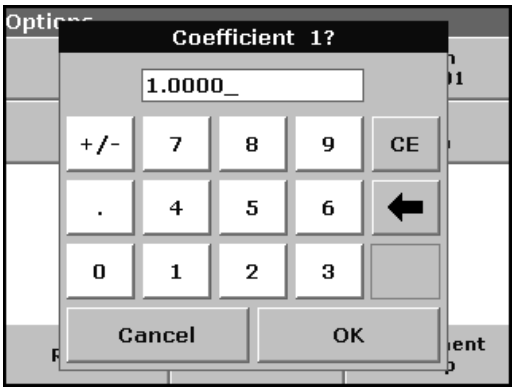
A_1 refers to the absorbance at wavelength 1

A_2 refers to the absorbance at wavelength 2, etc.

K_1 refers to the coefficient at wavelength 1

K_2 refers to the coefficient at wavelength 2, etc.

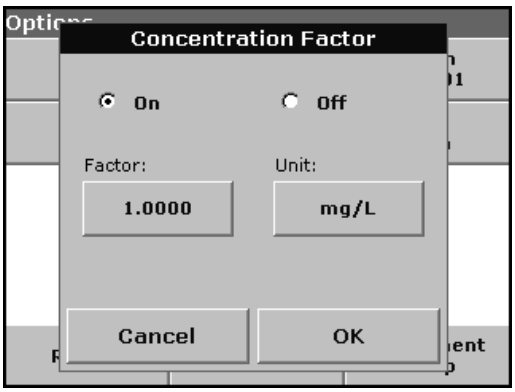
Coefficients can be set negative where subtraction is required.



3. To change a wavelength, press one of the "λx:" keys. Enter the desired wavelength coefficient into the numeric keypad. Press **OK** to confirm.

4. To change a coefficient, press one of the "K_X:" keys. Enter the desired coefficient into the numeric keypad. Press **OK** to confirm.

   Note: *The instrument allows entry of up to 5 significant digits, with a maximum of 4 significant digits after the decimal point.*

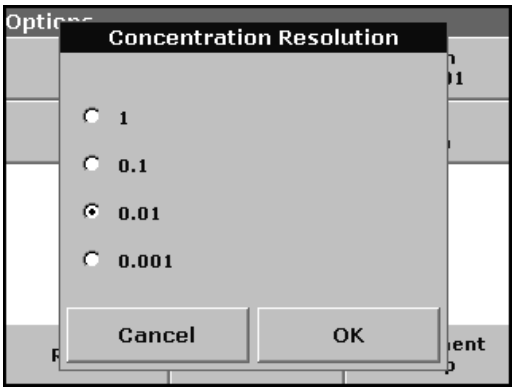


Concentration factor:

1. Press **Concentration Factor: Off** in the Options menu. Press **On** to highlight this feature.

2. Press the "Factor" key to enter the factor by which absorbance readings are to be multiplied. Press the "Unit" key to select the units for concentration measurements or to create a new unit.

3. Press **OK** to confirm.

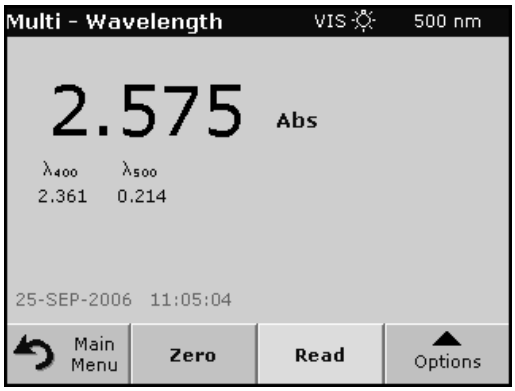


Concentration resolution:

1. Press **Concentration Resolution** in the Options menu.

2. Select the resolution and press **OK** to confirm.

6.5.2 Complete a measurement in the multi wavelength mode



1. Insert the blank cuvette/cell into the cuvette/sample cell holder. Press **Zero**.

   Note: *The **Read** key does not become active until the zero measurement has been completed.*

2. Insert the sample cuvette/cell into the cuvette/sample cell holder. Press **Read**.

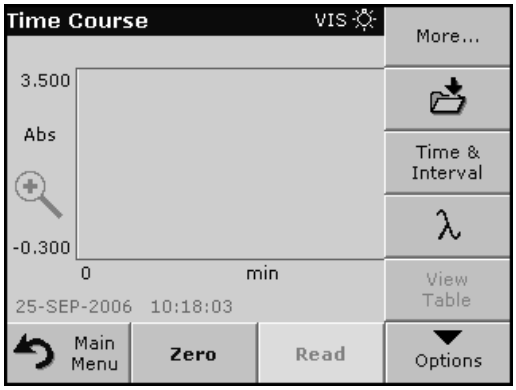
3. For data storage, see section 5.3.1 on page 37.

6.6 Time course of absorbance/transmittance

The Time Course Mode is used to collect data in either absorbance or transmittance for a user-specified length of time. After the data are collected, they can be displayed in either graphic or tabular format.

6.6.1 Time course setup parameters

Press **Time Course** mode in the Main Menu. Press **Options** to configure parameters.



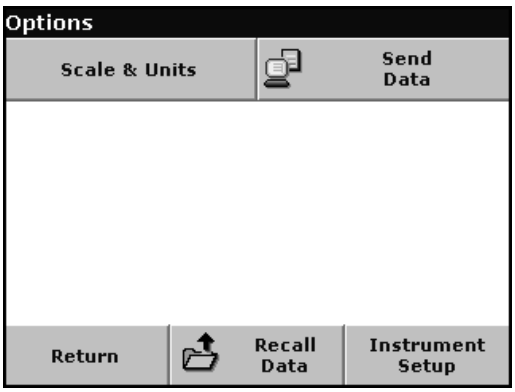
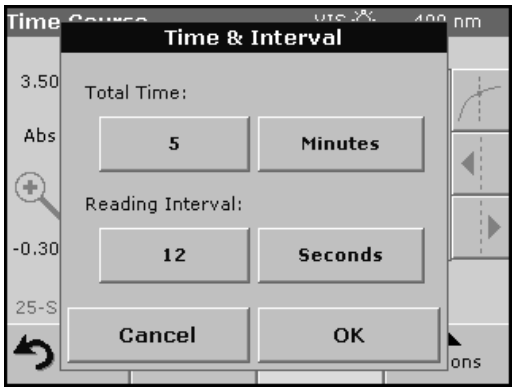


Table 14 Time course setup options

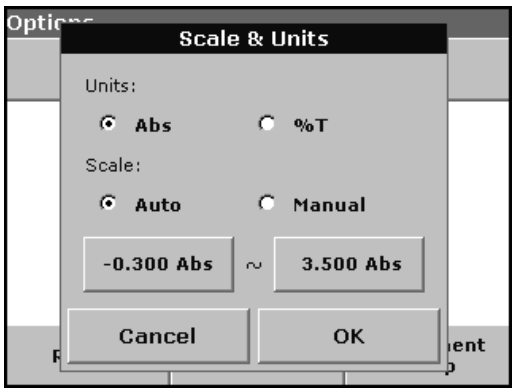
Option	Description
More	For further Options
Store icon	To store the scan data
Time & Interval	To input the total time for data collection and the time interval between the collection of data points
λ	To input the wavelength setting
View Table	To display readings in absorbance, transmittance or concentration. This can be changed after sample data are collected.
Scale & Units	**Scale:** In the automatic scaling mode, the y-axis is automatically adjusted so that the total scan is displayed. The manual scaling mode allows sections of the scan to be displayed. **Units:** Choice of absorbance or transmittance.
Send Data	To send Data to a printer, computer or USB memory stick (Type A)
Recall Data	Call up saved measurement data or time courses, see section 5.3 on page 37.
Instrument Setup	Basic data of the instrument, see section 5.2 on page 27.



Time & interval:

1. Press **Time & Interval** in the Options menu.
2. Input the total time and the reading time and press OK to confirm.

Note: *In total 500 measuring steps are possible. To select a total time and a time interval that would cause this number of measurements to be exceeded, the time interval is defined automatically and the* ***OK*** *key is inactivated.*



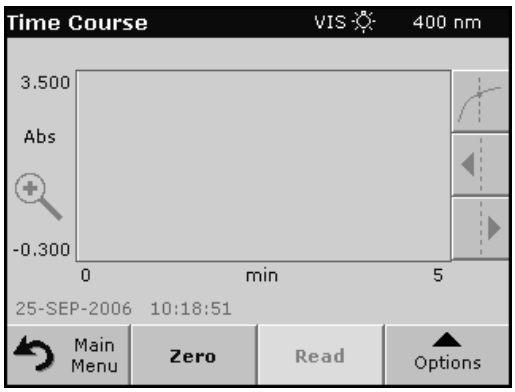
Scale & units:

1. Press **Scale & Units** in the Options menu.
2. Highlight **Abs** or **%T** as the required units.
3. Highlight **Auto** or **Manual** scaling on the graph´s y-axis.

Note: *If manual scaling is selected, use the alphanumeric keypad to set the limits* $y_{min.}$ *and* $y_{max.}$*. The graph is adjusted to display only the values in the selected range. If automatic scaling is selected, the instrument sets the limits automatically so that the total range can be displayed.*

4. Press **OK** to confirm.
5. Press **Return** to return to the scan mode.

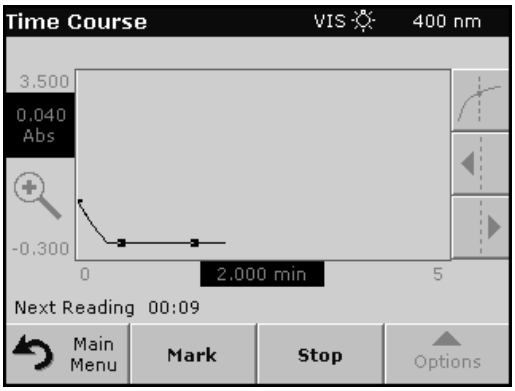
6.6.2 Time course scan reading



After the parameters have been selected, the instrument must be blanked, then the sample can be analyzed.

1. Insert the blank cuvette/cell into the cuvette/sample cell holder. Press **Zero**. The blank reading is shown on the display.
2. Insert the sample cuvette/cell into the cuvette/sample cell holder. Press **Read**. Start collecting time course (kinetic) data.

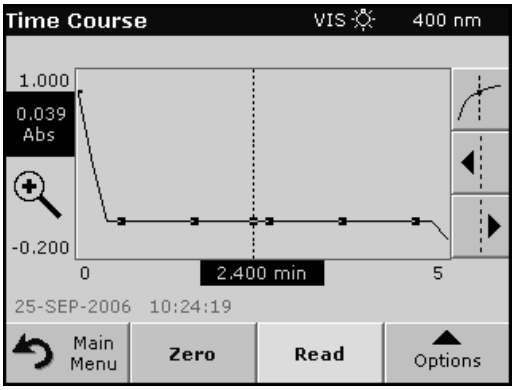
   Note: *During the measurement the* ***Zero*** *and* ***Read*** *keys change to* ***Mark*** *and* ***Stop****.*



- Select **Mark** to mark the next data point collected. This mark is not used by the instrument, but is available for the user and may indicate a significant event, such as the addition of a sample or other reagent. The mark is also shown in the table.
- Select **Stop** to stop taking sample readings.

6.6.3 Analysis of time course data

After the data are collected, the following manipulations can be done on the graphic data:



The **Time Course** Program is complete, if

- the sound is turned on, the instrument beeps when the readings are done
- the graph is shown fullsize,
- the scaling of the x-axis occurs automatically,
- the Cursor functions in the vertical navigation bar are highlighted.

6.6.3.1 Navigation of a time scan or a time scan analysis

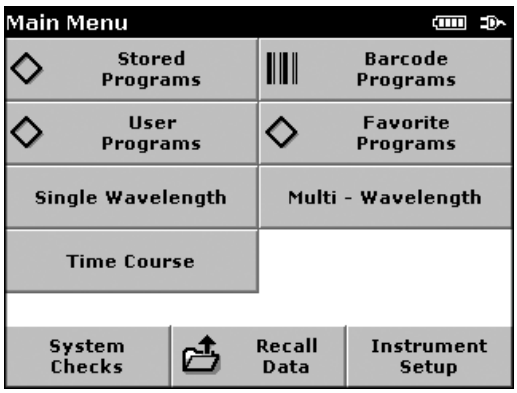
After a time scan has been completed, the time and the absorbance/transmittance data are displayed as a curve.

Where the cursor is positioned on the curve, the elapsed time up to this point and the corresponding absorbance are highlighted.

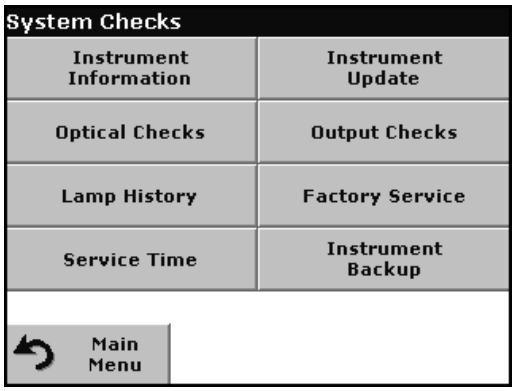
Table 15 Navigating the time scan

Cursor Function/ Zoom Function	Description
Curve Icon (Choice of Cursor Mode)	**Delta mode:** A second cursor is highlighted. The position of the fixed cursor was previously defined in Cursor Mode Single. Use the active cursor to select any point on the measurement curve. The difference to the fixed cursor is shown on the curve. The delta values are correspondingly highlighted and displayed on the x and y axes. The gradient of the curve and the correlation coefficient (r^2) between the cursor points in the Delta mode are shown under the curve.
	Cursor Mode Single: The cursor moves to each selected measurement point of the scan.
Arrow keys	The arrow keys (right/left) are used to move the cursor (depending on the selected mode) to the next data point. The data of the data point (wavelength/absorbance or transmittance value) are highlighted on the x and y axes. ***Note:*** *Press any point on the curve to display the associated data.*
Zoom Icon	This function is used to magnify the section of the curve in the vicinity of the cursor. The original curve size can be restored by pressing the zoom icon again.

6.7 System checks



1. Press **System Checks** in the Main Menu.



The System Checks menu contains instrument information and various performance tests.

6.7.1 Instrument information

1. Press **Instrument Information** in the System Checks menu.
2. The model, serial number and software version are displayed.

6.7.2 Upgrade of the instrument software

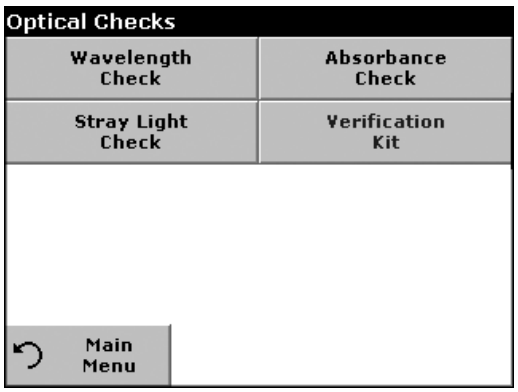
To obtain the software for the update from the Internet at **www.hach.com:**

1. Go to **http://www.hach.com**.
2. On the DR 2800 product page, click **Lab System Software/Software Update Downloads** under **Downloads**.
3. Locate the appropriate download and follow the prompts for saving the file(s) to the USB memory stick.
4. On the DR 2800, press **Instrument Update** in the System Checks menu.
5. Connect the USB stick to the USB interface on the DR 2800 (section 3.4 on page 13). Press **OK**.The link is established automatically and the software is updated.
6. Press **OK** to return to the System Checks menu.

Note: *When the instrument software has been updated, a prompt to restart the instrument is displayed.*

6.7.3 Optical checks

For each optical check, the measured results must be evaluated against user-specific requirements. The check options do not define tolerances.



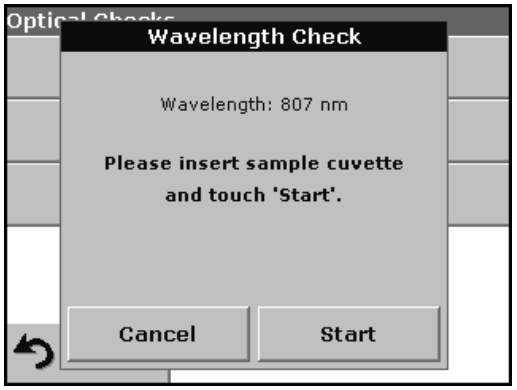
1. Press **Optical Checks** in the System Checks menu.

The Optical Checks menu contains programs for checking the wavelength accuracy, stray light and photometric accuracy.

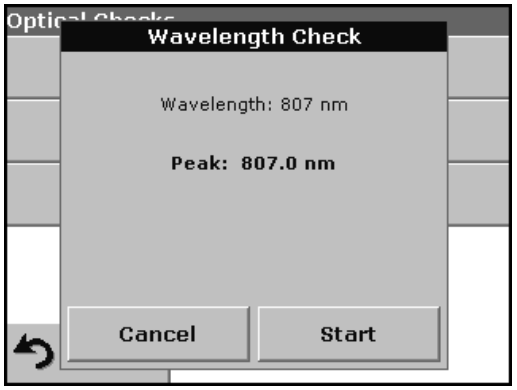
An optional test filter set (Verification Kit) (Section 9 on page 119) containing 6 precision glass filters, target values, tolerances and instructions is available and is recommended as an aid for carrying out comprehensive in-house instrument checks.

6.7.3.1 Wavelength check

The Wavelength Check test is used to check wavelength accuracy at 807 nm.



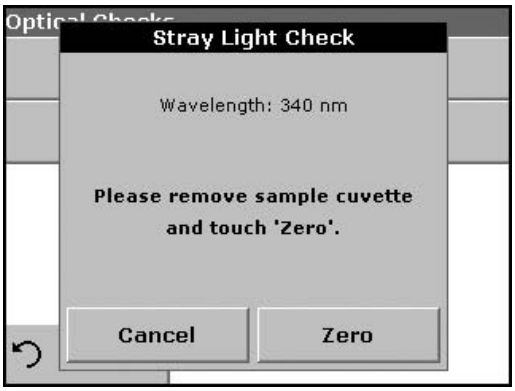
1. Press **Wavelength Check** in the Optical Checks menu.
2. Insert the adapter (A) for 10-mm rectangular cells in the cell compartment #2 and insert the sample cuvette/cell (Neodym or BG20/2) in the adapter. Close the cell compartment.
   Press **Start**.



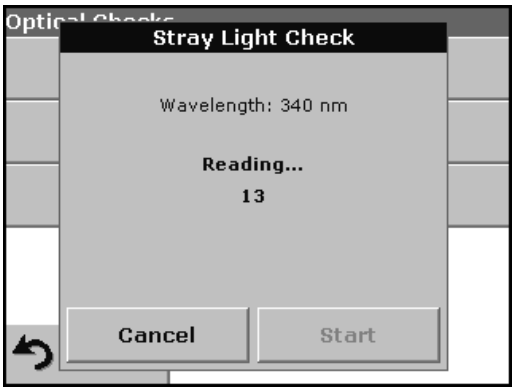
3. The result is displayed and is to be compared with the nominal/standard data (given in the quality control certificate) of the sample cuvette/cell.
4. Press **Cancel** to return to Optical Checks.

6.7.3.2 Stray light check

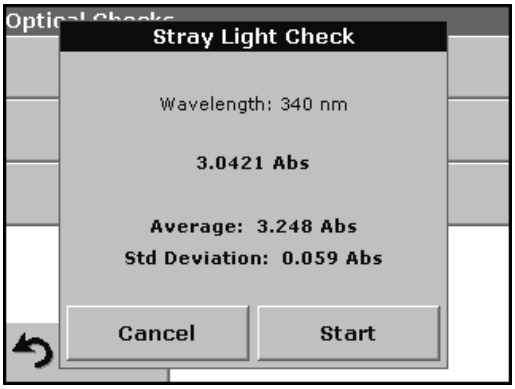
The Stray Light test is used to measure the stray light in the instrument at 340 nm.



1. Press **Stray Light Check** in the Optical Checks menu.
2. Remove any cuvette/sample cell from the cell compartment.
3. Insert the adapter (A) for 10-mm rectangular cells in the cell compartment #2. Press **Zero**.



4. Insert the sample cuvette/cell or reference filter into the cell compartment #2. Close the cell compartment. Press **Start**.

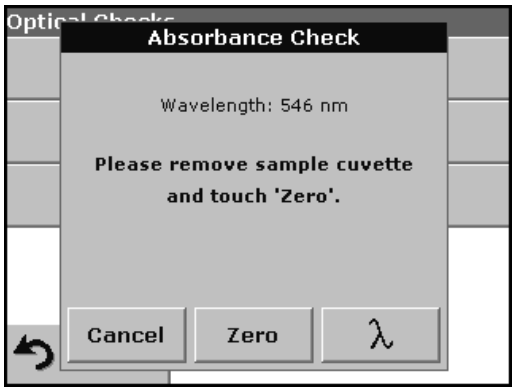


5. The ”Average“ and ”Std Deviation“ are calculated from 100 successive absorbance measurements. The result is displayed and is to be compared with the nominal/standard data (given in the quality control certificate) of the sample cuvette/cell.

   Note: *Failings and passings will be defined by the user.*

6. Press **Cancel** to return to Optical Checks.

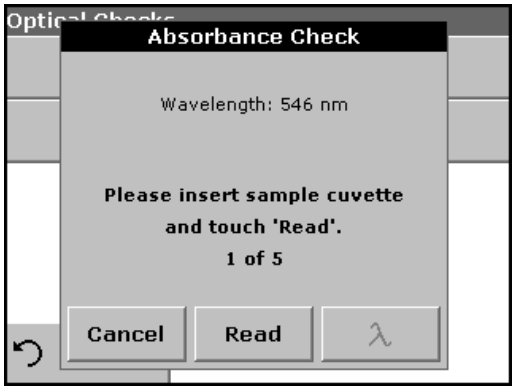
6.7.3.3 Absorbance check



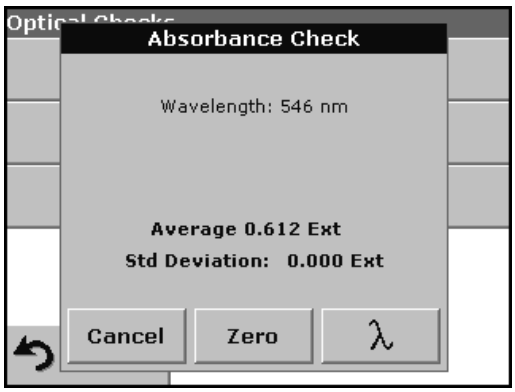
The Absorbance Check test is used to test the photometric accuracy and repeatability of the instrument.

This test can be used to test absorbance at any wavelength by a specific sample or test filter set (Refer to section 6.7.3.4 on page 102).

1. Press **Absorbance Check** in the Optical Checks menu.
2. Press λ to input the wavelength.
3. Input the wavelength and press **OK** to confirm.
4. Remove any cuvette/sample cell from the cell compartment and press **Zero**.



5. Insert sample cuvette/cell into the cell compartment and press **Read**.
6. 5 Replicates of blanking and reading lead to results.

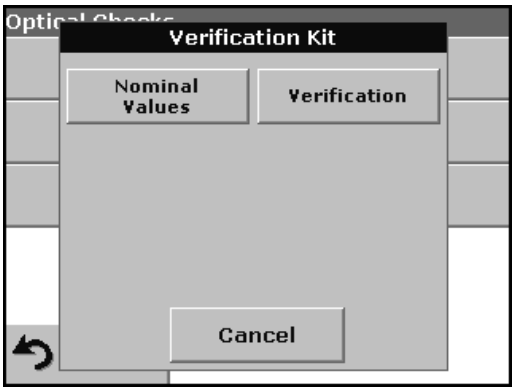


7. The result is displayed and is to be compared with the nominal/standard data of the sample cuvette/cell.
8. Press **Cancel** to return to Optical Checks.

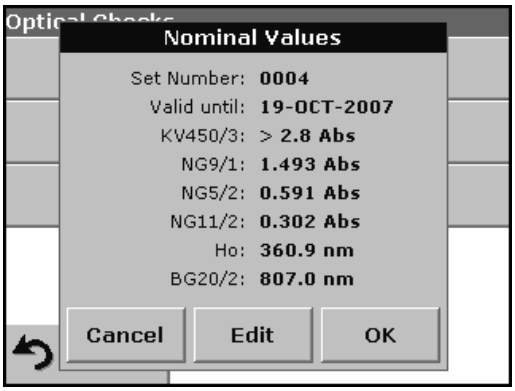
6.7.3.4 Verification kit

The Verification Kit, LZV537, (see Section 9 on page 119) is designed for periodic monitoring of scattered light, photometric accuracy and the wavelength accuracy of the spectrophotometers.

When results exceed allowable tolerances (given in the quality control certificate), contact the manufacturer.



1. Press **Verification Kit** in the Optical Checks menu.
2. Press **Nominal Values**.

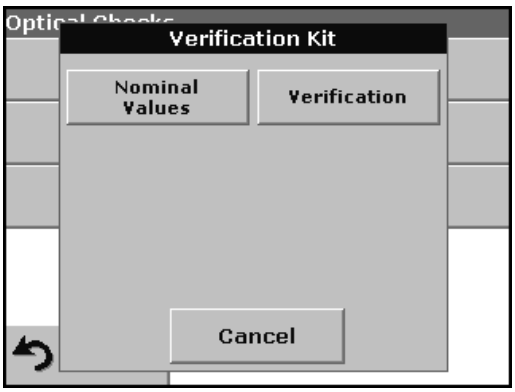


3. Press **Edit**.

   An automatic menu guidance queries values (filters, wavelength, nominal values and tolerances) given in the quality control certificate, to the following specifications:

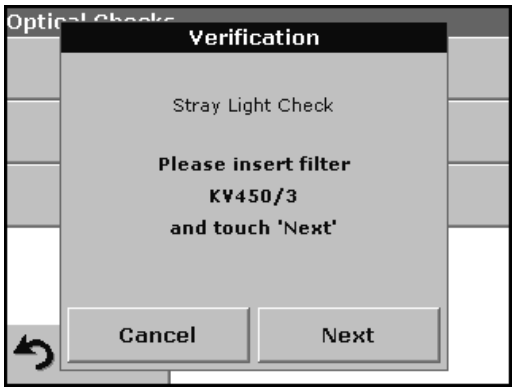
   - Stray Light
   - Photometrical accuracy
   - Wavelength accuracy

4. Press **OK** when all values are entered and the overview is displayed.

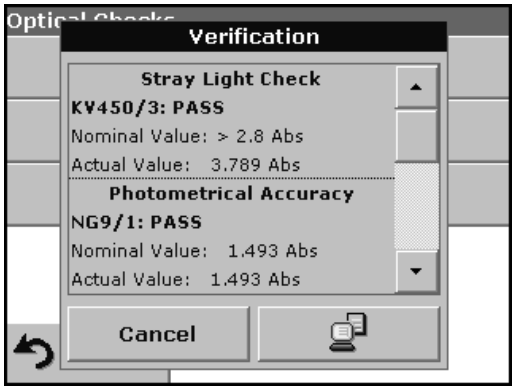


5. Press **Verification**.
6. Insert the adapter A (Figure 4 on page 15) in cell compartment #2.

7. Remove any cuvettes/cells from the cell compartment and press **Start**.



8. Insert the different filter in the given order one after the other. Press **Next** after inserting a filter.



After the last measurement the results are displayed.

9. Press **PC & Printer** icon to send the data to a USB memory stick, PC or to a printer.

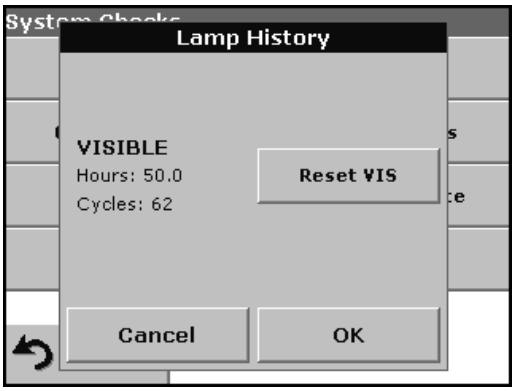
The files will be stored automatically as CSV file (Comma Separated Value). The file name will be formatted as “Verification.csv“.

6.7.4 Output checks

If a printer is connected a test printing of the current screen will be printed.

6.7.5 Lamp history

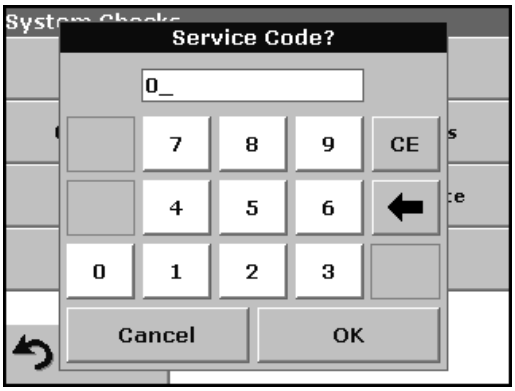
The Lamp History menu provides the amount of time that the lamp has been on (Hours).



After a lamp is replaced and the Lamp History is reset, the display of the total operating time is reset to 0.

1. Press **Lamp History** in the System Checks menu.
2. Press **Reset VIS** and the Visible Lamp will be reset.
3. Press **OK** to return to System Checks.

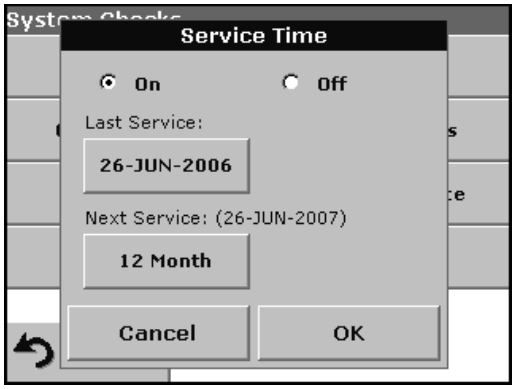
6.7.6 Factory service



The Factory Service menu is password protected. This menu is not intended for customer use.

6.7.7 Service time

In order to ensure a regular inspection, an automatic memory reference for the service times can be entered. After switching the instrument on this memory reference will be activated and indicated at the appropriate time.



1. Press **Service Time** in the System Checks menu.
2. Select **On** and then **Last Service** to enter the date of the last inspection.
3. Press **OK** to confirm.

4. Select **Next Service** to determine a specific period of time up to the next inspection.
5. Press **OK** to confirm.

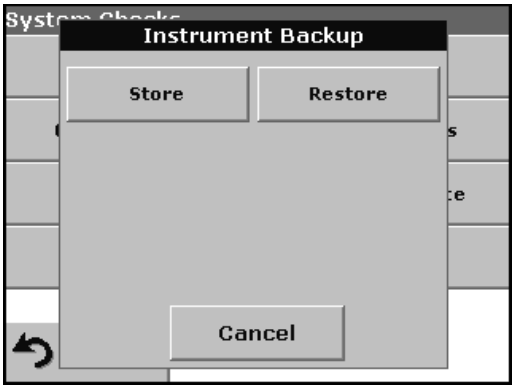
If the next service is due, the message "**Next service is due!**" is displayed after switching on the instrument.

6. Press **OK** to return to the Main Menu.

Contact the manufacturer or distributor to arrange an appointment for the next service.

6.7.8 Instrument Backup

Before the next service date the Instrument Backup menu offers the possibility to store all programs, measuring data, Operator ID, Sample ID, passwords and all adjustable data on a USB stick.



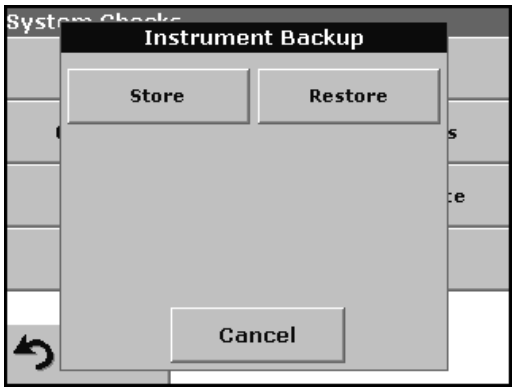
1. Press **Instrument Backup** in the System Checks menu.
2. Connect the USB memory stick (section 3.4 on page 13).
3. Press **Store** to start a Backup.

Note: *If the USB stick is not connected, the message "Please insert USB Memory" is displayed. Connect a USB stick, in order to store the data. Press* ***OK*** *to confirm and press* ***Store*** *again.*

Note: *If the Backup was already stored before, the message "Data already exists. Overwrite?" is displayed. Press* ***OK*** *to overwrite the data.*

If the file was stored the message Instrument Backup is stored to USB stick will be displayed.

4. Press **OK** to return to the System Checks menu.

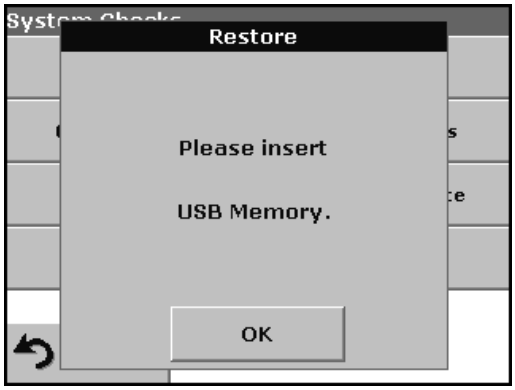


Restore backup data:
Important Note: *All current data will be overwritten when restoring the Backup file!*

1. Press **Instrument Backup** in the System Checks menu.

2. Connect the USB memory stick containing the Backup (section 3.4 on page 13).

3. Press **Restore** to pass back the data.



Note: *If the USB stick is not connected, the message "Please insert USB Memory" is displayed. Connect a USB stick, in order to store the data. Press **OK** to confirm and press **Restore** again.*

4. Press **OK** to confirm after the message “Instrument Backup from S/N XXXXXXX. Restore?“ is displayed.

5. After the backup start the instrument again.

CAUTION
Potential Chemical, Biological Eye and Skin Hazards.
Only qualified personnel should conduct the tasks described in this section of the manual.

Important Note: *Remove any cuvettes/cells that are still in the instrument and dispose of them or their contents using an approved disposal method.*

7.1 Cleaning requirements

CAUTION
Potential Pinch, Eye, Burn and Chemical Hazards.
Always disconnect power from the instrument before attempting any cleaning operations.

Important Note: *Under no circumstances should the instrument, display or the accessories be cleaned with solvents such as white spirit, acetone, etc.*

7.1.1 Spectrophotometer

- Clean the enclosure, cuvette/sample cell compartments and all accessories with a soft damp cloth. A mild soap solution can also be used. Do not get excess water in the cuvette/sample cell compartments. Do not insert a brush or sharp object into Cell Compartment #1 to avoid damaging the mechanical components.
- Dry the cleaned parts carefully with a soft cotton cloth.

7.1.2 Display

- Take care not to scratch the display. Do not touch the screen with ball pens, pencils or similar pointed objects.
- Clean the display with a soft, lint-free and oil-free cotton cloth. Diluted window cleaner liquid can also be used.

7.1.3 Cuvettes/sample cells

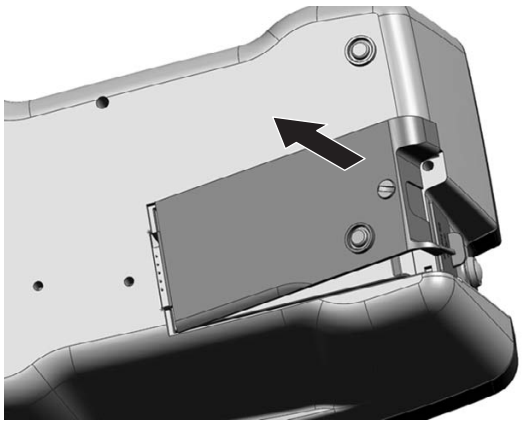
CAUTION
Potential Chemical/ Biological Exposure Hazards.
Use proper laboratory practices whenever there is a risk of chemical exposure.

1. After performing a procedure, clean glass cuvettes/sample cells with cleaning agents.

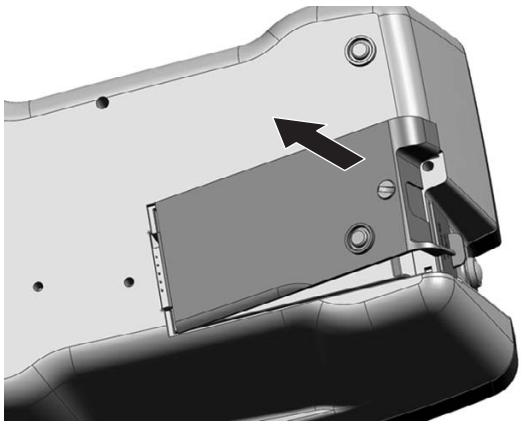
2. Afterwards, rinse the cuvettes/sample cells several times with tap water and then thoroughly with deionized water.

Important Note: *Glass cuvettes/sample cells that have been used for organic solvents (such as chloroform, benzene, toluene, etc.) must be rinsed with acetone before being treated with cleaning agents. In addition, another rinse with acetone is necessary as a final treatment step before the cuvettes/sample cells are dried.*

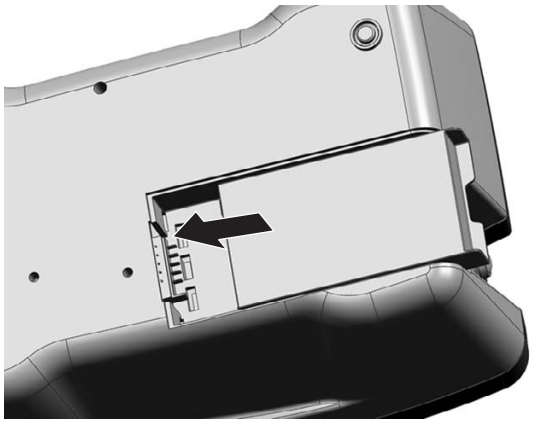
7.2 Insert or change of the battery



1. Remove the cuvette from the cell compartment.
2. Switch the instrument off.
3. Unplug the power cord.
4. Turn the instrument over carefully and place it on a soft surface.

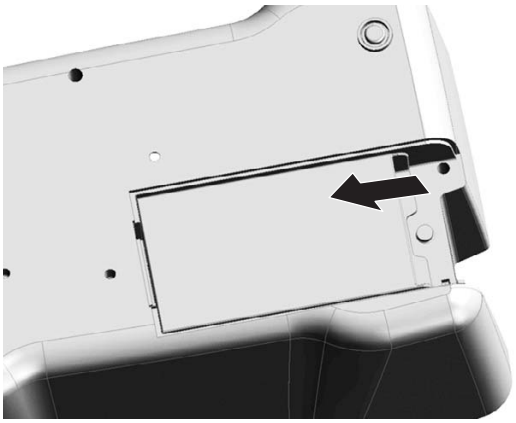


5. Use a screwdriver or a coin to remove the screw from the cover.
6. Remove the cover and place it to one side.



CAUTION
For quality and safety reasons, only the manufacturer's lithium batteries (LZV551) should be used for this instrument. Use of non-instrument-specific battery types may impair the functioning of and/or damage the instrument electronics by overloading them, or depending on battery types can even result in fire or explosion hazards.

7. Carefully push the battery from right to left into the compartment, with the labeled face upward.



8. Push in the battery so that the contacts of the instrument can be plugged into the socket of the battery.

Important Note: *Take care that the battery contacts are always clean. Soiled contacts can result in contact heating and voltage drop, interfering with the proper operation of the spectrophotometer.*

9. Use a screwdriver or a coin to replace the screw securing the cover.

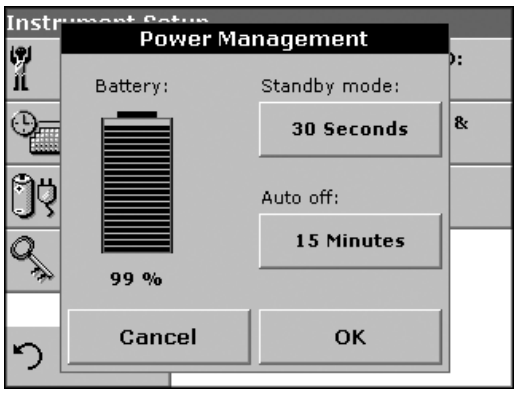
10. Carefully stand the instrument upright.

11. Plug in power supply again – the Instrument is now ready for use. The battery charges up.

7.2.1 Information about using the battery

7.2.1.1 Dispose of the lithium battery

A lithium battery allows the DR 2800 to be used as a portable instrument.



Please dispose the batteries of in compliance with the country-specific regulations (information about the charge status of the battery can be found in the "Instrument Setup" menu under 5.2.5 Power Management on page 29).

CAUTION
Pay special attention to the following information for the Lithium battery pack:

- Do not short-circuit the contacts.
- Keep the battery away from fire.
- Do not expose the battery to temperatures above 60°C (such temperatures may occur, for example, in a car left in the sun, or in direct sunlight).
- Take care that the battery does not become wet.
- Take care that the battery is not exposed to impacts, blows, etc. Do not drop it. Do not step on it by accident.
- Do not take the battery apart or modify it in any way.

- The performance of the battery decreases with the temperature.

7.2.1.2 Optimal operation of the battery

The battery is charged automatically when the plug-in power supply on the back of the instrument is connected and is plugged into a mains socket (100–240 Volt/50–60 Hz).

CAUTION
Use only the manufacturer's specified external power supply to operate the instrument and charge the internal lithium battery pack. The specified power supply and it's plug shape, it voltage rating (12V, 2A) and it's electromagnetic characteristics are critical to assuring the DR 2800's performance and the safety of the user.

Important Note: *We advise charging the battery at an ambient temperature between 10°C and 30°C, as this is the range in which it can be most effectively charged to 100%.*

7.2.1.3 Lifespan of the battery

The battery has a limited lifespan. The more it is used, the more its capacity is reduced. If a fully charged battery only allows the instrument to be used for a relatively short time, replace the battery with a new one.

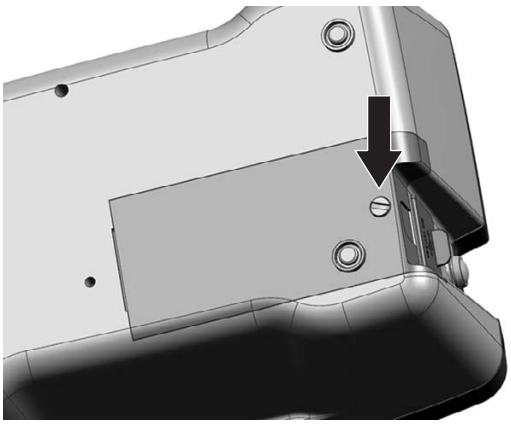
Note: *You need not run down the battery completely before recharging it. If you recharge the battery when it still has some residual charge, this does not reduce its capacity!*

7.2.1.4 Load the battery/Operating time

The time required to charge a newly installed battery is about 3.5 hours.

Operating time of a fully charged battery:
Charge: If a battery is charged to 100% capacity, and the user carries out 10 measurements per day, and the instrument is left on after each measurement for the full 15 minutes before it automatically switches off, the battery can be used for 6 to 7 days before it needs recharging.

7.3 Lamp replacement

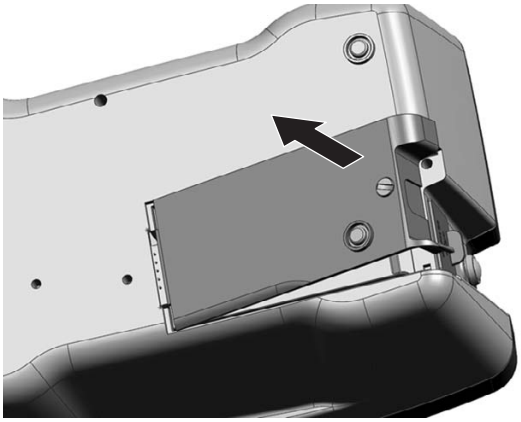


CAUTION
To avoid a possible electric shock, disconnect the instrument from the power source before servicing the lamp.

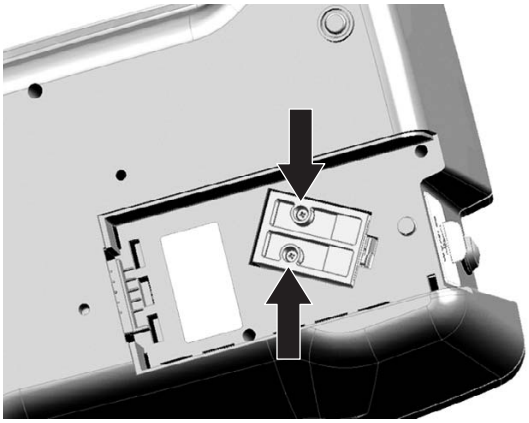
1. Remove the cuvette from the cell compartment.
2. Switch the instrument off.
3. Unplug the power cord.

WARNING
Burn Hazard. Wait until the lamp cools down. Contact with the hot lamp can cause burns.

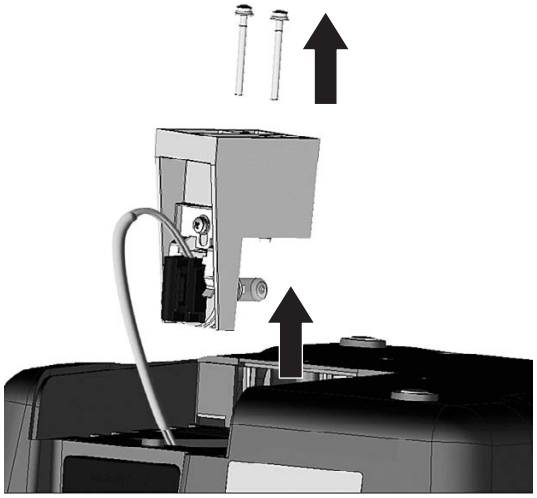
4. Turn the instrument over carefully and place it on a soft surface.
5. Use a screwdriver or a coin to remove the screw from the cover.



6. Remove the cover and place it to one side.
7. If there is a battery in the instrument, push it out and put it to one side too, see section 7.2.1 on page 111.



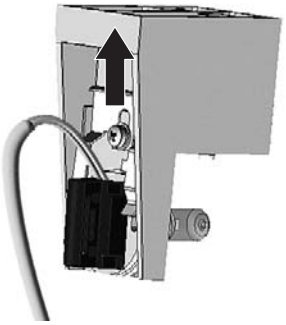
8. Use a screwdriver (slotted or cross-headed) to remove the screws from the lamp fitting.



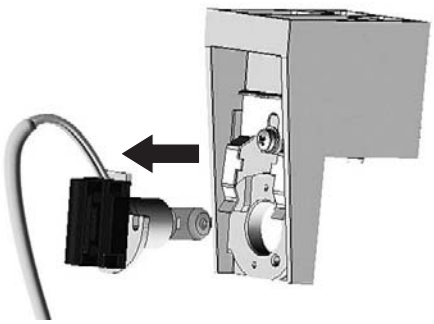
WARNING
Burn Hazard. Wait until the lamp cools down. Contact with the hot lamp can cause burns.

9. Lift the lamp fitting carefully.

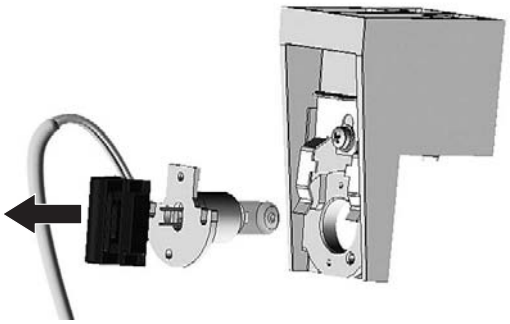
10. Put both screws to one side.



11. Push up on the pressure spring.



12. Remove the halogen lamp complete with the plug panel.



13. Carefully unplug the halogen lamp from the plug panel.

Important Note: *Hold the lamp by the fitting only. Avoid touching the glass, as substances on the skin can bake onto the lamp bulb and thus accelerate the aging process of the lamp.*

14. Plug a new halogen lamp to the panel.

15. Insert the halogen lamp with the half rounded part pointing down.

16. Press the plug with slight pressure into the direction of the halogen lamp and push the pressure spring down, so that it will engage.

17. Carefully insert the lamp fitting again.

18. Use a screwdriver (slotted or cross-headed) to replace the screws to secure the lamp fitting.

19. Put the battery back in place, see section 7.2.1 on page 111.

20. Use a screwdriver or a coin to screw the back cover to the instrument.

21. Carefully stand the instrument upright.

22. Plug in the power supply.

23. Reset the Lamp History, see section 6.7.5 on page 104.

Section 8 Troubleshooting

Problem/Display screen	Likely Cause	Action
Attention! **Please insert the light shield.**	Measurements with barcode cuvettes generally require the light shield.	Insert the light shield. Press **OK**.
Absorbance > 3.5!	The measured absorbance exceeds 3.5	Dilute the sample and repeat the measurement
Concentration too high!	Calculated concentration is higher than 999999	Dilute the sample and repeat the measurement
Error **Barcode controlnumber?** **Please update program data!**	Deviation to the stored data	Data updating
Error **Program not available.** **Please update program data!**	Barcode cuvette test missing	Data updating
Error **Clean Cuvette**	The cuvette is soiled or there are undissolved particles in the cuvette	Clean the cuvette; allow the particles to settle
Error **Selfcheck stopped.** **Please check the lamp.** **Please close the lid.** **Error [xx]**	Self Check Test stops while starting the instrument	Check the lamp and replace it if necessary. Close the lid. Press **Start Again**.
Error **Selfcheck stopped.** **Please remove the cuvette**	Self Check Test stops while starting the instrument	Remove the cuvette. Press **OK**.
Error **Selfcheck stopped.** **Hardware error.** **Error [x]**	Electronic defect	Contact the manufacturer or a sales representative and indicate the error number
It's recommended to execute a **Full System Check** **Please remove cuvette.** **Please close the lid.**	The instrument needs a new Full System Check.	Remove cuvette. Close the lid. Press **Start**. If the Full System Check fails, please contact the manufacturer or distributor.
Negative result!	The calculated result is negative	Check the concentration of the sample
No evaluation!	Error in the test database / user database	Check the programming Contact the manufacturer or a sales representative
Over measuring range	The measured absorbance is above the calibration range of the test	Dilute the sample and repeat the measurement
Please check the lamp.	The lamp output is too low	Check the lamp and replace it if necessary
Too much ambient light! **Move device into shade** **or close the lid!**	The instrument sensors detects to much ambient light.	Reduce ambient light. (Avoid direct sun light.) Close the lid.
Under measuring range	The measured absorbance is below the calibration range of the test	If possible, select a test with a lower measurement range or use a cuvette with a longer path length
Unstable lighting conditions!	Fluctuating light conditions during the measurement	Close the lid or place the Protective Cover on the cuvette compartment (2)

Section 9 Replacement Parts

Description	Cat. No.
Rechargeable lithium battery	LZV551
Tungsten Lamp	LZV565
Cuvette/ Sample cell adapter 10 mm (A)	LZV583
Cuvette/ Sample cell adapter 1 inch (C)	LZV584
Light Shield	LZV646
Cell adapter for Pour-Thru Cell (B)	LZV585
Cell adapter set (cuvette adapters A, B, and C)	LZV647
Wall power supply	LZV610
Protective Cover and adapter holder	LZV642
Dust cover	HYH019
USB-Memory Stick	2946900
USB-Interface Cable (1 m)	LZV567
USB-Keyboard (keyboard layout: US)	LZV582
USB-Barcode Scanner (handset scanner)	LZV566
Pour-Thru Kit	5940400
Cuvette/ Sample cell set (1-inch square cuvettes/cells, matched pair, glass (10 mL)	2495402
Hach Data Trans (PC software for data transfer)	LZY274
Certified test filter set for self-checks (6 precision glass filters with target values)	LZV537

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Section 11 Limited Warranty

Hach Company warrants its products to the original purchaser against any defects that are due to faulty material or workmanship for a period of one year from date of shipment unless otherwise noted in the product manual.

In the event that a defect is discovered during the warranty period, Hach Company agrees that, at its option, it will repair or replace the defective product or refund the purchase price excluding original shipping and handling charges. Any product repaired or replaced under this warranty will be warranted only for the remainder of the original product warranty period.

This warranty does not apply to consumable products such as chemical reagents; or consumable components of a product, such as, but not limited to, lamps and tubing.

Contact Hach Company or your distributor to initiate warranty support. Products may not be returned without authorization from Hach Company.

Limitations

This warranty does not cover:

- Damage caused by acts of God, natural disaster, labor unrest, acts of war (declared or undeclared), terrorism, civil strife or acts of any governmental jurisdiction
- Damage caused by misuse, neglect, accident or improper application or installation
- Damage caused by any repair or attempted repair not authorized by Hach Company
- Any product not used in accordance with the instructions furnished by Hach Company
- Freight charges to return merchandise to Hach Company
- Freight charges on expedited or express shipment of warranted parts or product
- Travel fees associated with on-site warranty repair

This warranty contains the sole express warranty made by Hach Company in connection with its products. All implied warranties, including without limitation, the warranties of merchantability and fitness for a particular purpose, are expressly disclaimed.

Some states within the United States do not allow the disclaimer of implied warranties and if this is true in your state the above limitation may not apply to you. This warranty gives you specific rights, and you may also have other rights that vary from state to state.

This warranty constitutes the final, complete, and exclusive statement of warranty terms and no person is authorized to make any other warranties or representations on behalf of Hach Company.

Limitation of Remedies

The remedies of repair, replacement or refund of purchase price as stated above are the exclusive remedies for the breach of this warranty. On the basis of strict liability or under any other legal theory, in no event shall Hach Company be liable for any incidental or consequential damages of any kind for breach of warranty or negligence.

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CAT. NO. 46500-88

PORTABLE TURBIDIMETER

Model 2100P

Instrument and Procedure Manual

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CERTIFICATION

Hach Company certifies this instrument was tested thoroughly, inspected and found to meet its published specifications when it was shipped from the factory.

The Model 2100P Portable Turbidimeter has been tested and is certified as indicated to the following instrumentation standards:

Product Safety

Battery/Eliminator Power Supply Only:
120 Vac, 60 Hz, UL Listed & CSA Certified, Class 2
230 Vac, 50 Hz, VDE Approved, GS & CE marked

Immunity

2100P Turbidimeter Tested with external Battery/Eliminator Power Supply:
EN 50082-1 (European Generic Immunity Standard) **per 89/336/EEC EMC:** Supporting test records with Dash Straus and Goodhue, Inc. (now Intertek Testing Services), certified compliance by Hach Company.

Standards include:
IEC 801-2 Electro-Static Discharge
IEC 801-3 Radiated RF Electro-Magnetic Fields
IEC 801-4 Electrical Fast Transients/Burst

Emissions

2100P Turbidimeter Tested with external Battery/Eliminator Power Supply:
EN 50081-1 (Emissions) **per 89/336/EEC EMC:** Supporting test records by Amador Corp. (now TUV Product Services), certified compliance by Hach Company

Standards include:
EN 55022 (CISPR 22) Emissions, Class B Limits

Canadian Radio Interference-Causing Regulation, Chapter 1374, Class A: Supporting test records by Amador Corp. (now TUV Product Services), certified compliance by Hach Company

This Class A digital apparatus meets all requirements of the Canadian Interference-Causing Equipment Regulations.

Cet appareil numérique de la classe A respecte toutes les exigences du Règlement sur le matériel brouilleur du Canada.

CERTIFICATION, continued

FCC Part 15, Class "A" Limits: Supporting test records by Amador Corp. (now TUV Product Services), certified compliance by Hach Company.

This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions:

1. this device may not cause harmful interference, and
2. this device must accept any interference received, including interference that may cause undesired operation.

Changes or modifications to this unit not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment.

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area may cause harmful interference in which case the user will be required to correct the interference at his own expense.

The following techniques of reducing interference problems are applied easily:

1. Disconnect the battery eliminator from it's power source and from the 2100P Portable Turbidimeter to verify if it is the source of the interference
2. If the battery eliminator for the 2100P Portable Turbidimeter is plugged into the same outlet as the device with which it is interfering, try another outlet.
3. Move the 2100P Portable Turbidimeter away from the device receiving the interference.
4. Reposition the receiving antenna for the device receiving the interference.
5. Try combinations of the above.

SAFETY PRECAUTIONS

Please read this entire manual before unpacking, setting up, or operating this instrument. Pay particular attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

To ensure the protection provided by this equipment is not impaired, do not use or install this equipment in any manner other than that which is specified in this manual.

Use of Hazard Information

If multiple hazards exist, this manual will use the signal word (Danger, Caution, Note) corresponding to the greatest hazard.

DANGER
Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION
Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTE
Information that requires special emphasis.

Precautionary Labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed.

⚠ This symbol, if noted on the instrument, references the instruction manual for operational and/or safety information.

SPECIFICATIONS

Specifications subject to change without notice.
Operating specifications applicable at 25 °C unless noted.
Program software copyrighted by Hach Company, 1991.

Measurement Method: Ratio Nephelometric signal (90°) scatter light ratio to transmitted light

Range: 0-1000 NTU with automatic decimal point placement or manual range selection of 0-9.99, 0-99.9 and 0-1000 NTU

Accuracy: ± 2% of reading plus stray light from 0-1000 NTU

Resolution: 0.01 NTU on lowest range

Repeatability: ±1% of reading or 0.01 NTU, whichever is greater (with Gelex standards)

Response Time: 6 seconds for full step change without signal averaging in constant reading mode

Stray Light: <0.02 NTU

Standardization: StablCal® Stabilized Formazin primary standards or Formazin primary standards

Secondary Standards: Gelex® Secondary Standards

Display: Four-digit liquid crystal; 10.16 mm (0.4 in) high digits with custom icons

Light Source: Tungsten filament lamp; lamp life typically greater than 100,000 readings

Detectors: Silicon photovoltaic

Signal Averaging: Operator selectable on or off

Sample Cells: (Height X width) 60.0 X 25 mm (2.36 X 1 in) Borosilicate glass with screw caps, marking band and fill line

Sample Required: 15 mL (0.5 oz.)

SPECIFICATIONS, continued

Storage Temperature: -40 to 60 °C (-40 to 140 °F) (instrument only)

Operating Temperature: 0 to 50 °C (32 to 122 °F) (instrument only)

Operating Humidity Range: 0 to 90% RH noncondensing at 30 °C;
0 to 80% RH noncondensing at 40 °C;
0 to 70% RH noncondensing at 50 °C

Power Requirements: Four AA Alkaline cells or optional battery eliminator

Battery Life: Typically 300 tests with signal average mode off; 180 tests with signal average mode on

Battery Eliminator (optional):

For 120 V eliminator: CSA and UL approved for 120 VAC ±10%, 60 Hz, 6 V at 800 mA DC output

For 230 V eliminator: CE (VDE) approval pending for 230 VAC ±10%, 50 Hz, 6 V at 900 mA DC output

Enclosure: High impact ABS plastic

Dimensions: 22.2 X 9.5 X 7.9 cm (8.75 X 3.75 X 3.12 in)

Instrument Weight: 520 kg (1 lb 2.5 oz)

Shipping Weight: 3.1 kg (6 lbs 8.5 oz)

OPERATION

DANGER

Handling chemical samples, standards, and reagents can be dangerous. Review the necessary Material Safety Data Sheets and become familiar with all safety procedures before handling any chemicals.

DANGER

La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les Fiches de Données de Sécurité des Produits (FDSP) et se familiariser avec toutes les procédures de sécurité avant de manipuler tous les produits chimiques.

PELIGRO

La manipulación de muestras químicas, estándares y reactivos puede ser peligrosa. Revise las fichas de seguridad de materiales y familiarícese con los procedimientos de seguridad antes de manipular productos químicos.

GEFAHR

Da das Arbeiten mit chemischen Proben, Standards und Reagenzien mit Gefahren verbunden ist, empfiehlt die Hach Company dem Benutzer dieser Produkte dringend, sich vor der Arbeit mit sicheren Verfahrensweisen und dem richtigen Gebrauch der Chemikalien vertraut zu machen und alle entsprechenden Materialsicherheitsdatenblätter aufmerksam zu lesen.

PERIGO

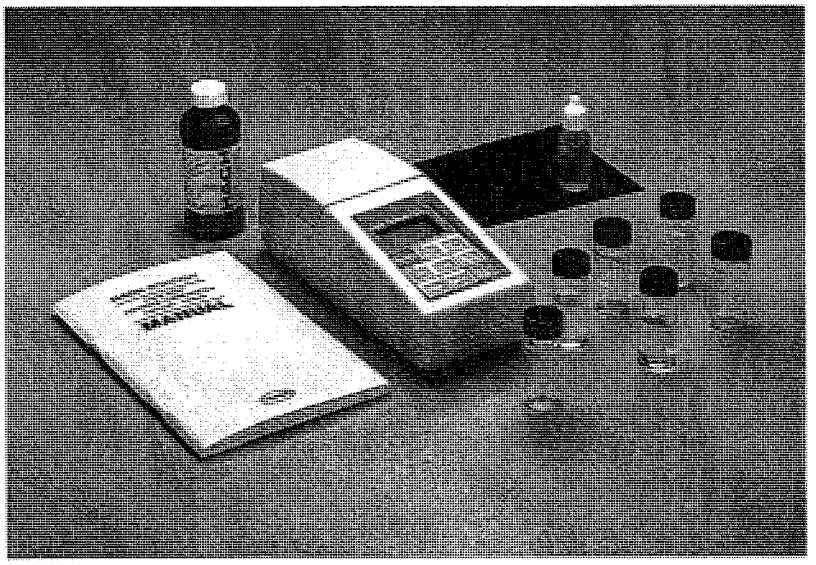
A manipulação de amostras, padrões e reagentes químicos pode ser perigosa. Reveja a folha dos dados de segurança do material e familiarize-se com todos os procedimentos de segurança antes de manipular quaisquer produtos químicos.

SECTION 1 DESCRIPTION

1.1 General Description

The Hach Model 2100P Portable Turbidimeter (*Figure 1*) measures turbidity from 0.01 to 1000 NTU in automatic range mode with automatic decimal point placement. The manual range mode measures turbidity in three ranges: 0.01 to 9.99, 10 to 99.9 and 100 to 1000 NTU. Designed primarily for field use, the microprocessor-based Model 2100P has the range, accuracy, and resolution of many laboratory instruments. The instrument operates on four AA batteries or with an optional battery eliminator. Rechargeable nickel-cadmium cells may be used, but cannot be recharged in the instrument. The instrument automatically shuts off after 5.5 minutes if no keystrokes occur (does not influence operation). If this occurs, simply turn the instrument on – the 2100P will resume operation as if the power had not been interrupted. The instrument, all standard accessories, and the optional battery eliminator may be conveniently stored in the carrying case.

Figure 1 2100P Turbidimeter and Accessories



Note: *Avoid prolonged exposure to ultraviolet light and sunlight.*

Note: *Do not hold the instrument during measurements; place the instrument on a flat, steady surface.*

1.2 Accessories

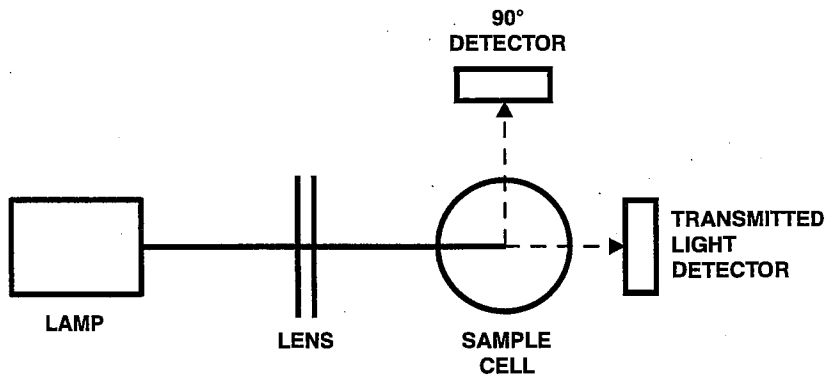
Accessories supplied with the turbidimeter include three sample cells; three Gelex® Secondary Standards; one 100 mL bottle each of: <0.1-NTU, 20-NTU, 100-NTU, and 800-NTU StablCal® Stabilized Formazin Standards; 4 AA alkaline batteries; 15 mL of silicone oil; oiling cloth; carrying case; instrument manual; and quick reference card.

1.3 Principle of Operation

The Model 2100P Portable Turbidimeter operates on the nephelometric principle of turbidity measurement. This instrument meets the design criteria specified by the United States Environmental Protection Agency, Method 180.1.

The optical system* (*Figure 2*) includes a tungsten-filament lamp, a 90° detector to monitor scattered light and a transmitted light detector. The instrument's microprocessor calculates the ratio of the signals from the 90° and transmitted light detectors. This ratio technique corrects for interferences from color and/or light absorbing materials (such as activated carbon) and compensates for fluctuations in lamp intensity, providing long-term calibration stability. The optical design also minimizes stray light, increasing measurement accuracy.

Figure 2 Ratio Optical System



* Patent number 4,198,161; other patents pending.

1.4 Preparation for Use

1.4.1 Unpacking

Remove the instrument and accessories from the shipping box and inspect them for damage that may have occurred due to rough handling or extreme weather conditions. Verify the following are present:

- Model 2100P Portable Turbidimeter
- Instrument Manual (with quick reference card)
- StablCal Stabilized Formazin Standard, <0.1 NTU*, 100 mL
  StablCal Stabilized Formazin Standard, 20 NTU, 100 mL
  StablCal Stabilized Formazin Standard, 100 NTU, 100 mL
  StablCal Stabilized Formazin Standard, 800 NTU, 100 mL
- Standardization Kit containing Gelex Secondary Standards (0-10, 0-100 and 0-1000 ranges) plus nine sample cells with caps.
- Silicone Oil, 15-mL (0.5 oz) dropping bottle
- Oiling Cloth
- Carrying Case
- Four AA alkaline batteries

If any of the items are missing or damaged, please contact the Customer Service Department, Hach Company, Loveland, Colorado. The toll-free number in the United States is 800-227-4224. International customers should contact the Hach office or authorized distributor serving your area. Refer to *REPAIR SERVICE* on page *68*. **Please do not return the instrument without prior authorization from Hach.**

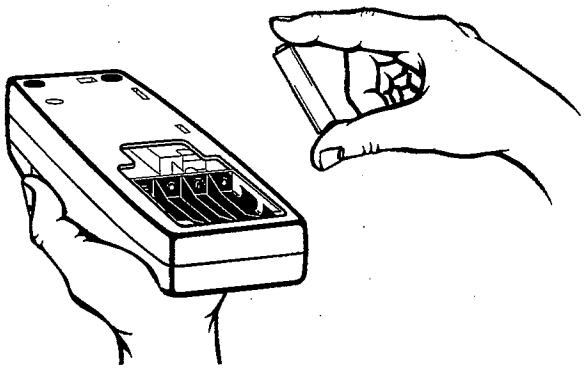
1.4.2 Battery Installation

The instrument is shipped completely assembled without the batteries installed. Before use, install the four AA alkaline batteries or connect the battery eliminator (*Figure 3*). For battery operation, remove the

* Used in place of the dilution water standard when performing a calibration.

battery compartment cover on the instrument bottom and install the batteries. Correct battery polarity is shown on the battery holder. The instrument will not function if the batteries are not installed correctly. Reinstall the battery compartment cover.

Figure 3 Battery Installation



1.4.3 Using the Battery Eliminator and Rechargeable Batteries

For operation with the optional battery eliminator, plug the eliminator jack into the connector on the turbidimeter side. The battery eliminator may be used with or without the batteries installed. **The eliminator will not charge batteries.** Rechargeable batteries may be used in the instrument, but must be removed for recharging. See *HOW TO ORDER* on page *67* for ordering information. To prolong battery life, the instrument lamp turns on temporarily when the **READ** key is depressed. Batteries are not necessary for battery eliminator operation.

1.4.4 Calibration

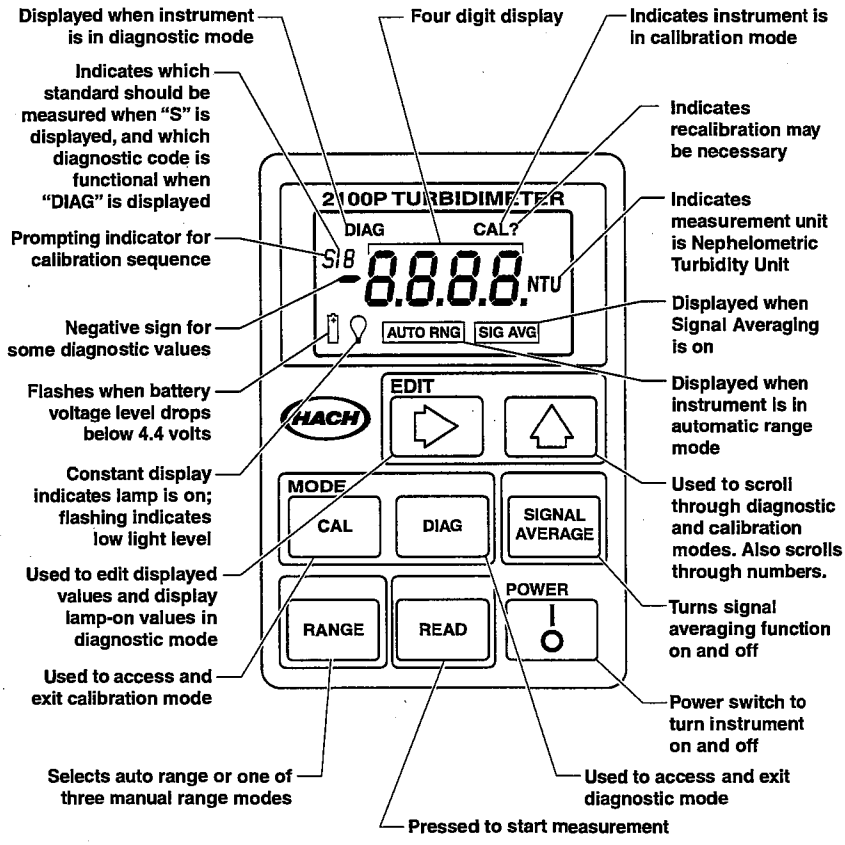
The 2100P Portable Turbidimeter is calibrated with Formazin Primary Standard at the factory and **does not require recalibration before use.** Hach recommends recalibration with formazin once every three months, or more often as experience dictates. The Gelex Secondary Standards supplied with the instrument are labelled with general ranges for application, but must be assigned values before use from formazin calibration. See *Section 3.6* on page *27* for calibration instructions.

SECTION 2 TURBIDITY MEASUREMENT

2.1 Operating Controls and Indicators

Figure 4 shows the 2100P controls and indicators. Refer to *SECTION 3* for a detailed description of each control and indicator.

Figure 4 Keyboard and Display with Descriptions



2.2 Turbidity Measurement

Measurements may be made with the signal average mode on or off and in manual or automatic range selection mode. Using automatic range selection is recommended. Signal averaging uses more power and should be used only when the sample causes an unstable reading. Signal averaging measures and averages ten measurements while displaying

intermediate results. The initial value is displayed after about 11 seconds and the display is updated every 1.2 seconds until all ten measurements are taken (about 20 seconds). After this, the lamp turns off, but the final measured turbidity value continues to be displayed until another key is pressed.

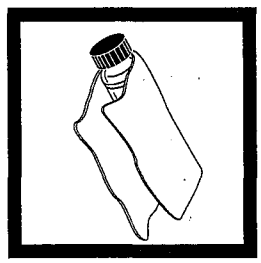
When not in signal average mode, the final value is displayed after about 13 seconds.

Accurate turbidity measurement depends on good measurement technique by the analyst, such as using clean sample cells in good condition and removing air bubbles (degassing). Refer to *Section 2.3* on page *10* for a detailed discussion of measurement techniques.

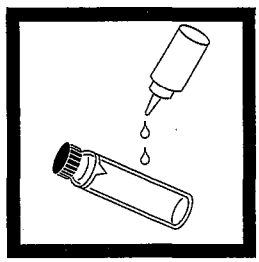
2.2.1 Turbidity Measurement Procedure

1. Collect a representative sample in a clean container. Fill a sample cell to the line (about 15 mL), taking care to handle the sample cell by the top. Cap the cell. (See *Section 2.3* on page *10* for more information about collecting a representative sample).

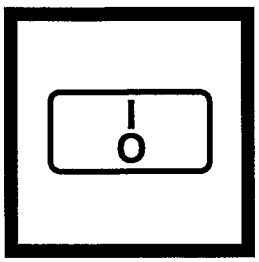
Note: *The instrument automatically shuts off after 5.5 minutes if no keystrokes occur. To resume operation, press* I/O.



2. Wipe the cell with a soft, lint-free cloth to remove water spots and fingerprints.

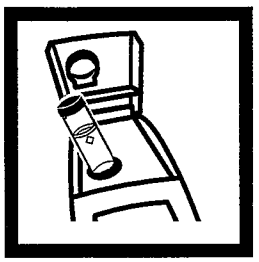


3. Apply a thin film of silicone oil. Wipe with a soft cloth to obtain an even film over the entire surface.

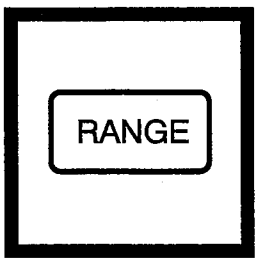


4. Press: **I/O**.

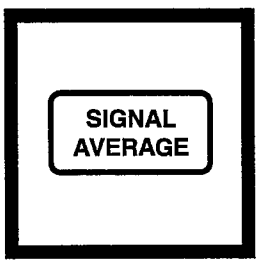
The instrument will turn on. Place the instrument on a flat, sturdy surface. Do not hold the instrument while making measurements.



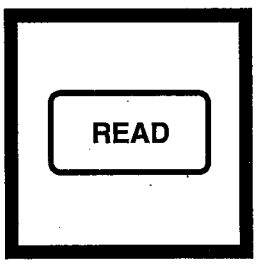
5. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. **Close the lid.**



6. Select manual or automatic range selection by pressing the **RANGE** key. The display will show **AUTO RNG** when the instrument is in automatic range selection.



7. Select signal averaging mode by pressing the **SIGNAL AVERAGE** key. The display will show **SIG AVG** when the instrument is using signal averaging. Use signal average mode if the sample causes a noisy signal (display changes constantly).



8. Press: **READ**

The display will show **- - - - NTU**, then the turbidity in NTU. Record the turbidity after the lamp symbol turns off.

Note: *The instrument defaults to the last operating mode selected. If automatic range mode and signal averaging were used on the previous measurements, these options will automatically be selected for subsequent samples.*

2.2.2 Measurement Notes

- Always cap the sample cell to prevent spillage of sample into the instrument.
- When taking a reading, place the instrument on a level, stationary surface. It should not be held in the hand during measurement.
- Always close the sample compartment lid during measurement and storage.
- Always use clean sample cells in good condition. Dirty, scratched, or damaged cells can cause inaccurate readings.
- Do not leave a sample cell in the cell compartment for extended periods of time. This may compress the spring in the cell holder.
- Remove sample cell and batteries from instrument if the instrument is stored for extended time period (more than a month).
- Avoid operating in direct sunlight.
- Make certain cold samples do not "fog" the sample cell.
- Avoid settling of sample prior to measurement.
- Keep sample compartment lid closed to prevent dust and dirt from entering.

2.3 Measurement Techniques

Proper measurement techniques are important in minimizing the effects of instrument variation, stray light and air bubbles. Regardless of the instrument used, measurements are more accurate, precise and repeatable if the analyst pays close attention to proper measurement techniques.

Measure samples immediately to prevent temperature changes and settling. Avoid sample dilution when possible. Particles suspended in the original sample may dissolve or otherwise change characteristics when the sample temperature changes or when the sample is diluted, resulting in a non-representative sample measurement.

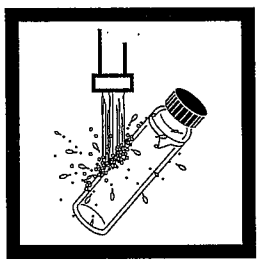
2.3.1 Cleaning Sample Cells

Cells must be extremely clean and free from significant scratches. The glass used to make cells is easily scratched – manufacturing cells free of minor scratches and other imperfections is difficult. However, minor imperfections are effectively masked by applying silicone oil as outlined in *Section 2.3.2*.

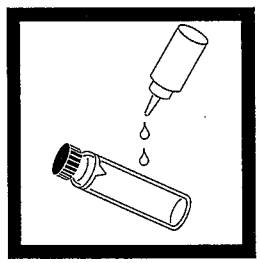
Clean the inside and outside of the cells by washing with laboratory detergent. Follow with multiple rinses of distilled or deionized water. Allow cells to air dry. Handle cells only by the top to minimize dirt, scratches and fingerprints in the light path.

2.3.2 Oiling the Sample Cell

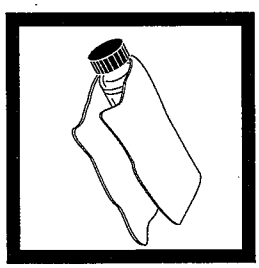
Applying a thin coat of silicone oil will mask minor imperfections and scratches which may contribute to turbidity or stray light. Use silicone oil equivalent to Hach Cat. No. 1269-36. This silicone oil has the same refractive index as glass. When applied in a thin, uniform coat, the oil fills in and masks minor scratches and other imperfections in the glass. Apply the oil uniformly by wiping with a soft, lint-free cloth.
Avoid application of excess oil. Applying excess oil may retain dirt and contaminate the instrument's cell compartment.



1. Thoroughly clean the sample cell.



2. Apply a small bead of silicone oil from the top to the bottom of the cell-- just enough to coat the cell with a thin layer of oil.



3. Using a soft, lint-free cloth, spread the oil uniformly, then wipe off the excess so that only a thin coat of oil is left. The cell should appear nearly dry with little or no visible oil.

Note: *Soft, lint-free cloth (velvet) works well for oiling. Store the oiling cloth with the sample cells and keep it free of dirt. After a few applications of oil, the cloth will contain enough residual oil that simply wiping the cell with the oiled cloth will provide a sufficient oil coat on the sample cell. Periodically, add a small amount of oil to the sample cell surface to replenish the oil in the cloth.*

Note: *Only a thin coat of oil on the sample cells is necessary.* ***Avoid using excessive amounts of oil.***

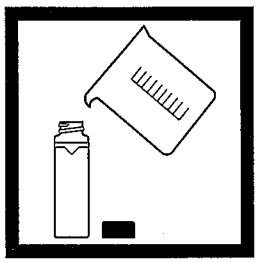
2.3.3 Orienting Sample Cells

Note: *When orienting and matching cells, it may be more efficient to use the continuous reading mode. The instrument performs continuous readings if the* **READ** *key is pressed and held. As long as the key is held, the lamp remains on and the display is updated every 1.2 seconds. The instrument cannot be used in continuous read mode if the Signal Averaging mode is on.*

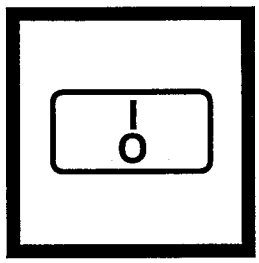
Precise measurements for very low turbidity samples require using a single cell for all measurements or optically matching the cells. Using one cell provides the best precision and repeatability. When one cell is used, an orientation mark (other than the factory-placed diamond) can be placed on the cell so it's inserted into the instrument with the same orientation each time.

2.3.3.1 Orienting a single cell

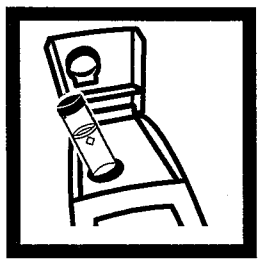
When using a single cell, make an index or orientation mark on the cell as follows:



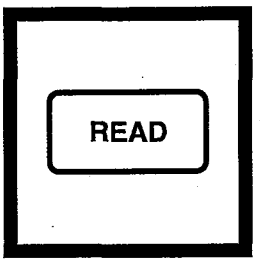
1. Fill the clean sample cell to the line with high quality water (< 0.5 NTU). Cap and wipe with lint-free cloth. Apply silicone oil. See *Section 3.6.2.2* on page *30* for more information about high quality water.



2. Press: **I/O** to turn the instrument on.



3. Insert the sample cell into the sample compartment. Close the cover.



4. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading.

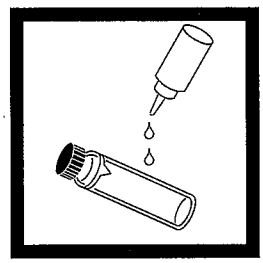
Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*

5. Remove the cell, rotate it slightly and reinsert it into the cell compartment. Close the cover, then press **READ**. Record the cell's position and the displayed reading.

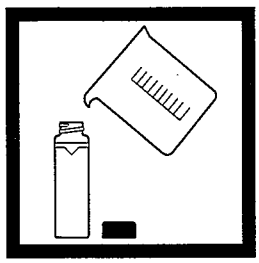
6. Repeat *step 5* until the lowest reading is displayed. Place an orientation mark on the cell's marking band near the top of the cell so the cell can be consistently inserted in the position that yields the lowest reading. When using the cell, always place it in the instrument so the orientation mark aligns with the raised mark on the instrument.

2.3.4 Matching multiple sample cells

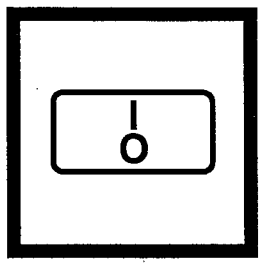
Precise measurements of very low turbidity samples require the cells be optically matched or a single cell be used for all measurements. If more than one cell is used, follow this procedure to match (index) the cells:



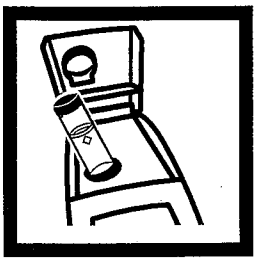
1. Clean and oil the sample cells as instructed in *Section 2.3.1* on page *11* and *Section 2.3.2* on page *11*.



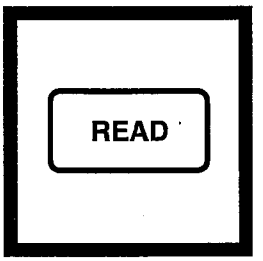
2. Fill the clean sample cells to the line with the same sample.



3. Press: **I/O** to turn the instrument on.



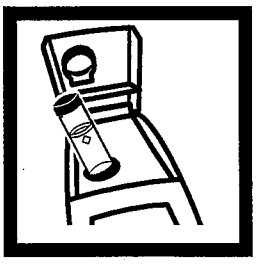
4. Insert the **first** sample cell into the sample compartment and close the cover.



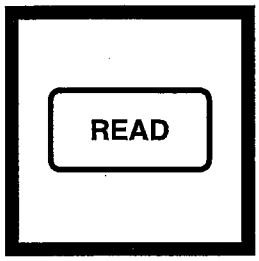
5. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading. Place an orientation mark on the cell's marking band.

Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*



6. Insert the **second** sample cell into the cell compartment and close the cover.



7. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading.

8. Remove the cell, rotate it slightly and reinsert into the cell compartment. Close the cover, then press **READ** again. Record the cell's position and the displayed reading.

9. Repeat *step 8* until the value displayed for the second cell is within 0.01 NTU (or 1%) of the value obtained for the first cell. Place an orientation mark on the second cell's marking band so it is consistently inserted in this position.

Note: *Due to variability in glass, it may not be possible to match all cells.*

10. Repeat *step 6* through *step 9* if matching other sample cells.

2.3.5 Removing Bubbles (Degassing)

Before measurement, removing air and other trapped gasses from the sample is strongly recommended, even if bubbles are not visible. Four degassing methods are commonly used:

1. applying a partial vacuum
2. adding a surfactant
3. using an ultrasonic bath
4. heating the sample

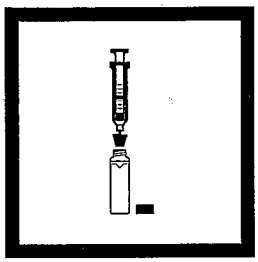
In some cases, more than one method may be necessary for effective bubble removal. For example, use of both a surfactant and ultrasonic bath may be necessary for some severe conditions. Use care with these techniques. If misused, sample turbidity can be altered.

Removing air bubbles by letting the sample stand for a period of time is not recommended. Particulates that cause turbidity may settle and the sample temperature may change. Both conditions may alter sample turbidity, resulting in measurements not representative of the original turbidity.

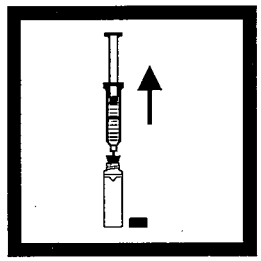
2.3.5.1 Application of vacuum

Apply a vacuum with any convenient, clean, oil-free vacuum source. The vacuum lowers the atmospheric pressure, allowing trapped bubbles to escape into the air above the sample. Vacuum works well with non-viscous samples (such as water) that don't contain volatile components. Applying vacuum to viscous, volatile-containing samples (paint resins) may cause the volatile components to come out of solution and aggravate the bubble problem.

To apply a vacuum, use a sample degassing kit equivalent to Cat No. 43975-00 (Degassing Kit) or 43975-10 (Degassing and Filtration Kit). These kits contain a syringe and rubber stopper for vacuum degassing. An electric or hand-operated pump equivalent to Cat No. 14283-00 or 14697-00, respectively, may also be used.



1. Fill a sample cell to the mark with sample. Insert a #2 single-hole rubber stopper and syringe into the cell. If using a pump, insert a piece of glass tubing into the stopper.



2. **Slowly** apply the vacuum by carefully pulling the plunger upward, then holding it. If using a hand or electric pump, connect the tubing to the vacuum pump with vacuum hose. Apply vacuum until visible gas bubbles disappear. **Slowly** release the vacuum. Remove the vacuum apparatus and cap the cell.

2.3.5.2 Adding a surfactant

Surfactants should be limited to severe problems when other degassing methods are ineffective. Surfactants change the surface tension of the water, which releases trapped gases. Hach recommends a surfactant such as Triton X-100 or the equivalent, Hach Cat No. 14096-37. Put one drop of Triton X-100 in the sample cell before adding sample.

Note: *Any turbidity contributed by surfactant addition is negligible.*

This technique is very effective when the water is super-saturated with air. However, changing the surface tension may accelerate settling of turbidity-causing particles. Mix the sample gently, but thoroughly, and analyze as soon as possible after adding the surfactant. Avoid vigorous mixing as the surfactant may foam. Rinse the sample cells thoroughly between samples to prevent surfactant accumulation.

2.3.5.3 Using an ultrasonic bath

Note: *The time necessary to expel bubbles may vary from a few seconds to a minute or more. To avoid excessive application of ultrasound, a simple procedure can be followed. First, apply ultrasound until all visible bubbles are absent. Then measure the sample turbidity. Apply ultrasound for a short time period and again measure turbidity. Continue for several repetitions, noting the treatment time and turbidity readings. If turbidity begins to increase instead of decrease, the ultrasound waves have probably started to alter the suspended particles. Note the time it takes for this to occur and record it as the maximum time limit for ultrasonic treatment.*

Ultrasonic baths effectively remove gas bubbles from most samples, especially viscous liquids. However, the ultrasonic waves which cause degassing may also alter the characteristics of the particles causing the turbidity. Turbidity depends on the size, shape, composition and refractive index of the suspended particles. Excessive ultrasound application may alter particle size and shape, thus changing sample turbidity. In some cases, ultrasound may aggravate air bubble removal by fracturing the bubbles, making degassing more difficult.

1. Fill a clean sample cell to the line with sample. Leave uncapped.

2. Immerse the cell (1/2 to 2/3 immersed) in an ultrasonic bath and allow it to stand until visible bubbles are expelled.

3. Remove the cell, cap, then thoroughly dry the cell. Apply silicone oil as directed.

2.3.5.4 Application of heat

Whenever possible, avoid using heat to degas samples because heat may change the characteristics of the suspended particles and cause volatile components to come out of solution. Gentle heating may be helpful for degassing some very viscous samples when combined with application of vacuum or ultrasound. If heat is necessary, heat the sample only until degassing occurs. The simplest technique is to prepare a warm water bath and partially immerse the filled sample cell. Use the shortest time necessary for expelling visible bubbles. Cool sample to original sample temperature before taking measurements.

2.3.6 Measuring Overrange Samples

Nephelometric turbidity measurement depends on detection of light scattered from particles suspended in the liquid. If the turbidity is very high, a significant amount of light is blocked or absorbed by the particles and only a small amount of light reaches the detector. This results in a negative interference – the measured turbidity is lower than the actual turbidity. This condition is called "going blind". A multidetector ratioing instrument, such as the Hach 2100P Turbidimeter, minimizes this effect and extends the instrument range. Highly turbid samples may also be diluted, but this should be avoided when possible since it may alter the characteristics of the suspended particles and produce erroneous results.

Light absorbing particles such as activated carbon and highly colored samples may also cause an instrument to "go blind". Dilution may not correct for these interferences. A ratioing instrument will correct for the presence of light absorbing particles and color.

2.3.7 Condensation (fogging)

Condensation may occur on the outside of the sample cell when measuring a cold sample in a warm, humid environment. Condensation interferes with turbidity measurement, so all moisture must be thoroughly wiped off the sample cell before measurement. If fogging recurs, let the sample warm slightly by standing at room temperature or immersing it in a warm bath for a short period. After warming, mix the sample thoroughly before measurement. Allowing samples to warm can alter sample turbidity, so it is best to avoid warming samples before measurement when possible.

2.3.8 Calibration

Turbidimeters must be properly calibrated with a primary standard. Hach recommends formazin or StablCal Stabilized Formazin for calibration. For U.S. Environmental Protection Agency (USEPA) reporting, calibrate at least as often as required by the appropriate regulatory agencies. The frequency of calibration depends on environmental conditions (humidity, temperature) and use. If necessary, calibrate more frequently.

Use secondary standards for periodic calibration checks. Please note that Gelex® standards must be assigned values after StablCal Stabilized Formazin calibration or formazin calibration and before use as secondary standards. Gelex standards must be recalibrated (values assigned) each time the instrument is calibrated with StablCal Stabilized Formazin or formazin. See *Section 3.6* on page *27* for detailed information on the use of StablCal Stabilized Formazin, formazin, and Gelex standards.

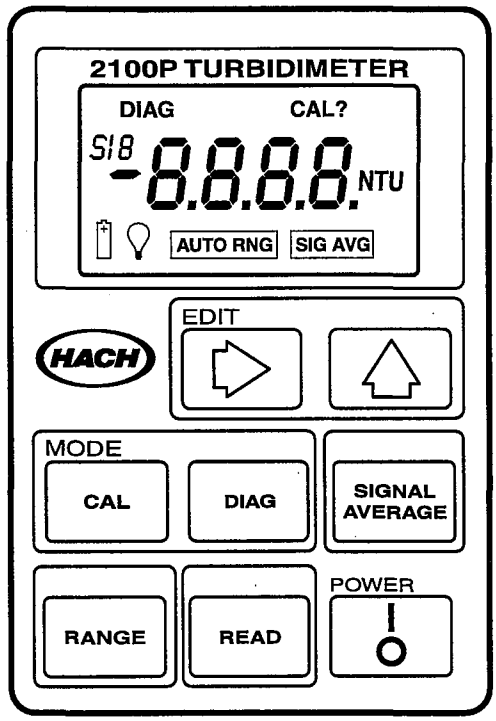
2.3.9 Representative Sampling

A representative sample accurately reflects the true condition of the water source from which the sample was taken. To ensure a representative sample, gently, but thoroughly, mix every sample before aliquots are taken. Do not allow the sample to settle.

When sampling from a tap in a distribution system or treatment plant, allow the water to run for at least five minutes before sampling. When sampling from a stream, reservoir, clarifier, or storage tank, collect at least one liter (1 quart) and thoroughly mix before measurement. If the water source is not uniform, it may be necessary to sample several locations at varying depths and combine the samples into a single, well-mixed composite sample before measurement.

SECTION 3 OPERATION

3.1 Operational Controls and Indicators



Key	Description
I O	Power key to turn instrument on and off. If no keys are pressed for 5.5 minutes, the instrument turns off automatically.
READ	Depressed to perform a measurement. To conserve battery power, the lamp turns on only when **READ** is depressed. A reading is displayed about 12 seconds after the key is depressed. During the delay, a flashing **NTU** is displayed. After the reading is displayed, the lamp turns off and the reading continues to be displayed. Continuous readings may be done by holding this key if **not** in the Signal Averaging mode. After the initial delay, the reading is updated every 1.2 seconds.
CAL	Used to perform a calibration or review calibration data. Also terminates a calibration or calibration review and returns to the 2100P measurement mode.

Key	Description
	Edits a flashing digit in the calibration mode or sequences through the calibration standards (S0,S1, S2, S3) or diagnostic menu.
	Used to move the editing cursor to the digits being edited in the calibration mode or initiate editing of a standard value.
SIGNAL AVERAGE	Turns the signal averaging function on or off.
DIAG	Selects the diagnostic mode.
RANGE	Selects Auto Range or Manual Range (one of three manual modes).

Display Icon	Description
DIAG	Turns on after the **DIAG** is pressed to access the diagnostic mode. A number displayed under the **DIAG** icon (1-9) indicates which diagnostic function is active. See *Section 5.1* on page *57* for more information on diagnostic codes.
CAL	Turns on after the **CAL** key is pressed to access the calibration mode and remains on during the calibration.
CAL?	Appears after calibration if a value entered during calibration is outside an acceptable range. May indicate an operator error or possible instrument malfunction. Flashing **CAL?** indicates the default calibration coefficients are being used (even after a user-calibration has been done) or that no calibration data is currently stored.
S__	Displayed during calibration. The S is followed by a number to indicate which standard value is currently being edited or displayed. Flashing number is prompting user for measurement of **S0**, **S1**, **S2** or **S3** to establish a calibration. Steady number identifies which standard's value is being displayed.
	Flashes when the battery voltage drops to 4.4 volts as an indication to change batteries. At <4.0 volts, the instrument automatically shuts off.
	The lamp symbol is constantly on when the lamp is on and flashes after a reading if a marginal light level reaches the transmitted light detector. A flashing icon indicates the sample may be too turbid (not within measurement range) and needs dilution or the lamp needs replacing.

Display Icon	Description
SIGNAL AVERAGE	Indicates the signal averaging mode is on. The icon turns off if signal averaging is not selected.
AUTO RNG	Indicates instrument is in automatic range mode. The icon turns off when manual range mode is selected.
8888	The 4-digit display is active when the instrument is on (measurements are displayed to three digits). After the **READ** key is pressed - - - - is displayed during wait periods.
NTU	Identifies the measurement units- Nephelometric Turbidity Units. This icon is active during measurements and in the calibration mode.

3.2 Using the Read Key

To preserve battery power and prolong lamp life, the lamp turns on only after the **READ** key is pressed. Pressing the key turns the instrument lamp on; after about 12 seconds, the lamp turns off, but the measurement value continues to be displayed. After the first measurement, a four-second recovery time occurs before another measurement can be started. If **READ** is pressed during the recovery time, the display will begin flashing, but the lamp will not turn on until the full four seconds have passed. If no other key strokes occur within 5.5 minutes, the instrument turns off.

3.2.1 Continuous Reading

The instrument cannot be used in continuous read mode if the Signal Averaging mode is on.

The instrument will perform continuous readings if the **READ** key is pressed and held. As long as the key is held, the lamp remains on and the display is updated every 1.2 seconds.

3.3 Using the Signal Averaging Key

The signal averaging mode compensates for reading fluctuations caused by drifting of sample particles through the light path. Signal averaging is turned on or off by pressing the **SIGNAL AVERAGE** key. The **SIG AVG** icon is displayed when signal averaging is on.

Signal averaging measures and averages ten measurements while displaying intermediate results. The initial value is displayed after about

11 seconds and the display is updated every 1.2 seconds until all ten measurements are taken (about 22 seconds). After 22 seconds, the lamp turns off, but the final measured turbidity value continues to be displayed until another key is pressed.

When signal averaging is off, the instrument takes three measurements, the microprocessor averages them, then displays the average. If the **READ** key is held during measurement, the initial value is displayed in 12 seconds and is updated every 1.2 seconds as long as the **READ** key is held.

When the instrument is turned on, the instrument defaults to the signal averaging mode which was used during the last measurement.

3.4 Using the Range Selection Key

As shipped, the instrument defaults to automatic range mode. The first time the **RANGE** key is pressed, the instrument goes into manual range mode. The second, third, and fourth key strokes put the instrument in the 0.00-9.99, 10 to 99.9 or 100-1000 NTU range, respectively. Another key stroke brings the selection back to automatic range mode. When the automatic range mode is selected, the **AUTO RNG** icon is displayed. Range selection can be done any time except when a measurement or calibration is in progress.

When the instrument is turned on, the instrument defaults to the range mode and measurement range which was used during the last measurement.

3.5 Restoring the Default Calibration

To restore and use the default calibration, turn the instrument off. Press and hold **DIAG**, then press and release **I/O**. Release **DIAG** when the software version number disappears from the display. (For models with serial number less than 920300000800, **2100** disappears). This clears any user-entered calibration from memory; the 2100P will use the default calibration for measurement. **CAL?** will appear and continue to flash until a user-entered calibration is successfully completed.

For best results, a user-entered calibration should be done every three months.

3.6 Calibration

Calibration of the 2100P Turbidimeter is based on formazin, the primary standard for turbidity. The instrument's electronic and optical design provide long-term stability and minimize the need for frequent calibration. The two-detector ratioing system compensates for most fluctuations in lamp output. **A formazin recalibration should be performed at least once every three months,** more often if experience indicates the need. When calibration is necessary, use a primary standard such as StablCal™ Stabilized Standards or formazin standards.

Hach Company only recommends the use of StablCal® Stabilized Formazin or formazin standards for the calibration of Hach turbidimeters. Hach Company cannot guarantee the performance of the turbidimeter if calibrated with co-polymer styrene divinylbenzene beads or other suspensions.

3.6.1 StablCal Stabilized Formazin Standards*

Most consistent results will be achieved with the use of StablCal Stabilized Formazin Standards for calibration. Refer to *Section 3.6.1.2* and *Section 3.6.1.3* for information on preparing the standards for use.

Note: *Hach StablCal Stabilized Formazin in 20-, 100-, and 800-NTU values is packaged in convenient sets for calibration of the 2100P Turbidimeter. The set may be ordered in 500-mL size bottles by specifying Cat. No. 26594-00, in 100-mL size bottles by specifying Cat. No. 26594-10 or in sealed vials by ordering Cat. No. 26594-05. (See OPTIONAL ACCESSORIES AND REAGENTS on page 61.)*

3.6.1.1 Storing and Handling StablCal Stabilized Standards

For optimum results when using StablCal Stabilized Standards, adhere to the following recommendations:

- Do not transfer the standard to another container for storage.
- Do not return standard from the sample cell back into the its original container. Standard contamination will result.
- Store standards between 0 and 25 °C.

* StablCal Stabilized Formazin is cited as a primary standard in Hach Method 8195, an acceptable version of USEPA Method 180.1.

- For long-term storage, refrigeration at 5 °c is recommended. Do not store above 25 °C.
- Allow the standard to acclimate to ambient instrument conditions before use (not to exceed 40 °C).
- Store away from direct sunlight. Store vials in their respective kit or shipping box with the cover in place.

3.6.1.2 Preparing Bulk StablCal Stabilized Standards

Bulk standards that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension into its original particle size. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*.

Important Note: These instructions do not apply to <0.1-NTU* StablCal Standards; <0.1NTU StablCal Standards should not be shaken or inverted.

1. Shake the standard vigorously for 2-3 minutes to resuspend any particles.
2. Allow the standard to stand undisturbed for 5 minutes.
3. Gently invert the bottle of StablCal 5 to 7 times.
4. Prepare the sample cell for measurement using traditional preparation techniques. This usually consists of oiling the sample cell (see*Section 2.3.2* on page *11*) and marking the cell to maintain the same orientation in the sample cell compartment (see *Section 2.3.3* on page *12*). This step will eliminate any optical variations in the sample cell.
5. Rinse the sample cell at least one time with the standard and discard the rinse.
6. Immediately fill the sample cell with the standard. Cap the sample cell and let it stand for one minute. The standard is now ready for use in the calibration procedure, *Section 3.6.3*.

* Used in place of the dilution water standard when performing a calibration.

3.6.1.3 Preparing StablCal Stabilized Standards in Sealed Vials

Sealed vials that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension into its original particle size. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*

Important Note: These instructions do not apply to <0.1-NTU* StablCal Standards; <0.1NTU StablCal Standards should not be shaken or inverted.

1. Shake the standard vigorously for 2-3 minutes to resuspend any particles.
2. Allow the standard to stand undisturbed for 5 minutes.
3. Gently invert the vial of StablCal 5 to 7 times.
4. Prepare the vial for measurement using traditional preparation techniques. This usually consists of oiling the vial (see *Section 2.3.2* on page *11*) and marking the vial to maintain the same orientation in the sample cell compartment (see *Section 2.3.3* on page *12*). This step will eliminate any optical variations in the sample vial.
5. Let the vial stand for one minute. The standard is now ready for use in the calibration procedure, *Section 3.6.3*.

3.6.2 Formazin Primary Standards

Perform the procedure in *Section 3.6.2.1* to prepare a 4000-NTU standard. Alternately, order a 4000-NTU stock solution from Hach by specifying Cat. 2461-49. Prepare the dilutions from the 4000-NTU stock solution by following the instructions in *Section 3.6.2.4*.

3.6.2.1 Preparing Formazin Stock Solution

Dilute formazin standard solutions from a 4000 NTU stock solution equivalent to Hach Cat. No. 2461-49. The prepared stock solution is stable for up to one year when properly prepared. An alternative to purchasing the 4000 NTU stock solution is preparing a stock solution as follows:

1. Dissolve 5.000 grams of reagent grade hydrazine sulfate ($N_2H_4 \cdot H_2SO_4$) in 400 mL of distilled water.

2. Dissolve 50.000 grams of pure hexamethylenetetramine in 400 mL of distilled water.

3. Pour the two solutions into a 1000-mL volumetric flask and dilute to the mark with distilled water.

4. Let the solution stand for 48 hours at 25 °C (77 °F) to develop the 4000-NTU stock suspension. The standing temperature is critical for correct formation of formazin polymers.

5. Mix the 4000 NTU suspension for at least ten minutes before use. Then it can be diluted with distilled or demineralized water to achieve a solution of the desired NTU value.

Instead of diluting a formazin stock solution, StablCal Stabilized Formazin Standards may be used. Order the StablCal Calibration Set for the 2100P Turbidimeter, Cat.No. 26594-00 (500-mL bottles), Cat. No. 26594-10 (100 mL bottles), or Cat. No. 26594-05 (sealed vials). (See *OPTIONAL ACCESSORIES AND REAGENTS* on page *61*.)

3.6.2.2 Correcting for Turbidity of Dilution Water

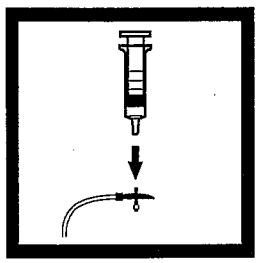
The 2100P Turbidimeter automatically compensates for turbidity contributed by dilution water when calculating the true value of the lowest formazin standard. Use high quality distilled or deionized water less than 0.5 NTU. The instrument will display E 1 after calibration if the dilution water turbidity is greater than 0.5 NTU. In this case, prepare the water as directed below.

The value of the dilution water can be arbitrarily forced to zero (see calibration procedure). This is not recommended for most applications and, if done, should be done only if the dilution water turbidity is less than 0.2 NTU.

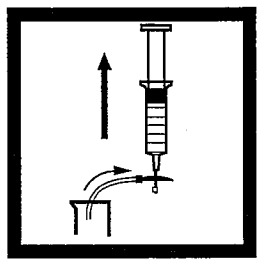
3.6.2.3 Preparing Dilution Water

Note: *Use the same dilution water for all dilutions and the sample blank.*

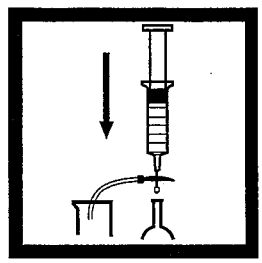
Collect at least 1000 mL of high quality dilution water (distilled or deionized water). The 2100P Turbidimeter, as received from the factory, is precalibrated and may be used to check the dilution water turbidity. If the turbidity is greater than 0.5 NTU, filter the water with the Sample Filtration and Degassing Kit (Cat. No. 43975-10) or equivalent. When measuring low range turbidity, clean all glassware with 1:1 hydrochloric acid and rinse several times with dilution water. If the glassware is not used immediately, use stoppers to prevent contamination from small particles.



1. Attach the syringe to the 3-way valve by gently twisting the square end into the syringe tip. Attach the connector, tubing and a 0.2 micron filter (clear part faces syringe) as shown. Be sure the connections are tight.



2. Fill a beaker or container with the water to be filtered. Insert the tubing into the container. Slowly draw the water into the syringe by pulling up on the syringe plunger.



3. Draw about 50 mL of sample into the syringe. Slowly push on the plunger to force the water through the filter and into a graduated cylinder or volumetric flask. Repeat Steps 2 and 3 until the desired amount of water is obtained.

Note: *As the filter clogs, it gets more difficult to push water through it. At this point, discard the filter and attach a new filter. Replacement filters are available in packages of 10 (Cat. No. 23238-10).*

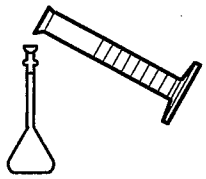
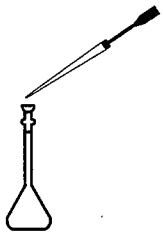
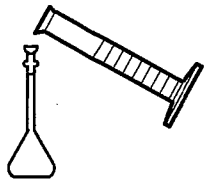
3.6.2.4 Preparing Formazin Dilutions (Factory recommended)

Hach Company recommends using 20, 100, and 800 NTU formazin standards for calibrating the 2100P Turbidimeter. Dilutions with other NTU values can be prepared and used (see *Section 3.6.3.1* on page *38*). If problems occur when using alternate solutions, use the dilutions specified here.

Prepare all formazin dilutions immediately before use and discard after calibration. The 4000 NTU solution is stable for up to a year, but dilutions deteriorate more rapidly. Use the same high quality water (turbidity <0.5 NTU) for the dilutions and the blank.

Preparing the 20, 100 and 800 NTU standards

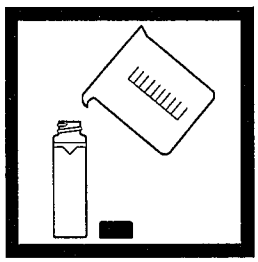
Table 1 Formazin Standard Preparation

	Step 1	Step 2	Step 3
Standards			
20 NTU	Add 100 mL of dilution water to a clean **200-mL** class A volumetric flask.	With a TenSette* pipet, add 1.00 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water. Stopper and mix.
100 NTU	Add 100 mL of dilution water to a clean **200-mL** class A volumetric flask.	With a TenSette pipet, add 5.00 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water. Stopper and mix.
800 NTU	Add 50 mL of dilution water to a clean **100-mL** class A volumetric flask.	With a TenSette pipet, add 20.00 mL of well-mixed 4000 NTU Formazin stock solution to the 100-mL flask.	Dilute to the mark with dilution water. Stopper and mix.

* A class A volumetric pipet may be used in place of a TenSette Pipet.

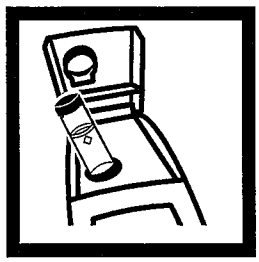
3.6.3 Calibrating the Turbidimeter

Note: *For best accuracy use the same sample cell or four matched sample cells for all measurements during calibration. Always insert the cell so the orientation mark placed on the cell during the matching procedure is correctly aligned. (See Section 2.3.4 on page 15 for matching sample cells).**



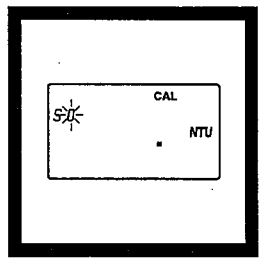
1. Rinse a clean sample cell with dilution water several times. Then fill the cell to the line (about 15 mL) with dilution water or use StablCal <0.1 NTU standard.

Note: *The same dilution water used for preparing the standards must be used in this step.*



2. Insert the sample cell in the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid. Press **I/O**.

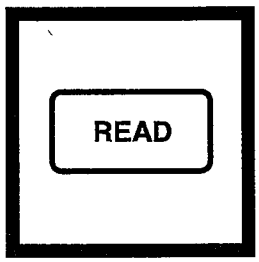
Note: *Choose signal average mode option (on or off) before pressing* **CAL** *– the* **SIGNAL AVERAGE** *key is not functional in calibration mode.*



3. Press: **CAL**

The **CAL** and **S0** icons will be displayed (the **0** will flash). The 4-digit display will show the value of the S0 standard for the previous calibration. If the blank value was forced to 0.0, the display will be blank (as shown). Press →to get a numerical display.

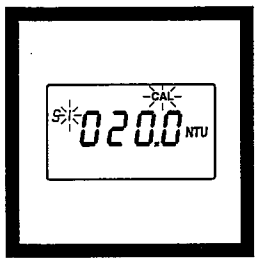
Hach Company only recommends the use of StablCal® Stabilized Formazin or formazin standards for the calibration of Hach turbidimeters. Hach Company cannot guarantee the performance of the turbidimeter if calibrated with co-polymer styrene divinylbenzene beads or other suspensions.



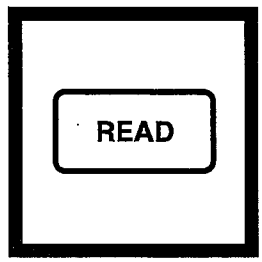
4. Press: **READ**

The instrument will count from 60 to 0, (67 to 0 if signal average is on), read the blank and use it to calculate a correction factor for the 20 NTU standard measurement. If the dilution water is ≥ 0.5 NTU, E 1 will appear when the calibration is calculated (See *Section 3.6.2.3* on page *31* for more dilution water information). The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

Note: *The turbidity of the dilution water can be "forced" to zero by pressing →rather than reading the dilution water. The display will show* **S0 NTU** *and the ↑ key must be pressed to continue with the next standard.*



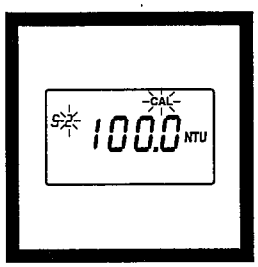
5. The display will show the **S1** (with the 1 flashing) and **20 NTU** or the value of the S1 standard for the previous calibration. If the value is incorrect, edit the value by pressing the →key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 20 NTU StablCal Standard or 20 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.



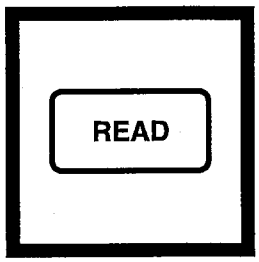
6. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

*

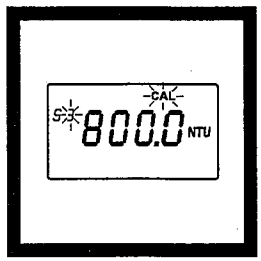


7. The display will show the **S2** (with the 2 flashing) and **100 NTU** or the value of the S2 standard for the previous calibration. If the value is incorrect, edit the value by pressing the →key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 100 NTU StablCal Standard or 100 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.

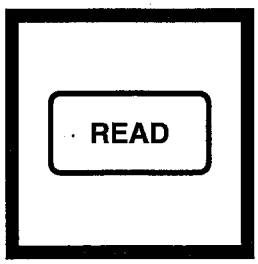


8. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then, the display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

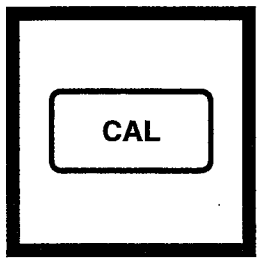


9. The display will show the **S3** (with the 3 flashing) and **800 NTU** or the value of the S3 standard for the previous calibration. If the value is incorrect, edit the value by pressing the →key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 800 NTU StablCal Standard or 800 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.



10. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then the display will increment back to the S0 display. Remove the sample cell from the cell compartment.



11. Press: **CAL** to accept the calibration. The instrument will return to measurement mode automatically.

Note: *Pressing* **CAL** *completes the calculation of the calibration coefficients. If calibration errors occurred during calibration, error messages will appear after* **CAL** *is pressed. If* **E 1** *or* **E 2** *appear, check the standard preparation and review the calibration; repeat the calibration if necessary. If* **CAL?** *appears, an error may have occurred during calibration. If* **CAL?** *is flashing, the instrument is using the default calibration.*

NOTES

- If the **I/O** key is pressed during calibration, the new calibration data is lost and the old calibration will be used for measurements. Once in calibration mode, only the **READ**, **I/O**, ↑ , and →keys function. Signal averaging and range mode must be selected before entering the calibration mode.

- If **E 1** or **E 2** are displayed, an error occurred during calibration. Check the standard preparation and review the calibration; repeat the calibration if necessary. Press **DIAG** to cancel the error message (**E 1** or **E 2**). To continue without repeating the calibration, press **I/O** twice to restore the previous calibration. If **CAL?** is displayed, an error may have occurred during calibration. The previous calibration may not be restored. Either recalibrate or use the calibration as is.

- To review a calibration, press **CAL** and then ↑ to view the calibration standard values. As long as **READ** is never pressed and **CAL** is not flashing, the calibration will not be updated. Press **CAL** again to return to the measurement mode.

3.6.3.1 Preparing User-selected Formazin Dilutions

The formazin solutions should span the entire range of the instrument. Hach recommends preparing three standards:

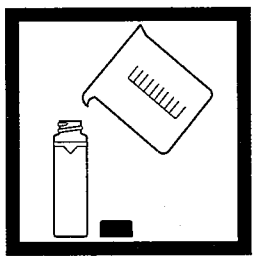
1. 10 to 30 NTU
2. 90 to 110 NTU
3. 700 to 900 NTU

The standards must have a difference of at least 60 NTU.
In addition, a blank made from the dilution water should be prepared.

Prepare the formazin standard solutions from the well mixed 4000 NTU stock solution as specified in *Section 3.6.2.4* on page *32* and dilution water as specified in *Section 3.6.2.2* and *Section 3.6.2.3* on page *31*. Make the standards **immediately** before use and discard them after calibration is done.

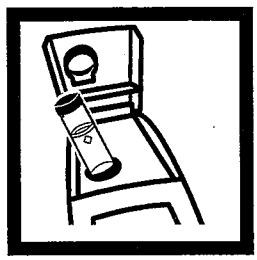
3.6.3.2 Calibrating with User-selected Standards

Note: *For best accuracy use the same sample cell or four matched sample cells for all measurements during calibration. Always insert the sample cell with the same orientation.*



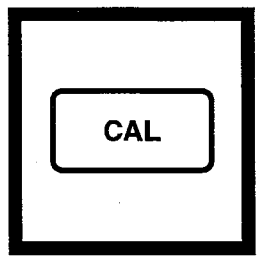
1. Fill a clean sample cell to the line (about 15 mL) with dilution water.

Note: *The same dilution water used for preparing the standards must be used in this step.*



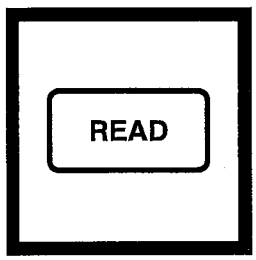
2. Insert the sample cell into the cell compartment and close the lid. Press **I/O**.

Note: *Choose signal average mode option (on or off) before pressing* **CAL** *– the* **SIGNAL AVERAGE** *key is not functional in calibration mode.*



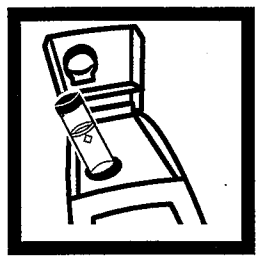
3. Press: **CAL**.

The **CAL** and **S0** icons will appear (the **0** will flash). The 4-digit display will show the value of the S0 standard for the previous calibration.



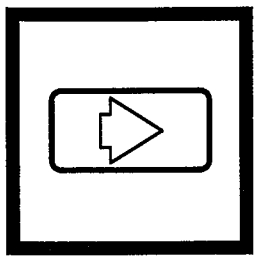
4. Press: **READ**.

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the blank and use it to calculate a correction factor for the lowest standard. If the dilution water is ≥0.5 NTU, **E 1** will appear (see *Section 3.6.2.3* on page *31* for more dilution water information). The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

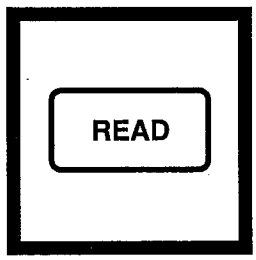


5. Thoroughly mix the 10 to 30 NTU range standard, then fill a clean sample cell to the line with the standard. Insert the sample cell into the cell compartment

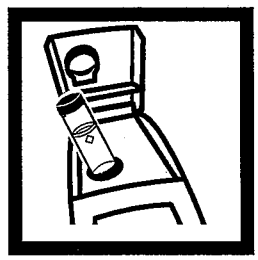
6. The display will show the **S1** icon (with the **1** flashing) and **20 NTU** or the value of the S1 standard for the previous calibration.



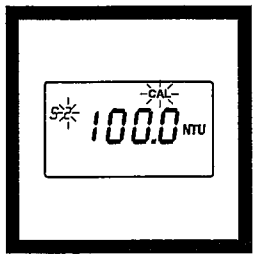
7. Edit the standard concentration by pressing →The **1** will stop flashing and the left digit in the display will flash. Press ↑ to scroll the digit up to the appropriate number. Press →again to move the cursor to the next digit and edit it in the same manner.



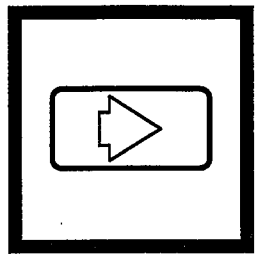
8. When all the digits show the appropriate value, press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.



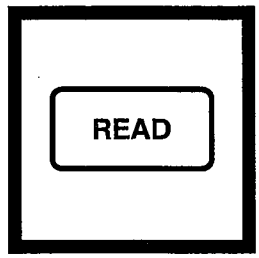
9. Thoroughly mix the 90 to 110 NTU standard, then fill a clean sample cell to the line with the standard. Insert the cell into the cell compartment.



10. The display will show the **S2** icon (with the **2** flashing) and **100 NTU** or the value of the S2 standard for the previous calibration.

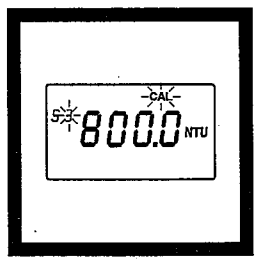


11. Edit the standard concentration by pressing → The **2** will stop flashing and the left digit in the display will flash. Press ↑ to scroll the digit up to the appropriate number. Press →again to move the cursor to the next digit and edit it in the same manner.

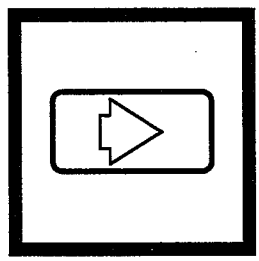


12. When all the digits show the appropriate value, press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Remove the sample cell from the cell compartment.

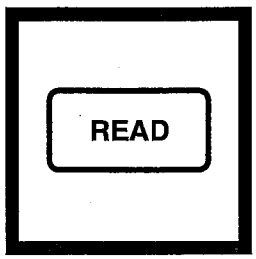
13. Thoroughly mix the 700 to 900 NTU standard, then fill a clean sample cell to the line with the standard. Insert the cell into the cell compartment.



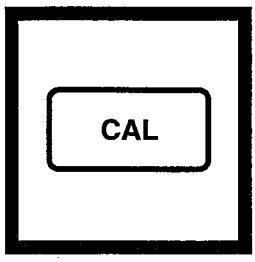
14. The display will show the **S3** icon (with the **3** flashing) and **800 NTU** or the value of the S3 standard for the previous



15. Edit the standard concentration by pressing → The **3** will stop flashing and the left digit in the display will flash. Press ↑ to scroll the digit up to the appropriate number. Press →again to move the cursor to the next digit and edit it in the same manner.



16. When all the digits show the appropriate value, press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The instrument will increment back to **S0**. Remove the sample cell from the cell compartment.



17. Press: **CAL**.

The instrument will store the new calibration data and return the instrument to the measurement mode. It will use the new calibration to calculate turbidity for subsequent measurements.

Note: *Pressing* **CAL** *completes the calculation of the calibration coefficients. If calibration errors occurred during calibration, error messages will appear after* **CAL** *is pressed. If* **E 1** *or* **E 2** *appear, check the standard preparation and review the calibration; repeat the calibration if necessary. If* **CAL?** *appears, an error may have occurred during calibration. If* **CAL?** *is flashing, the instrument is using the default calibration.*

NOTES

- If the **I/O** key is pressed during calibration, the new calibration data is lost and the old calibration will be used for measurements. Once in calibration mode, only the **READ**, **I/O**, ↑ , and →✳keys function. Signal averaging and range mode must be selected before entering the calibration mode.

- If **E 1** or **E 2** are displayed, an error occurred during calibration. Check the standard preparation and review the calibration; repeat the calibration if necessary. If the error messages recur, calibrate using the factory specified standards, *Section 3.6.2.4* on page *32* and *Section 3.6.3* on page *34*. Press **DIAG** to cancel the error message (**E 1** or **E 2**). To continue without repeating the calibration, press **I/O** twice to restore the previous calibration. If **CAL?** is displayed, an error may have occurred during calibration. The previous calibration may not be restored. Either recalibrate or use the calibration as is.

- To review a calibration, press **CAL** and then only ↑ to view the calibration standard values. As long as **READ** is never pressed and **CAL** isn't flashing, the calibration will not be updated. Press **CAL** again to return to the measurement mode.

3.6.4 Using Gelex® Secondary Turbidity Standards

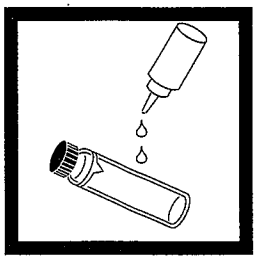
Note: *Store Gelex standards at room temperature. Do not allow to freeze or exceed 50 °C.*

The instrument comes with Gelex Secondary Standards which are particulate suspensions similar to formazin primary standards in light scattering characteristics. NTU values on the Gelex standards indicate the range for which they should be used. Due to minor variations in glass and individual instrument optical systems, the true value of the Gelex standards must be determined against formazin in the same instrument they will be used with for later calibration checks.

3.6.4.1 Assigning Values to Gelex Standards

1. Calibrate the instrument with formazin.

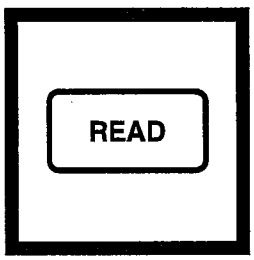
2. Select automatic range mode using the **RANGE** key.



3. Thoroughly clean the outside of the Gelex vials and apply a thin coating of silicone oil.

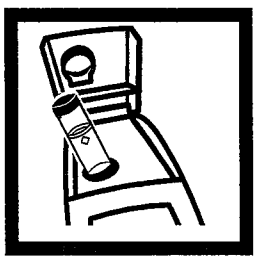
4. Place the 0-10 NTU Gelex standard in the cell compartment so the diamond on the vial aligns with the orientation mark on the instrument. Close the sample lid.

Note: *Correct cell orientation is essential to obtain accurate Gelex values. Always orient the cell so the diamond mark aligns with the orientation mark on the instrument.*



5. Press: **READ**.

Record the displayed value, remove the vial from the instrument and mark the value on the band near the top of the vial.



6. Repeat *step 3* through *step 5* for the other Gelex standards, being careful to orient the cells properly.

Re-assign with every formazin calibration

7. Re-assign values to the Gelex standards each time the instrument is calibrated with formazin.

3.6.4.2 Routine Calibration Check With Gelex Standards

The 2100P Turbidimeter does not require standardization before every measurement as some turbidimeters do. Periodically, as experience dictates, check the instrument calibration using the appropriate Gelex Secondary Standard. Be sure the Gelex standards are aligned correctly when inserting them (diamond aligns with orientation mark). If the reading is not within 5% of the previously established value, recalibrate the instrument with StablCal Stabilized Formazin Primary Standard or formazin primary standard (*Section 3.6.3* on page *34*).

MAINTENANCE

Some of the following manual sections contain information in the form of warnings, cautions and notes that require special attention. Read and follow these instructions carefully to avoid personal injury and damage to the instrument. Only personnel qualified to do so, should conduct the maintenance tasks described in this portion of the manual.

Certains des chapitres suivants de ce mode d'emploi contiennent des informations sous la forme d'avertissements, messages de prudence et notes qui demandent une attention particulière. Lire et suivre ces instructions attentivement pour éviter les risques de blessures des personnes et de détoriation de l'appareil. Les tâches d'entretien décrites dans cette partie du mode d'emploi doivent être seulement effectuées par le personnel qualifié pour le faire.

Algunos de los capítulos del manual que presentamos contienen muy importante información en forma de alertas, notas y precauciones a tomar. Lea y siga cuidadosamente estas instrucciones a fin de evitar accidentes personales y daños al instrumento. Las tareas de mantenimiento descritas en la presente sección deberán ser efectuadas únicamente por personas debidamente cualificadas.

Einige der folgenden Abschnitte dieses Handbuchs enthalten Informationen in Form von Warnungen, Vorsichtsmaßnahmen oder Anmerkungen, die besonders beachtet werden müssen. Lesen und befolgen Sie diese Instruktionen aufmerksam, um Verletzungen von Personen oder Schäden am Gerät zu vyyyermeiden. In diesem Abschnitt beschriebene Wartungsaufgaben dürfen nur von qualifiziertem Personal durchgeführt werden.

Algumas das seguintes secções do manual contêm informações em forma de advertências, precauções e notas que requerem especial atenção. Leia e siga atentamente as presentes instruções para evitar ferimentos pessoais e não danificar o instrumento. As tarefas de manutenção descritas nesta parte do manual só poderão ser executadas por pessoal qualificado.

SECTION 4 MAINTENANCE

4.1 Cleaning

Keep the turbidimeter and accessories as clean as possible and store the instrument in the carrying case when not in use. Avoid prolonged exposure to sunlight and ultraviolet light. Wipe spills up promptly. Wash sample cells with non-abrasive laboratory detergent, rinse with distilled or demineralized water, and air dry. Avoid scratching the cells and wipe all moisture and fingerprints off the cells before inserting them into the instrument. Failure to do so can give inaccurate readings. See *Section 2.3.1* on page *11* for more information about sample cell care.

4.2 Battery Replacement

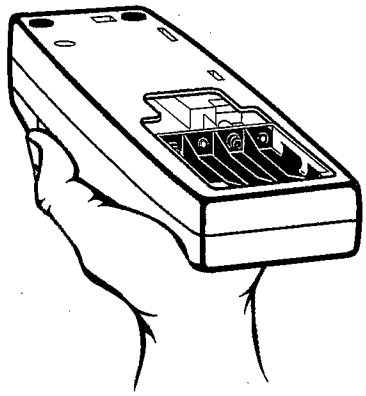
AA alkaline cells typically last for about 300 tests with the signal averaging mode off, about 180 tests if signal averaging is used. The "battery" icon flashes when battery replacement is needed. Refer to *Section 1.4.2* on page *5* for battery installation instructions. If the batteries are changed within 30 seconds, the instrument retains the latest range and signal average selections. If it takes more than 30 seconds, the instrument uses the default settings.

If, after changing batteries, the instrument will not turn off or on and the batteries are good, remove the batteries and reinstall them. If the instrument still won't function, contact Hach Service or the nearest authorized dealer.

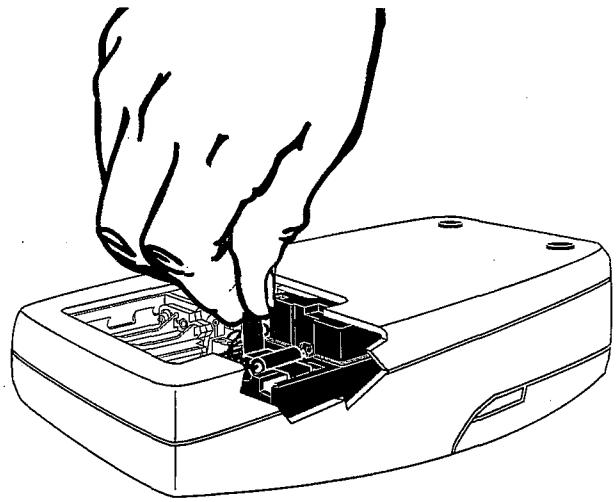
4.3 Lamp Replacement

The procedure below explains lamp installation and electrical connections. Use a small screwdriver to remove and install the lamp leads in the terminal block. The instrument requires calibration after lamp replacement.

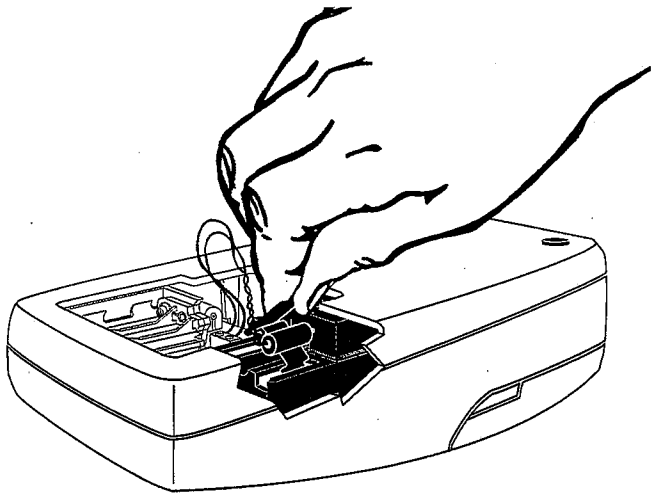
1. Orient the instrument so it is upside down and the top faces away from you. Remove the battery cover and at least one battery.



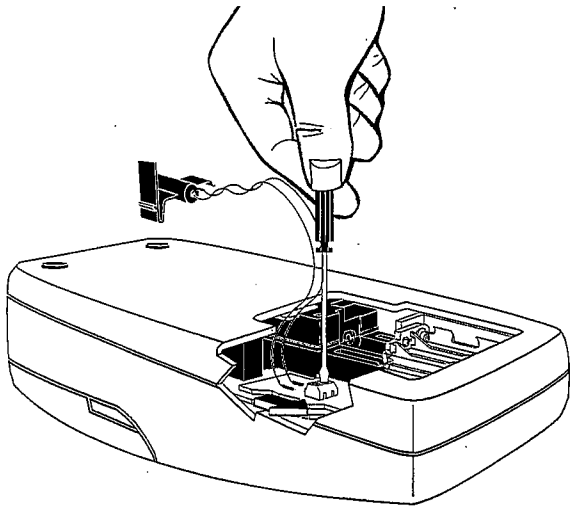
2. Remove the lamp assembly by grasping the tab on the left side of the assembly. Firmly, but gently, slide the assembly towards the rear of the instrument.



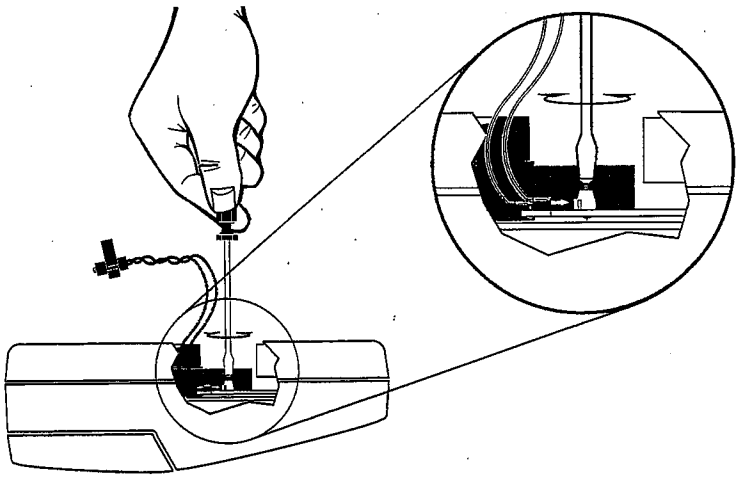
3. Rotate the tab towards the nearest outside edge. The assembly should release and slip out easily.



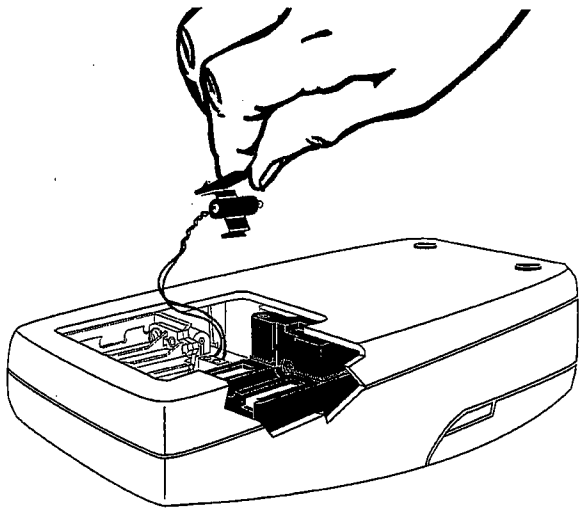
4. Back the terminal block screws **partially** out (1 to 2 turns) and remove the old lamp leads.



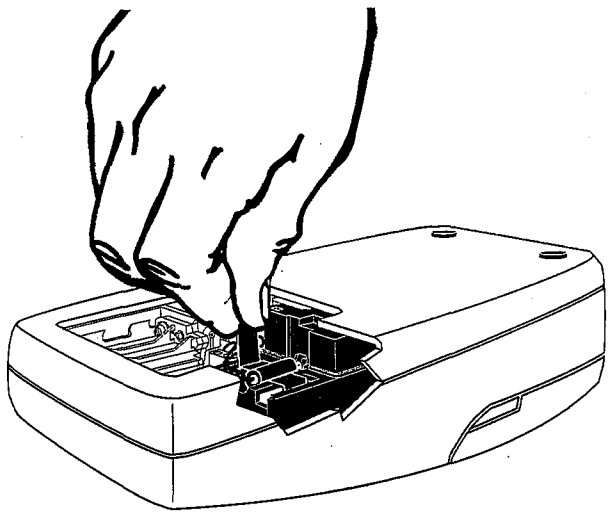
5. Gently bend the wires of the new lamp assembly into an "L" shape so they fit easily into the housing. Insert the leads into the terminal screws and tighten with clockwise turns. Gently tug on the wires to make sure they are connected to the terminal block.



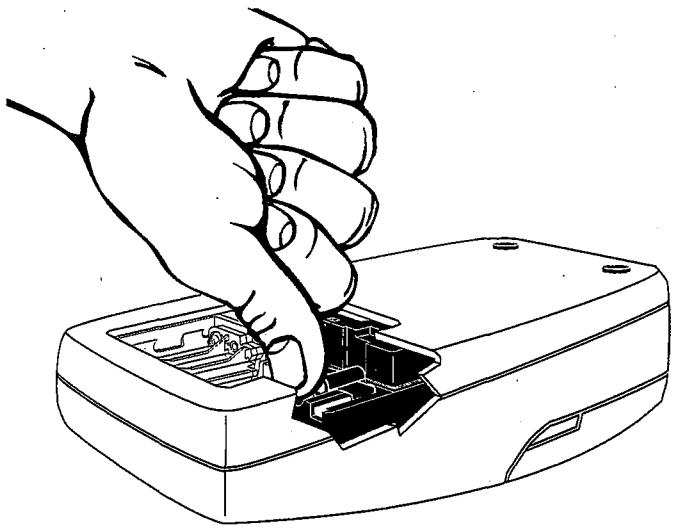
6. Hold the new lamp assembly by the tab with the lamp facing the tope (keyboard) of the instrument. Slide the small catch on the other side of the assembly into the black plastic slot (towards the nearest edge of the instrument).



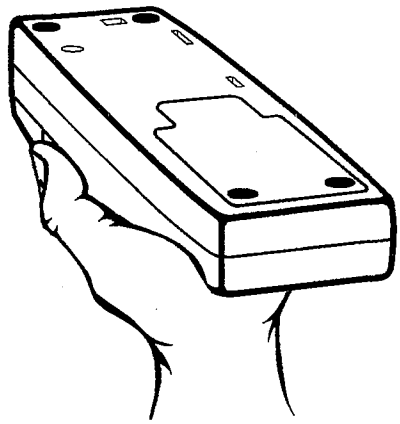
7. Snap the U-shaped bottom of the tab into the slot on the left side of the black plastic that holds the lamp assembly.



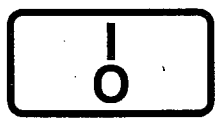
8. With your thumb, firmly slide the assembly forward until it stops. Again, push firmly against the tab to make sure the lamp is seated correctly.



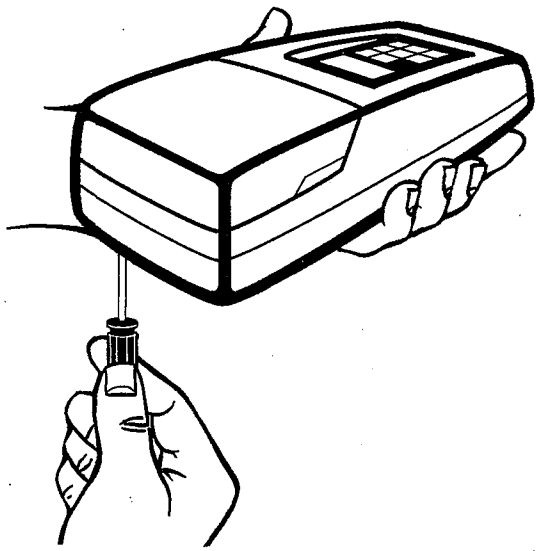
9. Replace the battery(s) and battery cover.



10. Insert the 800 NTU formazin standard into the sample cell. Press and hold **READ**. Then press **I/O**. Release the **READ** key after the software version number disappears from the display (for models with serial numbers less than 920300000800, **2100** disappears).



11. Adjust the scattered light amplifier output by inserting a small flat-bladed screwdriver into the trimpot hole (located on bottom). Adjust the display to read 2.5 ± 0.3 volts (2.0 volts for models that display **2100** when turned on).



12. Press **I/O** to exit gain adjust mode.

13. Perform a formazin calibration according to *Section 3.6.3* on page *34* or *Section 3.6.3.1* on page *38*.

SECTION 5 TROUBLESHOOTING

5.1 Using the Diagnostic Functions Key

Enter the diagnostic mode by pressing the **DIAG** key. Exit this mode at any time by pressing the key again. The diagnostic mode allows access to information about instrument function which may be useful for servicing and troubleshooting.

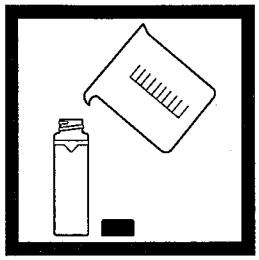
5.1.1 Basic Diagnostic Codes

The diagnostic codes are:

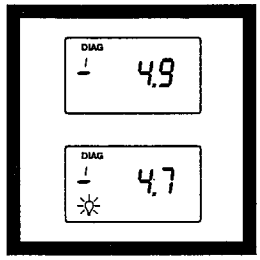
Code	Description
1	Checks the battery voltage with the lamp on, then with the lamp off. This is a dual diagnostic code.
2	Displays calibration coefficient a_0
3	Displays calibration coefficient a_1
4	Displays calibration coefficient b_0
5	Displays calibration coefficient b_1
6	Displays the lamp voltage (about 3 volts)
7	Displays the dark voltage of the transmitted light detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.
8	Displays the high gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.*
9	Displays the low gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.

* Samples with turbidity >10 NTU may display - - - for the lamp-on amplifier voltage.

5.2 The Diagnostic Procedure

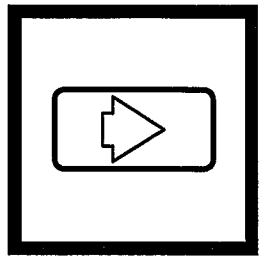


1. Fill a clean sample cell to the line with clear water, cap the cell and place it in the cell compartment. Press the **READ** key and wait until the reading is finished.

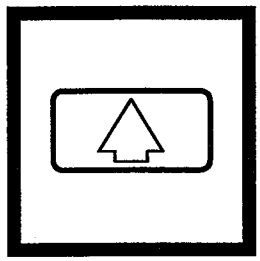


2. Press: **DIAG**

The **DIAG** icon will turn on and **1** will be displayed below the icon. The instrument will measure the battery voltage with the lamp off and display the result in volts (V). Then the lamp icon will turn on and the instrument will measure the voltage with the lamp on. The value is briefly displayed before defaulting to the lamp-off reading. To repeat the measurement, press **READ**.



3. To continuously display the lamp-on voltage, press → The lamp icon will flash. Press →to turn the lamp icon off (the lamp is not on during this display).



4. Press the ↑ key to scroll through the other diagnostics. Each press of the key increments the digit in the small numerical display below the **DIAG** icon and the result of the diagnostic measurement is then displayed. Each press of the **READ** key updates the value. For measurements made with the lamp off and again with the lamp on, the measurement with the lamp off is displayed when the diagnostic is entered. To see the second measurement with the lamp on, press the →key (only works with diagnostic codes 1, 7, 8, & 9). The lamp icon will flash and the lamp-on measurement will be displayed in volts. Press →to turn the lamp icon off.

Note: *DIAG 8 will display ---- for the lamp-on voltage if a of >10 NTU is placed in the cell compartment.*

5.3 Other Instrument Diagnostics

5.3.1 Display Test

Pressing and holding the **I/O** key turns on all the display icons and elements so you can determine if all the elements and icons are functioning. The display test sequence will cycle as long as the key is held down.

5.4 Error Messages

Error messages indicate sample interferences and/or instrument malfunction.

5.4.1 Flashing Numeric Display

If the highest value in the range selected is flashing in the display, the sample is too turbid (or overrange) for the selected range. In automatic or manual range, **1000** flashes if the sample is over the instrument's range. In manual range mode, select the next higher range mode if **9.99** or **99.9** flashes. See *Section 2.3.6* on page *21* for measuring overrange samples. The display will stop flashing if a sample within range is inserted and read.

5.4.2 E Messages

An error message indicates either an instrument failure or an operation cannot be performed. **An error message can be cleared by pressing DIAG** (display will return to previous measurement or calibration value). The meter continues to operate as best it can. If the message occurs during a calibration, calibration can continue. If the error message occurs when a calibration is being calculated, the instrument will discard the new calibration and retain the old calibration. Error messages and corrective actions are listed below.

5.4.3 CAL?

A flashing **CAL?** appears when the instrument is using the default calibration programmed at the factory. It will appear if the analyst has erased the user-entered calibration using the procedure to restore the default calibration or after and E 4 error is cleared by pressing DIAG. Recalibrate as soon as possible when **CAL?** appears. **CAL?** (not flashing) appears when a calibration has questionable validity.

Message*	Probable Cause	Corrective Action
E1	Dilution water is ≥ 0.5 NTU.	Start calibration over with better quality dilution water or use a membrane filter to filter the water before use.
E2	Two standards have the same value or their difference is less than 60 NTU. Not all standards were read during the calibration. Standard 1 is too low (<10 NTU).	Recheck preparation of standards and repeat calibration.
E3	Low light error.	Re-read measurement. Check lamp** Check for obstructed light path. Dilution may be necessary.
E4	EEPROM malfunction.	Check sum failed. Press **I/O**. If E 4 reappears, call Hach service. If **CAL?** appears, recalibrate.
E5	A/D overrange.	Check for obstructed light path. Call Hach Service.
E6	A/D underrange.	Check for open lid during reading and re-read. Check for obstructed light path. If persists, call Hach Service.
E7	Light Leak.	Close lid before pressing **READ** key.
E8	Bad lamp circuit.	Reinsert lamp leads at terminal block-make sure the lead ends are not touching each other.If this fails, call Hach Service.

* Error messages 4, 5, and 6 may indicate a failure in the internal electronics.
** Check lamp by inserting a pencil or piece of paper into the cell compartment and pressing READ. Light should be visible on the inserted object.

GENERAL INFORMATION

At Hach Company, customer service is an important part of every product we make.

With that in mind, we have compiled the following information for your convenience.

REPLACEMENT PARTS & ACCESSORIES

REPLACEMENT PARTS

Description	Cat. No.
<0.1 NTU* StablCal®** Stabilized Formazin Standard, 100 mL	26597-42
20 NTU StablCal® Stabilized Formazin Standard, 100 mL	26601-42
100 NTU StablCal® Stabilized Formazin Standard, 100 mL	26602-42
800 NTU StablCal® Stabilized Formazin Standard, 100 mL	26605-42
AA Batteries, 4/pkg	19380-04
Battery Door	46005-00
Carrying Case	46506-00
Gelex® Standards, set (includes standards and 3 sample cells)	24641-05
Instrument Manual	46500-88
Lamp Assembly, with leads	46539-00
Mounting Feet, 4/pkg	41093-00
Oiling Cloth	47076-00
Sample Cells, 1 inch, with cap, 6/pkg	24347-06
Silicone Oil, 15 mL	1269-36

OPTIONAL ACCESSORIES AND REAGENTS

Deionized Water, 3.78 L	272-17
Bath, Ultrasonic, 2.8 L (0.75-gal), w/heater	24895-00
Battery Charger, 120 V	46479-00
Battery Charger, 230 V	46479-01
Battery Eliminator, 120 V	46079-00
Battery Eliminator, 230 V	46080-00
Filter, 0.2 micron, 10/pkg	23238-10
Formazin, 4000 NTU, 500 mL	2461-49
Formazin, 4000 NTU,100 mL	2461-42
Hexamethylenetetramine, 100 g	1878-26
Hexamethylenetetramine, 500 g	1878-34
Hydrazine Sulfate, 20 g	742-46
Hydrazine Sulfate, 100 g	742-26
NiCad Rechargeable Battery (4 required)	16077-00
Pipet, serologic, 1.00 mL	532-35

* <0.1 NTU StablCal® Standard is used in place of dilution water standard when performing a calibration.

** StablCal® is a registered trademark of Hach Company.

OPTIONAL ACCESSORIES AND REAGENTS, continued

Description	Cat. No.
Pipet, TenSette®*, 1-10 mL	19700-10
Pipet Tips, for 1-10 mL TenSette Pipet, 50/pkg	21997-96
Pipet Tips, for 1-10 mL TenSette Pipet, 1000/pkg	21997-28
Pipet, Volumetric, Class A, 1.00 mL	14515-35
Pipet, Volumetric, Class A, 5.00 mL	14515-37
Pump, Vacuum, Hand-Operated	14283-00
Pump, Vacuum, 115 V, 60 Hz	14697-00
Pump, Vacuum, 230 V, 50 Hz	14697-02
Sample Degassing Kit	43975-00
Sample Filtration and Degassing Kit	43975-10
StablCal® Low Level Turbidity Standards for Instrument Verification**	
0.1 NTU, 100 mL	27233-42
0.3 NTU, 100 mL	26979-43
0.5 NTU, 100 mL	26980-42
StablCal® Calibration Set for 2100P Turbidimeter	
<0.1, 20, 100, 800 NTU, 500 mL each	26594-00
<0.1, 20, 100, 800 NTU, 100 mL each	26594-10
<0.1, 20, 100, 800 NTU, sealed vials	26594-05
Triton-X Solution, 118 mL (4 oz)	14096-32
Volumetric Flask, 100 mL	14574-42
Volumetric Flask, 200 mL	14574-45

* TenSette™ is a Hach Company trademark.

** Do not use these standards for instrument calibration.

HOW TO ORDER

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(800-227-4224)
By FAX:
(970) 669-2932

By Mail:
Hach Company
P.O. Box 389
Loveland, Colorado 80539-0389
U.S.A.
Ordering information by E-mail:
orders@hach.com

Information Required

- Hach account number (if available)
- Your name and phone number
- Purchase order number
- Brief description or model number
- Billing address
- Shipping address
- Catalog number
- Quantity

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Telephone: (204) 632-5598; FAX: (204) 694-5134

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Authorization must be obtained from Hach Company before sending any items for repair. Please contact the Hach Service Center serving your location.

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In Canada:
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FAX: (204) 694-5134

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Hach Company World Headquarters,
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Loveland, Colorado 80539-0389 U.S.A.
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FAX: (970) 669-2932

In Europe, the Middle East, or in Mediterranean Africa:
Hach Europe, S.A./N.V.
Chaussée de Namur, 1
B-5150 Floriffoux (Namur), Belgium
Telephone: (32)(8l) 44.71.71
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REGISTER YOUR INSTRUMENT NOW

The 2100P Portable Turbidimeter instrument manual is designed to be updated as new information becomes available. Update information will be provided periodically through our company publications.

Update information will be mailed directly to end users who complete and return the attached registration form. Information about new accessories also will be provided directly to you. To ensure you are kept up to date, please complete and mail the registration today.

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Meter Catalog Number _____

Purchase Date _____ In Service Date _____

Serial Number _____ Customer Number _____

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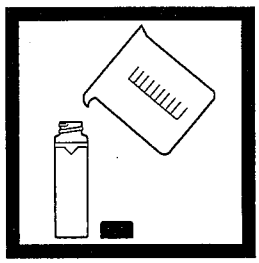
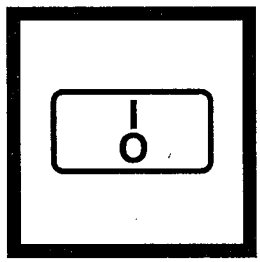
Quick Reference Card
PORTABLE TURBIDIMETER

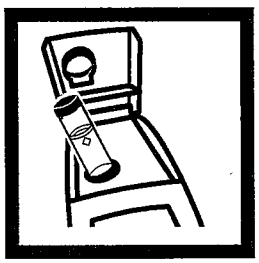
Turbidity Measurement Procedure



1. Fill and cap sample cells. Wipe cells.



2. Turn instrument on. Select measurement range and turn signal averaging on or off.



3. Insert sample cell. Press: **READ**.

4. Record turbidity after lamp icon turns off.

Calibration (factory standards)

1. Prepare stock formazin solution 48 hours before calibration. See *Calibration* in the instrument manual.
2. Prepare 20, 100, and 800 NTU standards. See *Preparing Formazin Dilutions (Factory recommended)* in the instrument manual.
3. Perform calibration. See *Calibrating the Turbidimeter* in the instrument manual.

Error Codes

Message*	Probable Cause
E1	Dilution water is > 0.5 NTU. See *Correcting for Turbidity of Dilution Water* in the instrument manual.
E2	Two standards have values ≤60 NTU apart. See *Preparing Formazin Dilutions (Factory recommended)* or *Preparing User-selected Formazin Dilutions* in the instrument manual. Not all standards were read during the calibration. See *Calibrating the Turbidimeter* or *Preparing User-selected Formazin Dilutions* in the instrument manual. Standard 1 is too low (<10 NTU). See *Section 3.6.3* on page *34* or *Section 3.6.3.1* on page *38*
E3	Low light error. See *E Messages* in the instrument manual.
E4	EEPROM malfunction. Call Hach Service.
E5	A/D overrange. Check for light path obstruction. Call Hach Service if error persists.
E6	A/D underrange. Be sure lid is closed and re-read. Call Hach Service if error persists.
E7	Light Leak. Close lid before pressing **READ** key.
E8	Bad lamp circuit. Ensure lamp lead ends are not touching. Call Hach Service if error persists.

* Error messages 4, 5, and 6 may indicate a failure in the internal electronics.

Error Indicators

Flashing maximum concentration: Concentration exceeds upper limit of range selection. See *Measuring Overrange Samples* or *Flashing Numeric Display* in the instrument manual.

Flashing CAL?: Default calibration values in use. See *Calibration* in the instrument manual to recalibrate.

Flashing lamp icon: Low light level. See *Operational Controls and Indicators* in the instrument manual.

Flashing battery icon: Low battery. See *Battery Replacement* in the instrument manual.

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Nitrogen, Ammonia

★Method 8038

Nessler Method*

(0.02 to 2.50 mg/L NH_3–N)

Scope and Application: For water, wastewater, and seawater; distillation is required for wastewater and seawater; USEPA accepted for wastewater analysis (distillation required); see *Distillation* following this procedure.

* Adapted from *Standard Methods for the Examination of Water and Wastewater 4500-NH_3 B & C*.

Tips and Techniques

- For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the DR/2500 instrument manual for more information on *Running a Reagent Blank*.
- Nessler Reagent is toxic and corrosive. Pipet carefully, using a pipet filler.
- When dispensing reagent from a dropper bottle, hold the bottle vertically. Do not hold the bottle at an angle.
- Nessler Reagent contains mercuric iodide. Both the sample and the blank will contain mercury (D009) at a concentration regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See *Section 4* for further information on proper disposal of these materials.
- If using the Flow Cell, periodically clean the cells by pouring a few sodium thiosulfate pentahydrate crystals into the cell funnel. Flush with enough deionized water to dissolve. Rinse the cell thoroughly.
- A yellow color will develop if ammonia is present. (The reagent will cause a faint yellow color in the blank.)

Method 8038

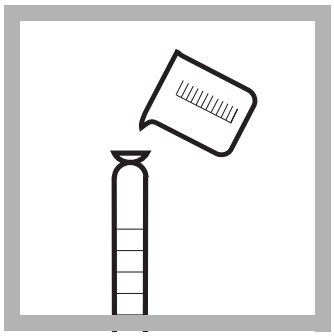
1. Touch

Hach Programs.

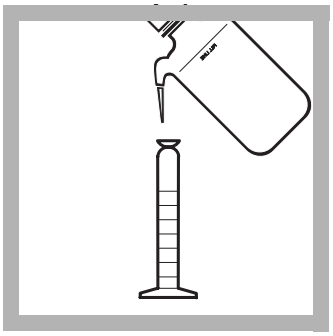
Select program

380 N, Ammonia, Ness.

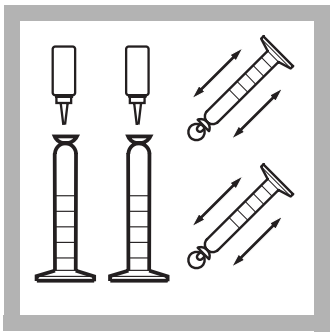
Touch **Start**.



2. Fill a 25-mL mixing graduated cylinder to the 25-mL mark with sample (this is the prepared sample).

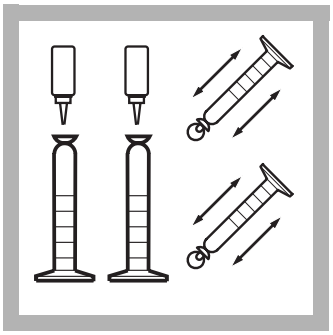


3. Fill a 25-mL mixing graduated cylinder to the 25-mL mark with deionized water (this is the blank).



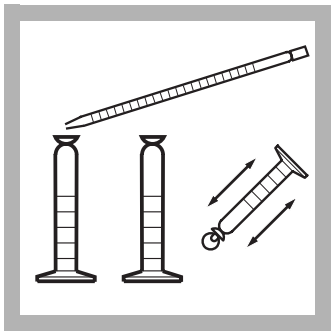
4. Add three drops of Mineral Stabilizer to each cylinder.

Stopper and invert several times to mix.



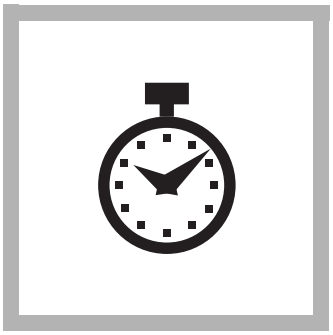
5. Add three drops of Polyvinyl Alcohol Dispersing Agent to each cylinder.

Stopper and invert several times to mix.



6. Pipet 1.0 mL of Nessler Reagent into each cylinder.

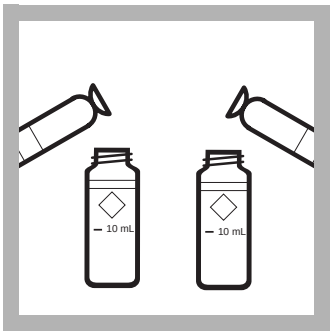
Stopper and invert several times to mix.



7. Touch the timer icon.

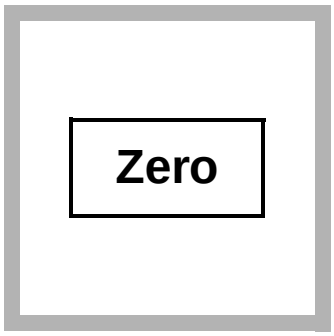
Touch **OK**.

A one-minute reaction period will begin.



8. Pour each solution into a round sample cell.

9. When the timer beeps, place the blank into the cell holder.



10. Touch **Zero**.

The display will show:

0.00 mg/L NH_3 –N

11. Wipe the prepared sample and place it into the cell holder.

Results will appear in mg/L NH_3–N.

Interferences

Interfering Substance	Interference Levels and Treatments
Chlorine	Remove residual chlorine by adding 2 drops of sodium arsenite for each mg/L Cl from a 250 mL sample. Sodium thiosulfate can be used in place of sodium arsenite. See *Sample Collection, Storage, and Preservation* below.
Hardness	A solution containing a mixture of 500 mg/L $CaCO_3$ and 500 mg/L Mg as $CaCO_3$ does not interfere. If the hardness concentration exceeds these concentrations, add extra Mineral Stabilizer.
Iron	Interferes at all levels by causing turbidity with Nessler Reagent.
Seawater	May be analyzed by adding of 1.0 mL (27 drops) of Mineral Stabilizer to the sample before analysis. This complexes the high magnesium concentrations found in sea water, but the sensitivity of the test is reduced by 30 percent due to the high chloride concentration. For best results, perform a calibration, using standards spiked to the equivalent chloride concentration, or distill the sample as described below.
Sulfide	Interferes at all levels by causing turbidity with Nessler Reagent.
Glycine, various aliphatic and aromatic amines, organic chloramines, acetone, aldehydes and alcohols	May cause greenish or other off colors or turbidity. Distill the sample if these compounds are present.

Sample Collection, Storage, and Preservation

Collect samples in clean glass or plastic bottles. If chlorine is present, add one drop of 0.1 N Sodium Thiosulfate (Cat. No. 323-32) for each 0.3 mg/L Cl_2 in a 1-liter sample. Preserve the sample by reducing the pH to 2 or less with sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide (Cat. No. 2450-32) before analysis. Correct the test result for volume additions; see *Section 3.1.3 Correcting for Volume Additions* on page *31*.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Touch **Options**. Touch **Standard Additions**. A summary of the standard additions procedure will appear.
3. Touch **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Touch **Edit** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See *Standard Additions* in the DR/2500 instrument manual for more information.
4. Snap the neck off a Nitrogen Ammonia Voluette® Ampule Standard, 50-mg/L NH_3–N.
5. Prepare three sample spikes. Fill three mixing cylinders (Cat. No. 1896-40) with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of the 50 mg/L standard, respectively, to each sample and mix thoroughly.
6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by touching **Read**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, the display will show the extrapolated concentration value and the "best-fit" line through the standard additions data points, accounting for matrix interferences.

See *Section 3.2.2 Standard Additions* on page *36* for more information.

Standard Solutions Method

1. To check accuracy, use a 1.0-mg/L Nitrogen Ammonia Standard Solution. Or, prepare a 1.0-mg/L ammonia nitrogen standard solution by pipetting 1.00 mL of Nitrogen Ammonia Voluette® Ampule Standard, 50-mg/L, into a 50-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the Nessler procedure as described above.
2. To adjust the calibration curve using the reading obtained with the 1.0-mg/L Nitrogen Ammonia Standard Solution, touch **Options** on the current program menu. Touch **Standard Adjust**.
3. Touch **On**. Touch **Adjust** to accept the displayed concentration. If an alternate concentration is used, touch the number in the box to enter the actual concentration, then touch **OK**. Touch **Adjust**.

See *Section 3.2.3 Adjusting the Standard Curve* on page *38* for more information.

Method Performance

Precision

Standard: 1.00 mg/L NH_3–N

Program	95% Confidence Limits of Distribution
380	0.93–1.07 mg/L NH_3–N

See *Section 3.4.3 Precision* on page *42* for more information, or if the standard concentration did not fall within the specified range.

Sensitivity

Portion of Curve	ΔAbs	ΔConcentration
Entire range	0.010	0.02 mg/L NH_3–N2

See *Section 3.4.5 Sensitivity* on page *43* for more information.

Distillation

1. Measure 250 mL of sample into a 250-mL graduated cylinder and pour into a 400-mL beaker. Destroy chlorine, if necessary, by adding 2 drops of Sodium Arsenite Solution per mg/L Cl_2.
2. Add 25 mL of Borate Buffer Solution and mix. Adjust the pH to about 9.5 with 1 N sodium hydroxide solution. Use a pH meter.
3. Set up the General Purpose Distillation Apparatus (Cat. No. 22653-00) as shown in the *Hach Distillation Apparatus Manual*. Pour the solution into the distillation flask. Add a stir bar.
4. Use a graduated cylinder to measure 25 mL of deionized water into a 250-mL Erlenmeyer flask. Add the contents of one Boric Acid Powder Pillow. Mix thoroughly. Place the flask under the still drip tube. Elevate so the end of the tube is immersed in the solution.

5. Turn on the heater power switch. Set the stir control to 5 and the heat control to 10. Turn on the water and adjust to maintain a constant flow through the condenser.
6. Turn off the heater after collecting 150 mL of distillate. Immediately remove the collection flask to avoid sucking solution into the still. Measure the distillate to ensure 150 mL was collected (total volume = 175 mL).
7. Adjust the pH of the distillate to about 7 with 1 N sodium hydroxide. Use a pH meter.
8. Pour the distillate into a 250-mL volumetric flask; rinse the Erlenmeyer with deionized water. Add the rinsings to the volumetric flask. Dilute to the mark. Stopper. Mix thoroughly. Analyze as described above.

Summary of Method

The Mineral Stabilizer complexes hardness in the sample. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color is formed proportional to the ammonia concentration. Test results are measured at 425 nm.

Required Reagents

Description	Quantity Required Per Test	Unit	Cat. No.
Ammonia Nitrogen Reagent Set			24582-00
Includes:			
Nessler Reagent	2 mL	500 mL	21194-49
Mineral Stabilizer	6 drops	50 mL SCDB	23766-26
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	23765-26
Water, deionized	25 mL	4 liters	272-56

Required Apparatus

Cylinder, graduated, mixing, 25-mL	2	each	21190-40
Pipet, serological, 1-mL	2	each	9190-02
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 10-mL, w/cap	2	6/pkg	24276-06

Required Standards

Nitrogen, Ammonia Standard Solution, 1-mg/L NH_3–N	500 mL	1891-49
Nitrogen, Ammonia Standard Sol., 10-mL Voluette® Ampule, 50-mg/L NH_3–N	16/pkg	14791-10

Distillation Reagents

Borate Buffer Solution	1000 mL	14709-53
Boric Acid Powder Pillows	100/pkg	14817-99
Sodium Arsenite Solution, 5.0-g/L	100 mL MDB	1047-32
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

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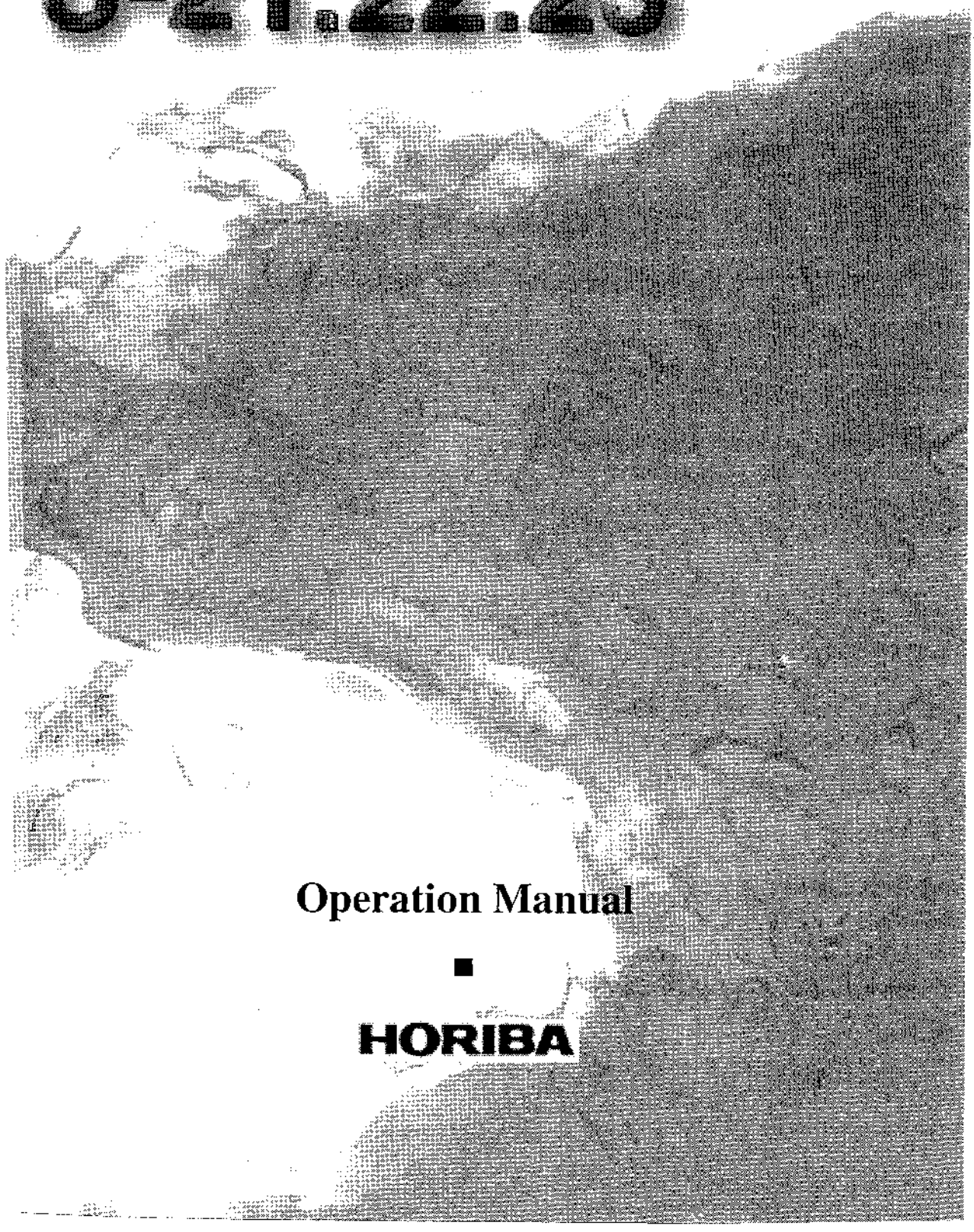
MULTI-PARAMETER WATER QUALITY MONITORING SYSTEM

U-20 Series

U-21,22,23



Operation Manual

■

HORIBA

HORIBA's Warranty and Responsibility

Your U-20 series multi-parameter water quality monitoring system is covered by HORIBA's warranty for a period of one (1) year, under normal use. Although unlikely, if any trouble attributable to HORIBA should occur during this period, necessary exchange or repairs shall be conducted by HORIBA, free of charge. The warranty does not cover the following:

- Any trouble or damage attributable to actions or conditions specifically mentioned in the operation manuals to be avoided
- Any trouble or damage attributable to use of the multi-parameter water quality monitoring system in ways or for purposes other than those described in the operation manuals
- If any repairs renovations, disassembly, etc. are performed on this multi-parameter water quality monitoring system by any party other than HORIBA or a party authorized by HORIBA
- Any alteration to the external appearance of this multi-parameter water quality monitoring system attributable to scratches, dirt, etc. occurring through normal use
- Wear and tear to parts, the exchange of accessories, or the use of any parts not specified by HORIBA

Conformable Directive

CE

This equipment is in conformity with the following directives and standards:

Directives: The EMC Directives 89/336/EEC as amended by 91/263/EEC, 92/31/EEC and 93/68/EEC, in accordance with the Article 10 (1) of the Directive.

Standards: EN55011: 1991 Class B Group I
EN50082-1: 1992

FCC Warning

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Unauthorized reprinting or copying of this operation manual

No unauthorized reprinting or copying of all or part of this operation manual is allowed. The utmost care has been used in the preparation of this operation manual. If, however, you have any questions or notice any errors, please contact the HORIBA customer service printed on the back cover of this operation manual.

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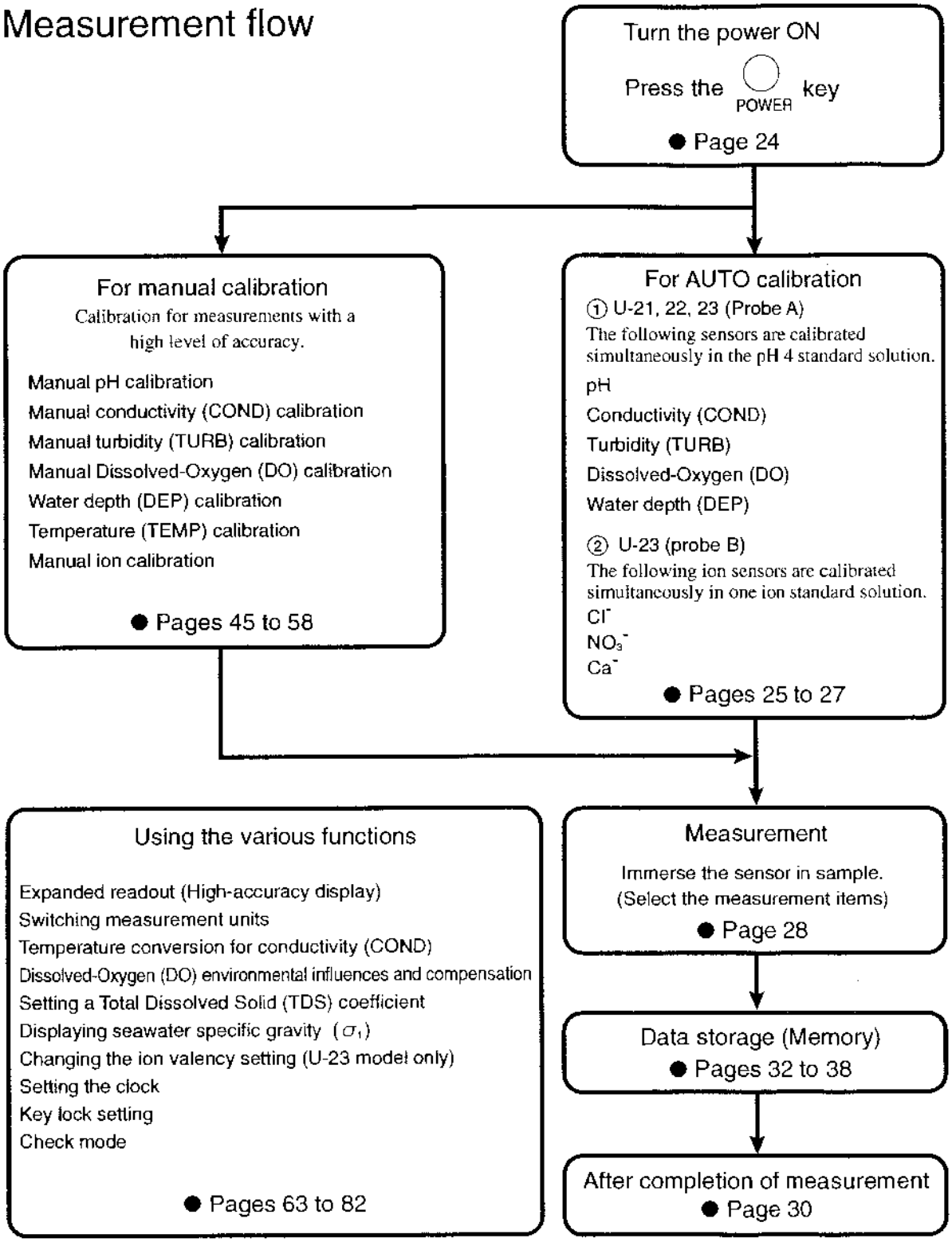

Preface

Thank you very much for purchasing HORIBA's "MULTI-PARAMETER WATER QUALITY MONITORING SYSTEM" U-20 Series.

Compact and one-hand-held, our multi-parameter water quality monitoring system makes measurements about a large number of items simultaneously.

The instrument uses a large-sized LCD display and has a variety of functions through easy operation, being useful for use at sites where measurements are to be made.

The water-proof construction of the instrument is compliant with IP-67 of IEC 529, "Water-proof test on electrical and mechanical equipment and tools and protection grade against entry of solids." Please use the instrument by following the information in this Operation Manual to maintain the water-proof construction of the instrument.

IP-67 standards

- Keeping dust and grit out of the instrument
- Up to 5 C difference between water and an instrument employed and no entry of water into the inside of the instrument at a depth of 1 m for 30 minutes

This Operation Manual contains information on the basic way of handling the instrument, notes, etc. for the user. Be sure to read through the Operation Manual before use.

Symbols employed

The symbols employed herein have the following meanings:

⚠ **CAUTION** : The improper use of the instrument may cause the following dangers:
- Danger of injury
- Danger of damage to the instrument, its peripherals, and data

Important : Explanation necessary for the proper operation of the instrument

Note : Explanation that is useful and necessary for handling the instrument

☞ : Refer to the item shown.

Symbols employed in screen description

The symbols and used in screen description have the following meanings:

: The letters and numbers in this symbol are blinking on the screen.

: The letters and numbers in this symbol are lighting up on the screen.

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1. Introduction

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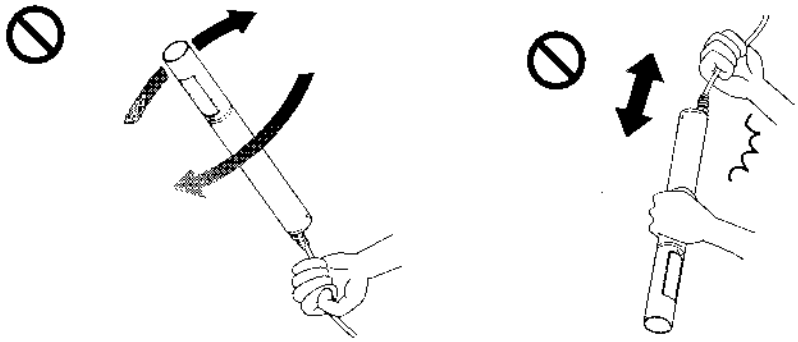
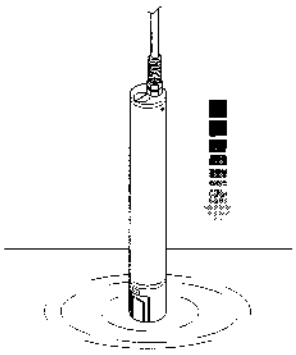
1.1 Notes on handling the instrument

① Handling the sensor

• Do not swing the sensor around or pull the cord on the sensor.



• Slowly lower the sensor probe into the sample.

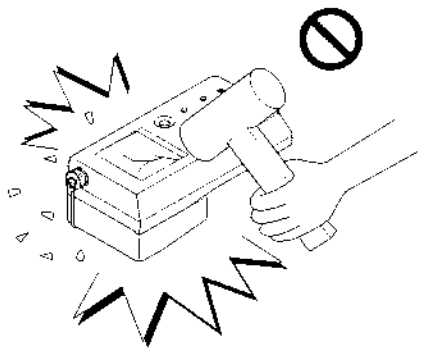


⚠WARNING

- In place with a large distance to the water level or with a rapid water flow, fix the sensor probe hook to a point except your body before use for safety purposes.
  Be careful not to let go off the sensor probe by mistake. Otherwise, the sensor probe together with the instrument will fall into the water or a sharp shock will occur to yourself while you are holding the instrument.

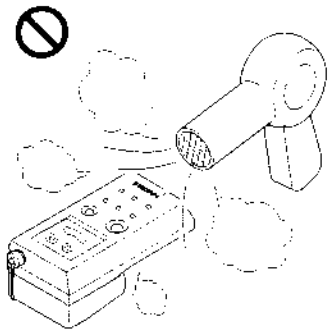
② Shock

• Do not give a shock to or drop the sensor or instrument.

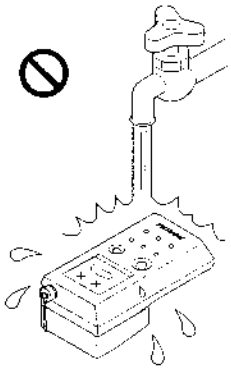


) Water-proof ability of the instrument

The instrument will be water-proof in construction (IP-67) when the sensor connector is connected to the instrument.
However, if the instrument has been dropped into water or become wet, use a soft cloth to dry up the instrument.
Do not use a hair dryer to dry up the instrument.

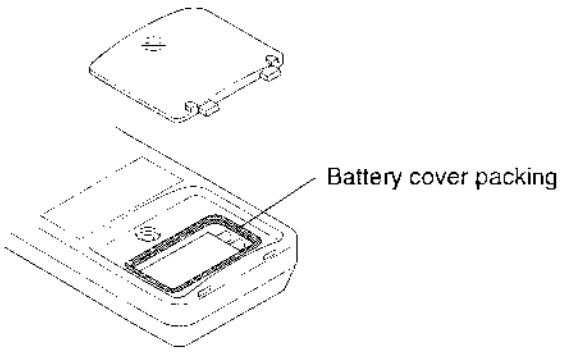


Do not wash directly the instrument using tap water from the faucet.



) Opening and closing the instrument battery cover and the expansion unit

Foreign matter on the battery cover packing can cause water to enter the instrument. Check for foreign matter on the battery cover packing before closing the battery cover and the expansion unit.
If the battery cover packing is twisted, do not close the battery cover.



r a long use

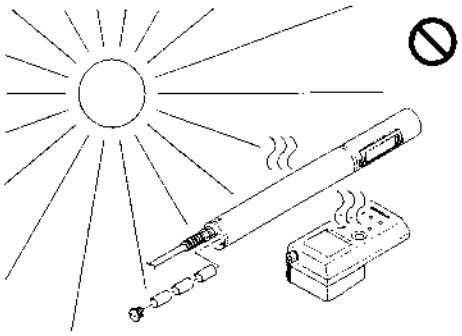
e recommend that the battery cover packing be replaced once a year.
r battery cover packing replacement, contact your sales agent.

Replacing the sensor probe battery

Do not replace the sensor probe battery in very hot and wet atmosphere.
Replace the battery with the instrument connected to the probe.

⑥ No direct sunlight to the instrument

• The display part includes LCD. Do not expose the instrument to ultraviolet rays for a long time. Otherwise, the LCD may deteriorate.



⑦ Chemical resistance

• Do not soak the instrument or sensor in alcohol, organic solvent, strong acid, or strong-alkaline solution.
The instrument includes ABS and acrylic resins and various types of rubber. The sensor part includes polycarbonate resins, denatured PPO resins, polypropylene resins, polysulfone resins, POM resins, nylon resins, and various types of rubber.

For a long use

If the instrument and sensor probe become contaminated, use a soft cloth damped with water or diluted detergent to wipe off the contamination.

⑧ Note on place for use

• Do not use the instrument in the atmosphere with ambient temperatures below 0 C (incl.) or above 55 C (incl.).
Also avoid using the instrument in the atmosphere with sharp vibrations and corrosive gases.
Do not use the instrument near a source of strong electromagnetic field such as high-voltage cables and motors.

⑨ Batteries

The improper use of batteries may cause leaks and explosion.
Observe the followings:

- Set the batteries in place properly while paying attention to the plus (+) and minus (-) poles.
- Do not use both an old and new batteries or batteries of different types.
- Batteries for use in the instrument are not of the rechargeable type.
- Remove the batteries when not in use for a long time.
  In case of leaks, wipe off the solution in the battery case thoroughly and place new batteries in position.

⑩ Do not disassemble the instrument.

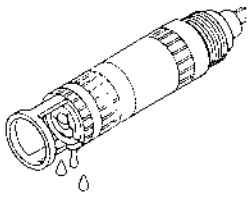
• Never disassemble the instrument. Otherwise, the water-proof performance of the instrument will be impaired.

Handling the DO sensor

For any broken DO sensor diaphragm, replace the DO sensor.

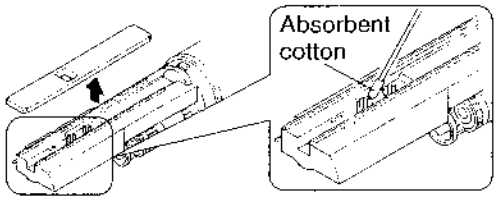
⚠CAUTION

The DO sensor holds a strong-alkaline solution. Protect the eye and skin from the solution. If there is any solution in the eye or on the skin, immediately use sufficient water to wash off the solution. Consult a doctor as required.



Handling the COND/TURB unit

When cleaning the COND/TURB unit, use an absorbent cotton to avoid damage to the TURB cell.



Handling the pH sensor

The pH sensor has a glass electrode at the end. Handle the sensor carefully to avoid a break in the glass electrode.

⚠CAUTION

Be careful not to break the glass on the top of the sensor. Otherwise you may get hurt with a piece of glass.

Handling the Connector section

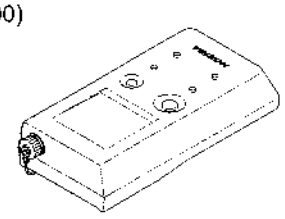
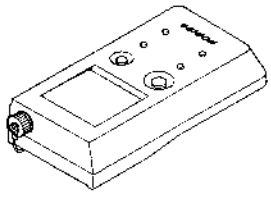
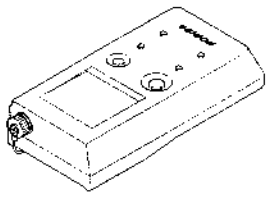
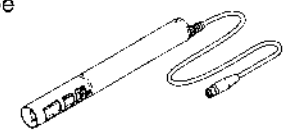
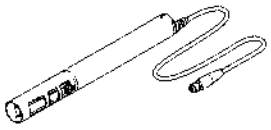
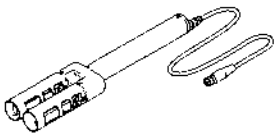
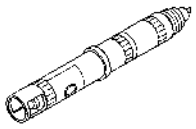
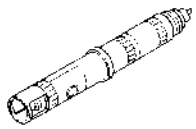
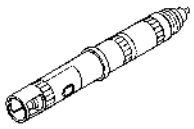
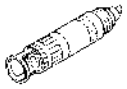
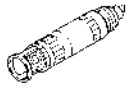
Handle the sensor connector section carefully to protect it from water, sensor-contained solution, sample and contamination.

Disposal

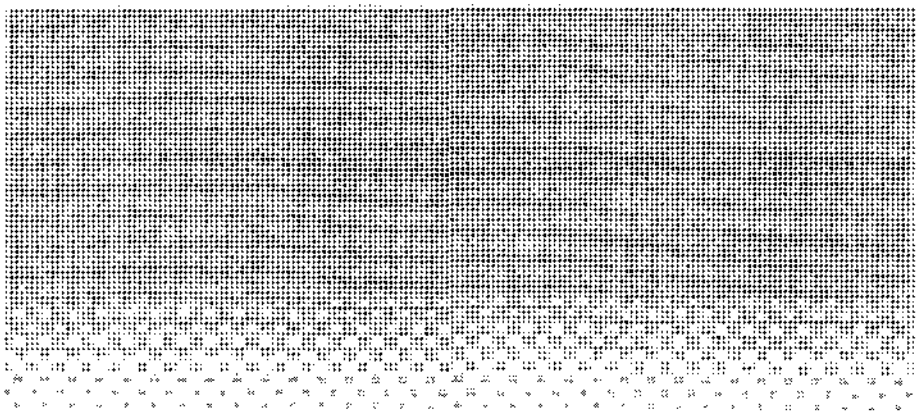
Dispose of this product as industrial waste. Otherwise this may affect the environment.

1.2 Packing list

The U-20 series is comprised of the following items.

Model	U-21	U-22	U-23
Meter (U-2000)			
Sensor probe			
Sensor	pH sensor 	pH/ORP sensor 	pH/ORP sensor 
	DO (Dissolved-Oxygen) Sensor 	DO (Dissolved-Oxygen) Sensor 	DO (Dissolved-Oxygen) Sensor
			Ion sensor (3 types) Cl^- . NO_3^- . Ca^{2+}
Accessories	pH4 standard solution (500 ml) pH internal solution (250 ml) Syringe (with needle)	pH4 standard solution (500 ml) pH internal solution (250 ml) Syringe (with needle)	pH4 standard solution (500 ml) Ion standard solution (250 ml) pH internal solution (250 ml) Cl^- ion internal solution (50ml) NO_3^- ion internal solution (50 ml) Ca^{2+} ion internal solution (50 ml) Syringe (with needle) Syringe (3 pieces) Connector plug for the probe (3 pieces)
	Sensor spanner	Sensor spanner	Sensor spanner
	Calibration beaker	Calibration beaker	Calibration beaker (2 pieces)
	Grip holder	Grip holder	Grip holder
	Carrying case	Carrying case	Carrying case
	Alkaline battery 6LR61 (S006P) (1 piece)	Alkaline battery 6LR61 (S006P) (1 piece)	Alkaline battery 6LR61 (S006P) (1 piece)
	Alkaline batteries LR03 (AAA) (3 pieces)	Alkaline batteries LR03 (AAA) (3 pieces)	Alkaline batteries LR03 (AAA) (3 pieces)
	Operation manual	Operation manual	Operation manual

- The included battery is for the monitor. Its life is not guaranteed.



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2.1 Introduction to the U-20 series models

2.1.1 Measurement items

Components that can be measured with the U-20 series models are as follows:

Model	U-21	U-22	U-23
Measurement items			
pH	○	○	○
Dissolved Oxygen (DO)	○	○	○
Conductivity (COND)	○	○	○
Salinity (SAL) [Conductivity conversion]	○	○	○
Total dissolved solids (TDS) [Conductivity conversion]	○	○	○
Specific gravity of seawater [Conductivity conversion]	○	○	○
Temperature (TEMP)	○	○	○
Turbidity (TURB)	○	○	○
Water depth (DEP)	–	○	○
Oxidation-Reduction Potential (ORP)	–	○	○
Chloride ion Cl^-	–	–	○
Nitric acid ion NO_3^-	–	–	○
Calcium Ca^{2+}	–	–	○
Fluoride ion F^-	–	–	(○)
Kalium ion K^+	–	–	(○)
Ammonia gas NH_3	–	–	(○)

○ ..... Measurable

(○) ..... Measurable in case of use of an optional sensor

2.1.2 Introduction to functions of the instrument

Outline of the functions of the instrument is described below.

Feature	Function name	Page
Data obtained during measurement can be saved in the memory.	Manual data storage	Page 32
Data can be automatically saved in the memory at constant time intervals. (U-22,U-23 models)	Auto data storage	Page 34
Saved data can be called.	DATA OUT	Page 39
The latest date of calibration and its details can be called.	Calibration history	Page 41
Enlarged display is available.	Expand readout	Page 63
Measurement units can be switched.	Switching measurement unit	Page 64

Other functions possible in the check mode are available. (☞ Page 74)

2.1.3 Functions of expansion units

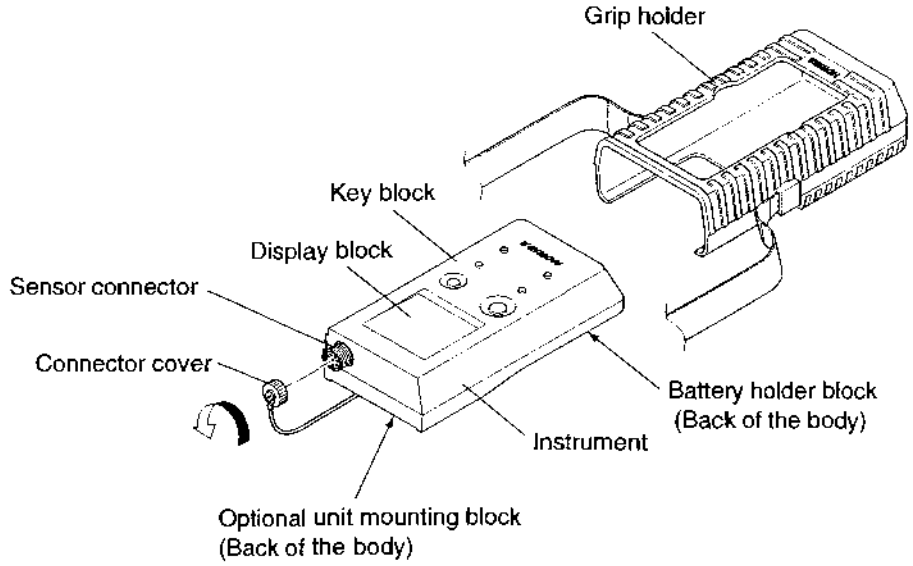
For the U-20 series, use of expansion units allows communications with personal computers through RS-232C, the storage of G.P.S. data in the memory, and printer output, and commercial power supply.
Expansion units are available in the following two types:

Unit/name	Contents	Functions
U-2001	• Expansion adaptor	<RS-232C communications, G.P.S connection, and printer output>
Expansion adaptor	• Software for PC	The above functions cannot be used at the same time. One of the connectors for these three functions needs to be used.
U-2002	• System unit contain case	<RS-232C communications, G.P.S connection, printer output, battery power supply*>
System unit	• Software for PC	The above functions can be used at the same time.
	• G.P.S. unit	* A battery power supply can be used for measurements outdoors for 30
	• Printer set	consecutive days.

U-2001 and U-2002 can operate on a commercial power supply through the use of an AC adapter (optional). However, the AC adapter cannot be used for the G.P.S. unit or printer set.

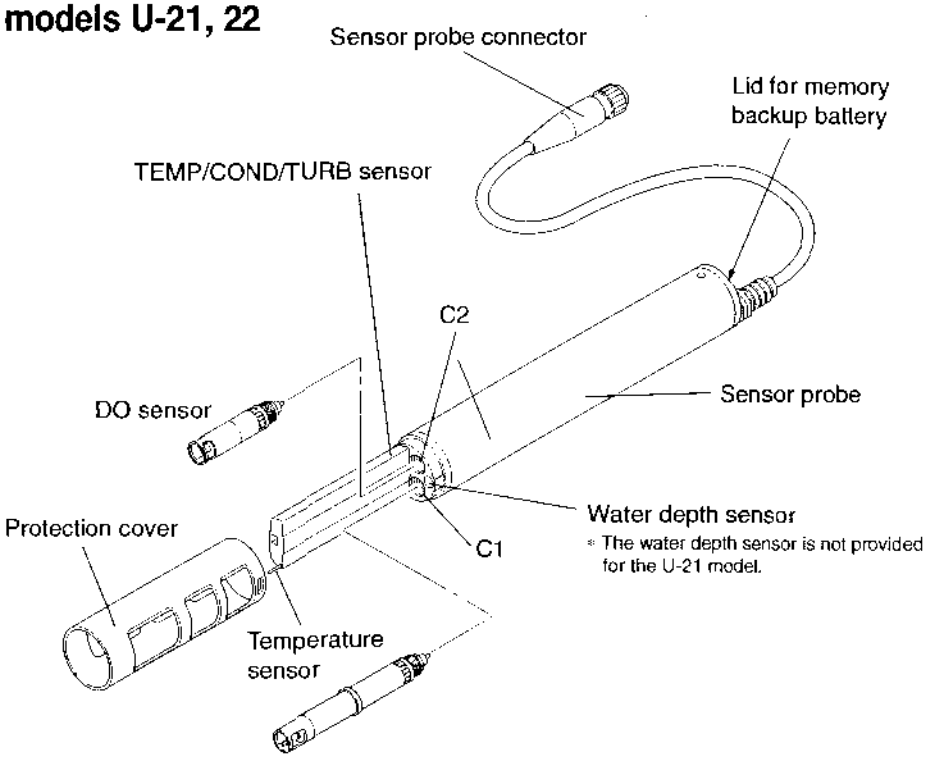
2.2 Names of the parts

2.2.1 Instrument name

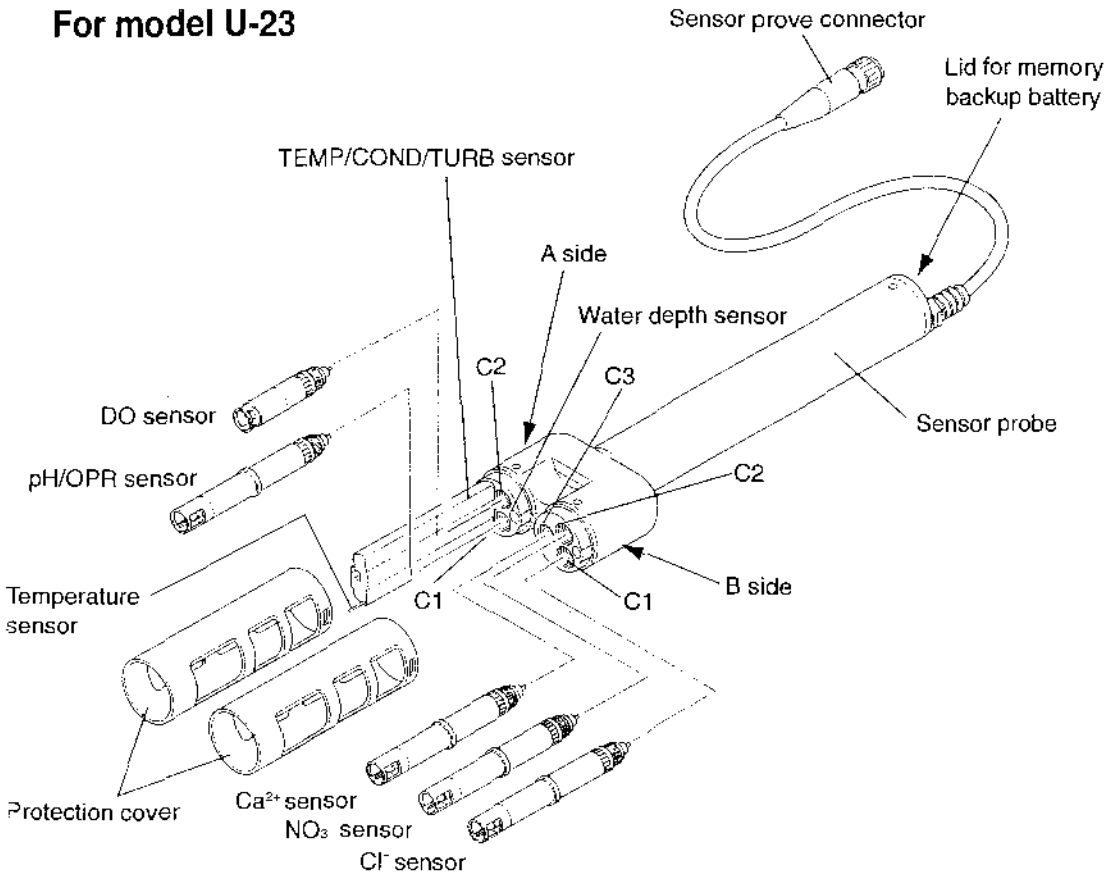


2.2.2 Sensor probe names

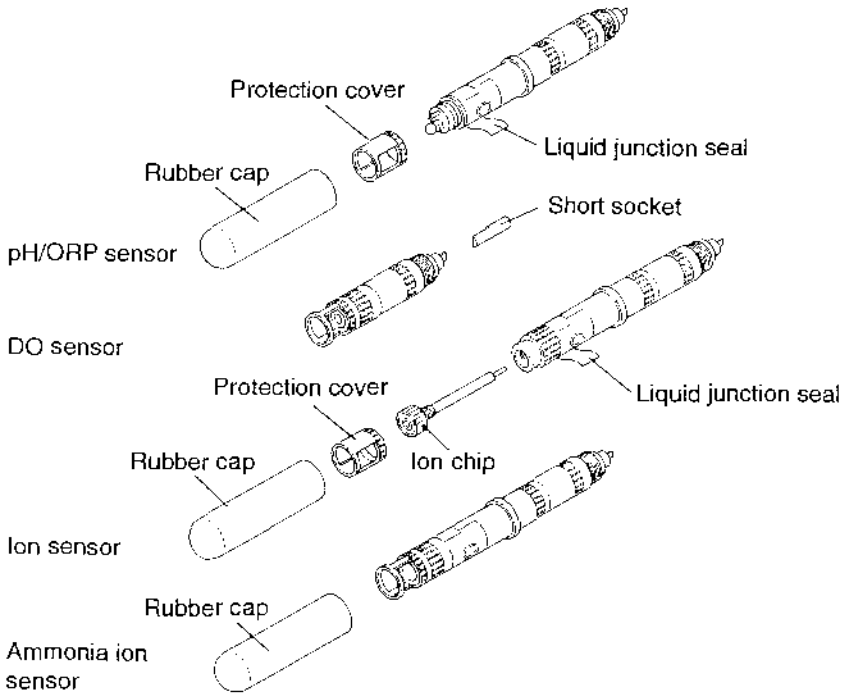
For models U-21, 22



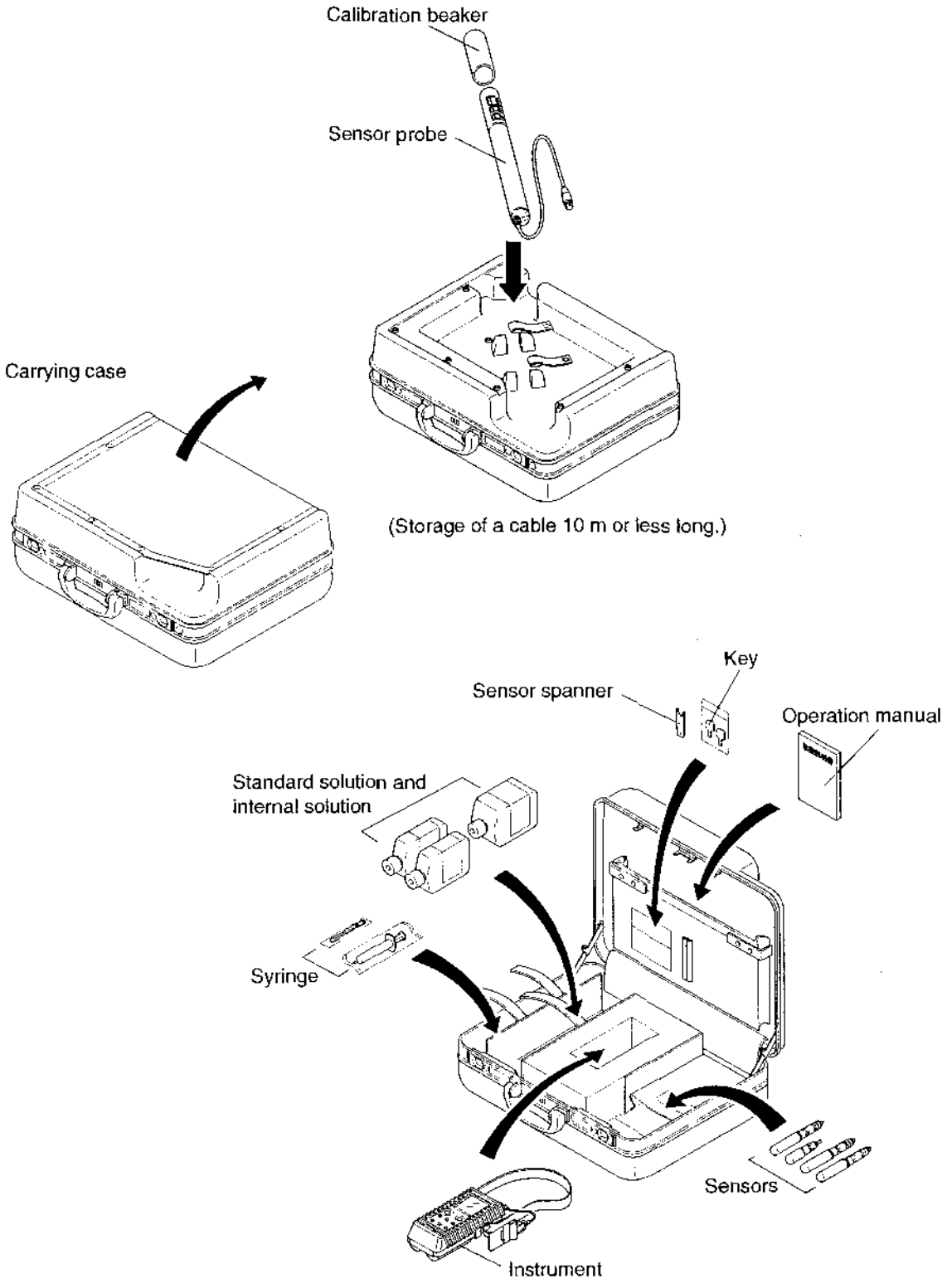
For model U-23



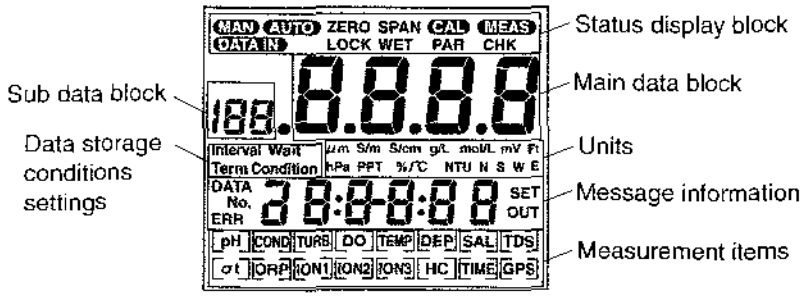
2.3 Sensor names



2.2.4 Use of carrying case



!.2.5 Display



tatus display block

MAN ..... On when the data memory and calibration settings are set to manual.
AUTO ..... On when the data memory and calibration settings are set to automatic.
DATA IN ..... On when the data memory operation and the data memorys operation settings are being performed. Blinking during calibration.
ZERO ..... On in the Zero calibration mode.
SPAN ..... On in the Span calibration mode.
CAL ..... On in the Calibration mode.
MEAS ..... On in the Measurement mode. (Measurements are being made when light up.)
LOCK ..... On when the keys are locked.
CHK ..... On when the instrument is in the check mode.

ub data block

Display of the pH, Latitude (degree), Longitude (degree), Year and Check No.

ain data block

Display of Measurement data, Latitude (minute, 〔second〕), Longitude (minute, 〔second〕), and month and day.

ata storage conditions setting

Interval ..... On when a time interval is set for storage of data.
Wait ..... On when a time is set for waiting from the automatic data storage instruction until the start and during data processing through individual operations.
Term ..... On when a period is set for automatic data storage.

nits

Displays the units for measurement items.

essage information

Displays the stored data (data mode) and the data No. when the data is stored.
SET ..... Indicates that the instrument is in Set mode.

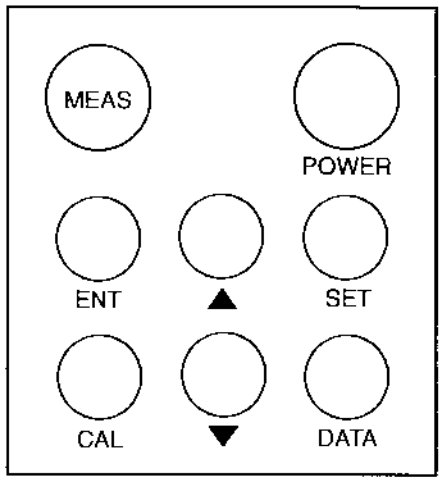
easurement items

Displays the measurement items for the data in the main data block display. The display is read as follows.
Items without brackets ([ ]) ..... Items with the highlighted text will be stored in the data memory.
(☞ *Measurement item setting*, page 77)
Items with brackets ([ ]) ..... Displays the measurement items with data display.
(☞ *Measurement item setting*, page 77)

Note

Because of the instrument's automatic power off function, the indication will disappear if the nnit is not used for about 30 minutes. For operation of the unit and display of the indication, turn ON the instrument again.

2.2.6 Key names



POWER: Power key

Turns the instrument On and Off. Immediately after the power is switched on, the initial screen is displayed to indicate the status of the instrument.

MEAS: Measurement key

In the Measurement mode (MEAS is on), this key switches the measurement item. In addition, pressing the MEAS key returns you from the Setting, Calibration and Memory Call Up modes to the Measurement mode.

Note

- Regardless of which mode the instrument is in, it is always possible to return to the Measurement mode by pressing the MEAS key.

ENT: Enter key

In the Measurement mode (MEAS is on), pressing the ENT key stores the data in memory.
In the Calibration mode (CAL is on), pressing the ENT key performs calibration.
In the Setting mode, pressing the ENT key switches the setting and registers entered setting values.

CAL: Calibration key

Pressing the CAL key switches the instrument to the Calibration mode. If automatic data storage is in progress, it is aborted.

SET: Set key

Pressing the SET key switches the instrument from the Measurement mode to the Set mode. If the SET key is pressed on the "year, month, day, time" display screen, it switches the instrument to the Check mode.

DATA: DATA key

Pressing the DATA key switches the instrument to the Data mode.

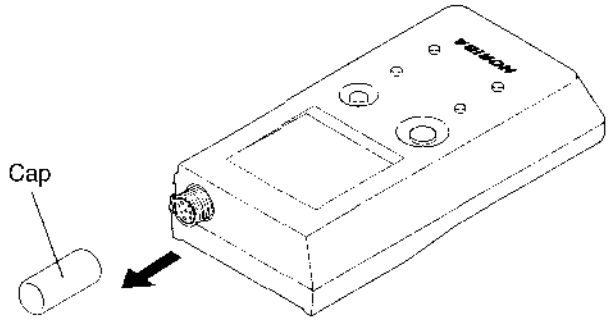
▲▼ : UP/DOWN keys

Use the UP/DOWN (▲▼) keys to set the calibration value in the Manual mode.

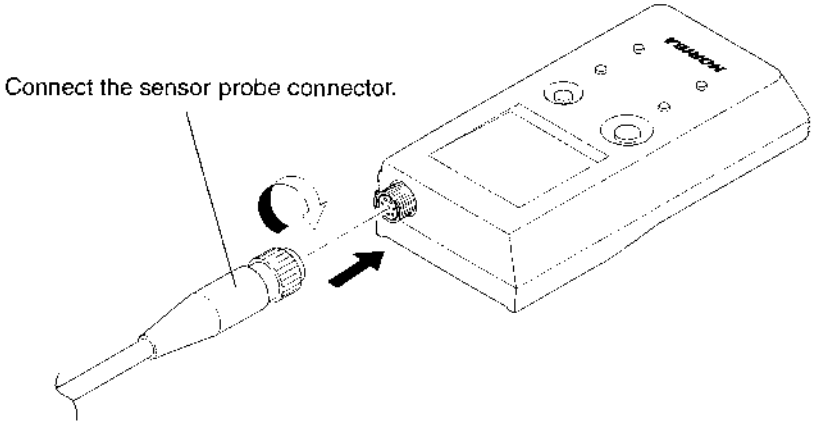
2.3 Setting up the U-20 series models

2.3.1 Instrument and sensor probe connection

1. Remove the cap from the instrument's connector.



2. Connect the sensor probe connector.



Note

Tighten the sensor probe connector until it will not turn any more.

⚠ CAUTION

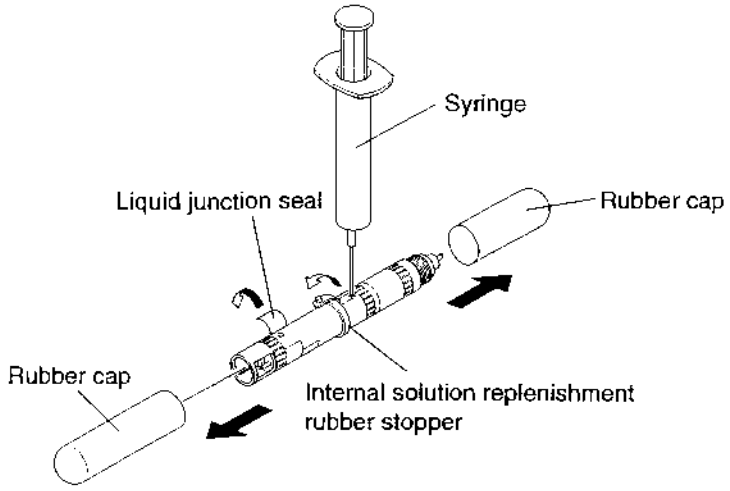
The connector cover or sensor probe connector should be connected to the instrument. Otherwise, the instrument will not be waterproof.

2.3.2 Sensor installation

Connect the Dissolved Oxygen (DO), pH and ion (Cl^-, No_3^-, Ca^{2+}) sensor to the sensor probe.

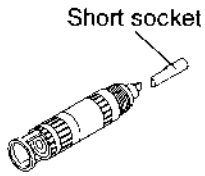
Preparing pH (pH/ORP) sensor

1. Remove the liquid junction seal and rubber caps.
2. Open the internal solution replenishment rubber stopper. Then use a syringe to take internal solution (#330).
   Air bubbles in the internal solution may impair the pressure compensation of the sensor. Allow as few air bubbles as possible to enter the inside solution.



Preparing DO sensor

1. Remove the short socket.

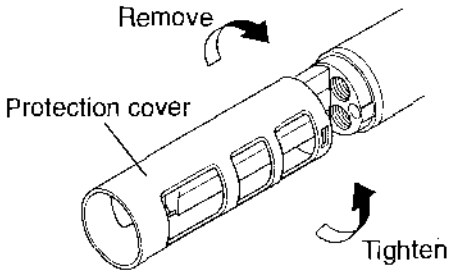


Important

- Provide the DO sensor with a short socket or connect the sensor to the sensor probe for storage. Otherwise, stable instructions may not be obtained.
- The short socket is used when storing. Do not throw it away.

Installation procedure

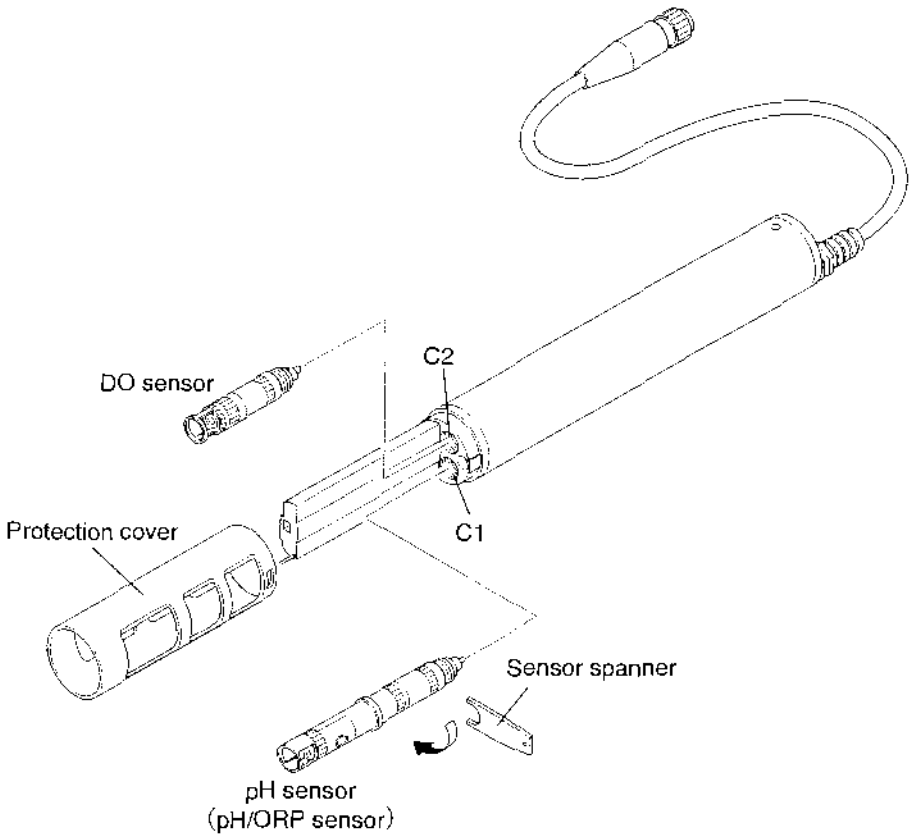
1. Remove the calibration beaker and remove the protection cover from the sensor probe.



2. Align each sensor with the groove in the sensor probe and insert and screw each into the sensor probe.
Then use the sensor spanner to tighten each sensor.

For models U-21, 22

pH (pH/ORP) sensor ..... C1 (For the U-23 model, C1 on the sensor probe A side.)
DO sensor ..... C2 (For the U-23 model, C2 on the sensor probe A side.)

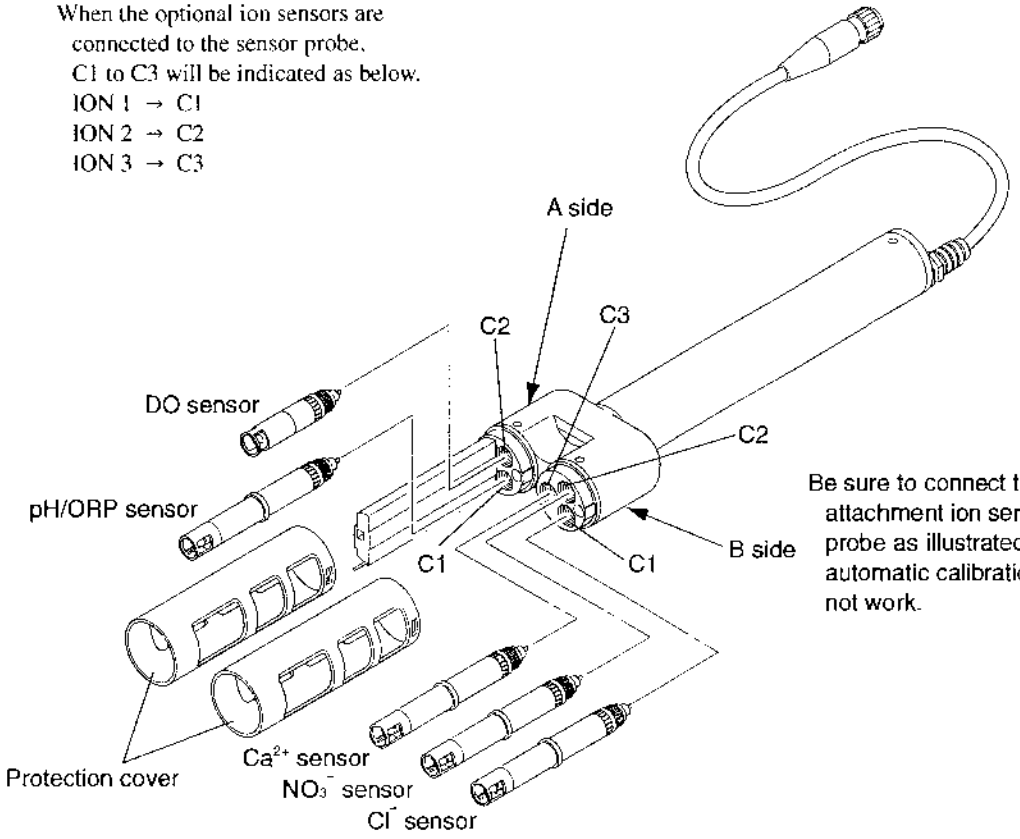


For model U-23

Prepare the sensors described in the Electrode instruction manual. Then connect the sensors to the Sensor probe as illustrated below.

Cl^-..... C1 on the sensor probe B side.
NO_3^-..... C2 on the sensor probe B side.
Ca^{2+} ..... C3 on the sensor probe B side.

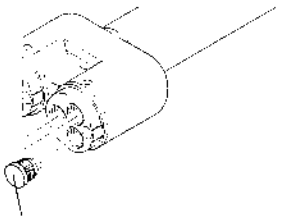
When the optional ion sensors are connected to the sensor probe, C1 to C3 will be indicated as below.
ION 1 → C1
ION 2 → C2
ION 3 → C3



Be sure to connect the standard attachment ion sensor to the sensor probe as illustrated. Otherwise, the automatic calibration function would not work.

⚠ CAUTION

- If any of the sensors is not used, be sure to attach a connector plug for the probe to the opening for the relevant sensor probe instead of the sensor. Otherwise, the sensor probe will not be waterproof.



Connector plug for the probe

3. Attach the removed protection cover to the sensor probe as it was.

Important

- Before attaching each sensor to the sensor probe, do not soak the connector block in water.
- Be careful not to contaminate or wet the sensor probe or sensor connector.

2.3.3 Installation and replacement of the battery

The U-20 series is shipped from the factory with the battery packed separately.
When using the instrument for the first time or replacing the battery, perform the following procedure:

Type of battery:

nstrument (U-2000) ..... Alkaline battery 6LR61 (Manganese battery 6F22 [006P])
1 piece. (Battery for instrument operation)
Sensor probe (U-21, 22, 23) ..... Alkaline batteries LR03 [AAA] (Manganese battery [R03])
3 pieces. (Battery for memory backup)

Notes on handling the battery

The improper use of batteries may cause leaks and explosion.
Observe the followings:

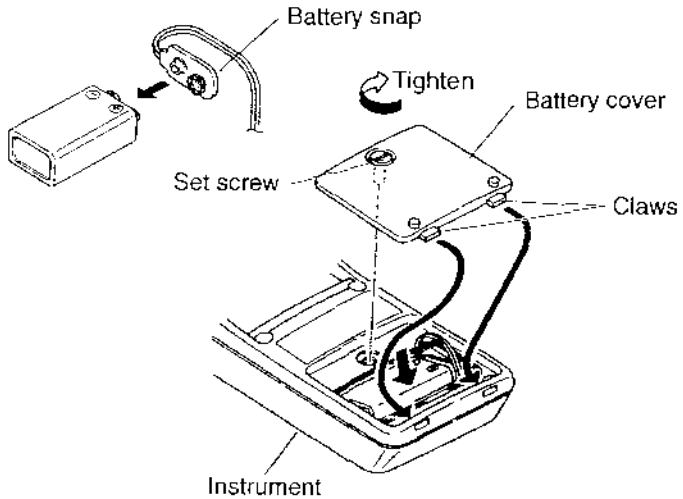
- Set the batteries in place properly while paying attention to the plus (+) and minus (-) poles.
- Do not use both an old and new batteries at a time or batteries of different types.
- Batteries for use in the instrument are not of the rechargeable type.
- Remove the batteries when not in use for a long.
  In case of leaks, wipe off the solution in the battery case thoroughly and place new batteries in position.

[Note]

The battery originally attached to your unit is for monitor and the service life of the battery cannot be guaranteed.

nstrument (U-2000)

1. Loosen the set-screw on the battery cover and remove the cover.
2. Remove any old battery.
3. Fit the battery snaps to a new battery and insert the battery assembly into the instrument.
4. Insert the claws on the battery cover into the grooves in the instrument. Then tighten the set screw.
   The battery snap may be loose for some batteries. In such a case use radio pliers and tighten the metal snap fittings.

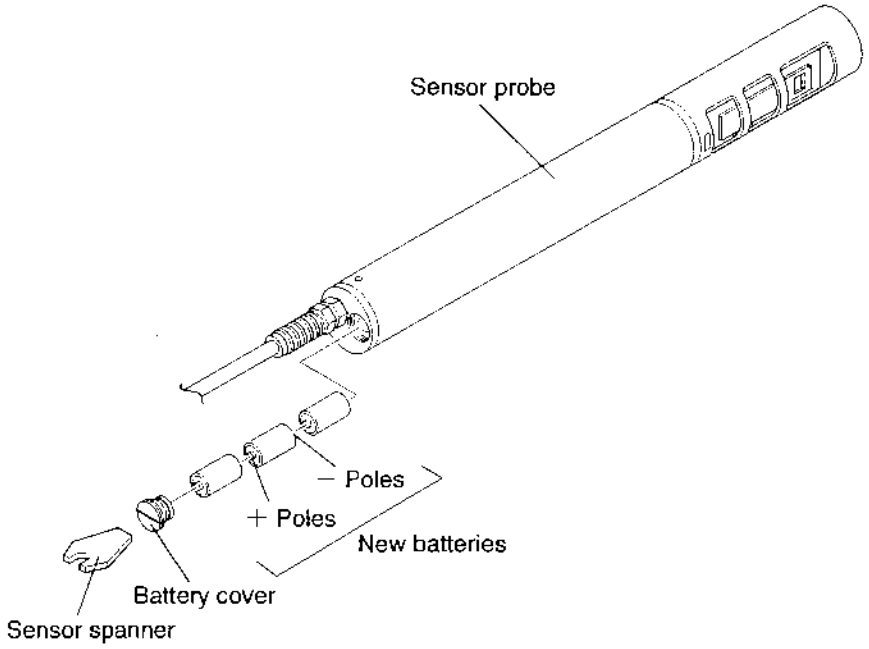


Important

When removing the battery snap, do not pull it too strongly.

Sensor probe (for memory back up)

1. Remove the battery cover using a sensor spanner or a suitable object.
2. Remove any old batteries.
3. Insert new batteries making sure that the plus (+) and minus (-) poles match the terminals correctly.
4. To keep the sensor probe water-resistant, use a chip spanner as illustrated below and tighten the battery cover until the cover does not turn any more.

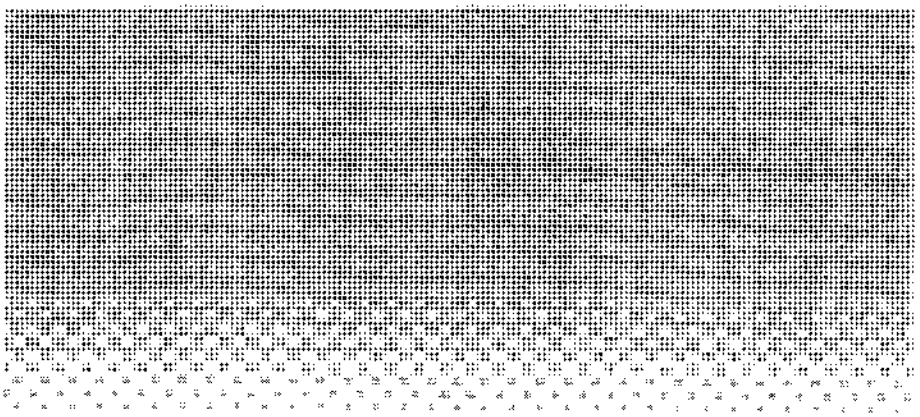


⚠ CAUTION

- When replacing the batteries of the sensor probe, be sure to connect the sensor probe to the instrument. Otherwise, the memory will be reset and all the data saved in the memory will disappear.

◖Note◗

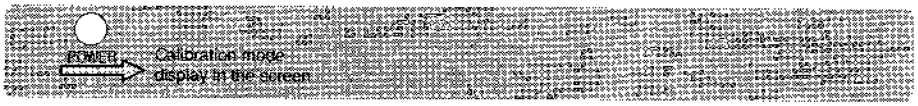
- The battery on the main unit is used up first allowing up to 30 hours use at room temperature. (When using alkaline batteries.)
- Life is reduced by approximately one half when manganese batteries are used.



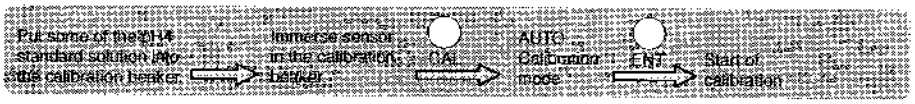
3. Basic operation

The pH, conductivity (COND), turbidity (TURB), dissolved-oxygen (DO), water depth (DEP) and ion (ION1, 2, 3) sensors can be calibrated automatically. Upon completion of this chapter, even beginners should be able to make measurements easily.

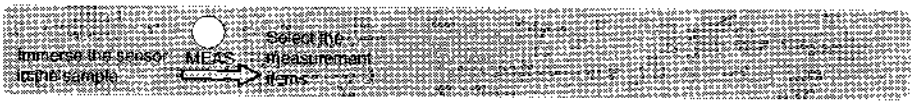
3.1 Key operations and mode switching 22
3.2 Operation procedure 24
3.2.1 Power ON 24



3.2.2 AUTO calibration method 25



3.2.3 Measurement 28



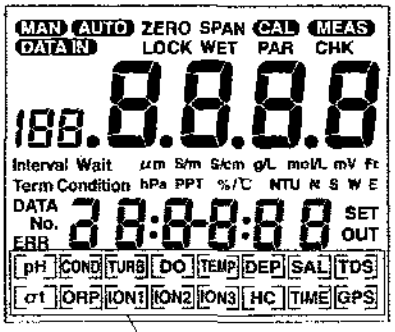
3.2.4 After completion of measurement 30

3.1 Key operations and mode switching

Measuring items and displays which are switched with the MEAS key

The items measurable with individual models are displayed. The items selected with the MEAS key will be indicated with [ ].

Example: In the pH Measurement mode: [pH]



Display block

The symbols displayed and their meanings are as follows:

- pH ..... pH
- COND ..... Conductivity
- TURB ..... Turbidity
- DO ..... Dissolved-Oxygen
- TEMP ..... Temperature
- DEP ..... Depth
- SAL ..... Salinity
- TDS ..... Total dissolved solids
- σ_t ..... Specific gravity of seawater
- ORP ..... Oxidation-reduction potential
- ION1 ..... Cl (Chloride) ion
- ION2 ..... NO_3 (Nitric acid) ion
- ION3 ..... Ca^{2+} (Calcium) ion

(ION1–ION3: if standard attachment ion sensors are used)

- TIME ..... Display of date and time
- GPS ..... G.P.S. (Global Positioning System) for information of position

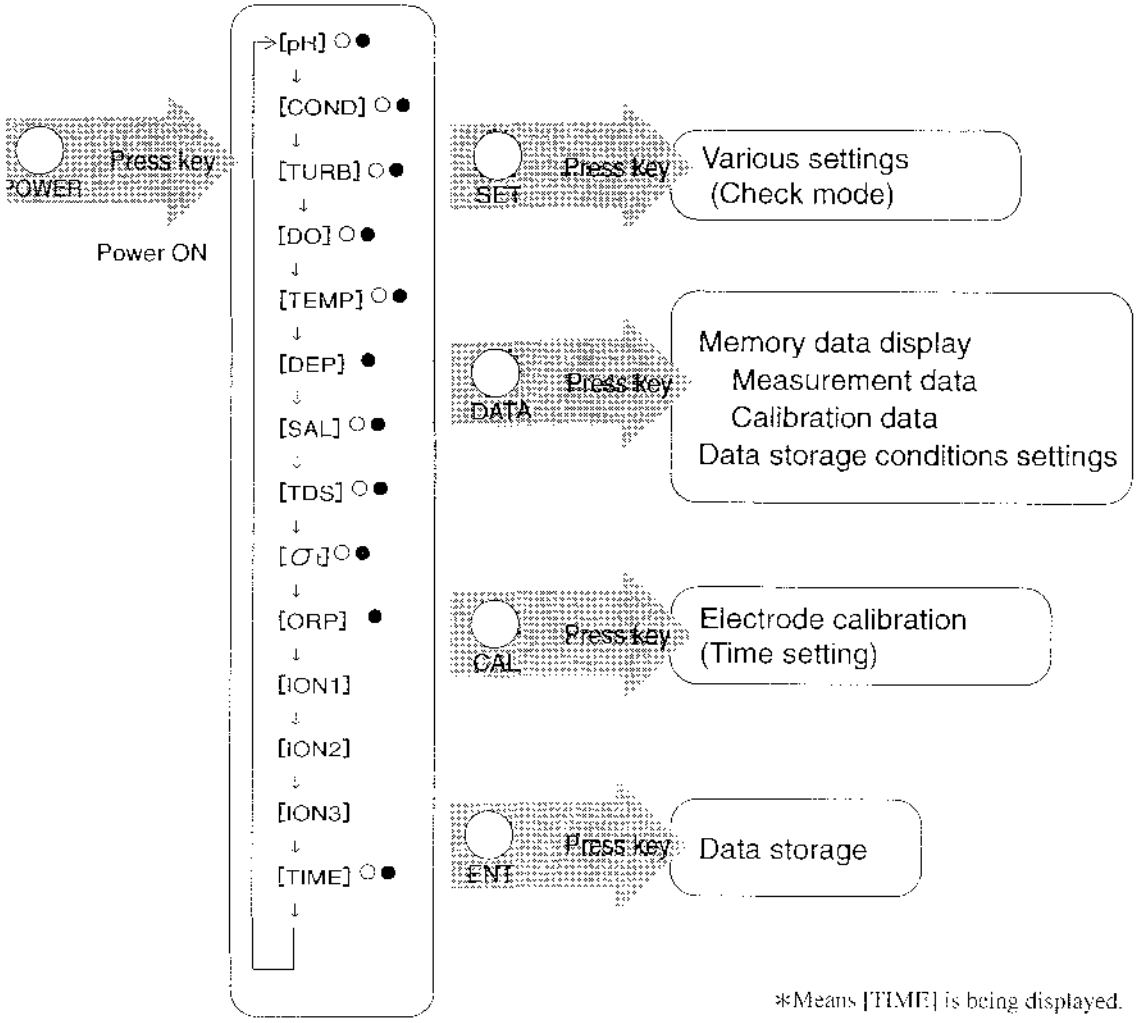
* When optional sensors C1, C2, and C3 are connected to the instrument, ION1, ION2, and ION3 appears for the optional sensors C1, C2, and C3, respectively.

Note

- [GPS] lights up when the optional G.P.S. sensor has been connected to the instrument and position information is received from the G.P.S. sensor during the measurement. For more information, refer to the instruction manual for the expansion units.

U-23 Measurement mode

(MEAS) When the MEAS key is pressed, the next measurement item appears.



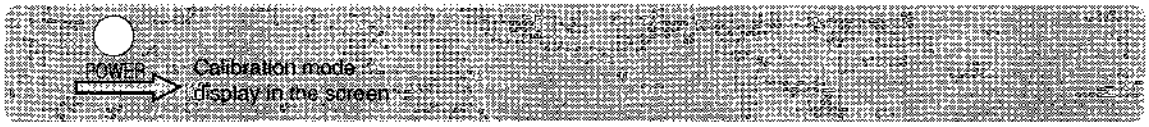
*Means [TIME] is being displayed.

Note

The measurement items for the U-21 model and the U-22 model are indicated with ○ and ●, respectively. "*Measurement item setting*" on page 77 explains how to set the display so items are not displayed.

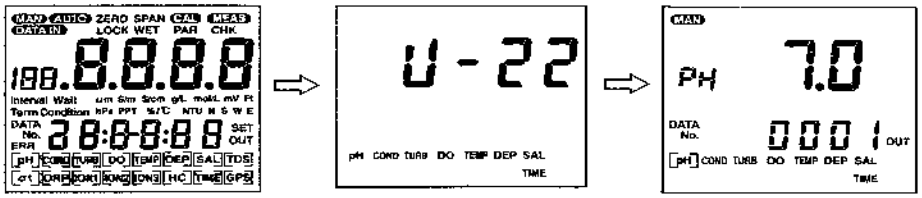
3.2 Operation procedure

3.2.1 Power ON



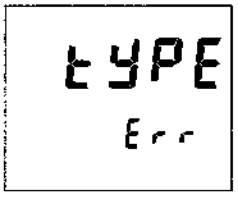
1. Press the **POWER** key.

The display will change in the order of All segment display → Sensor detector display → pH Measurement mode.



For U-22 model

With the sensor probe is not connected,



is displayed.

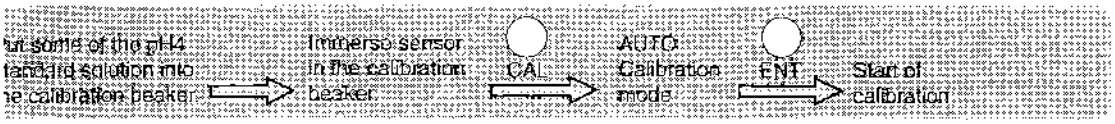
Before turning ON the instrument, connect the sensor probe properly.

.2.2 AUTO calibration method

obtain correct measurement, it is necessary to calibrate the sensor using the standard solution before performing measurement.

Note

In the AUTO calibration mode, the pH, COND, and TURB sensors are calibrated in the pH4 standard solution, and the DO and DEP sensors in the atmosphere simultaneously.
Values may be unstable if there is temperature fluctuation. Calibrate after waiting for about an hour.

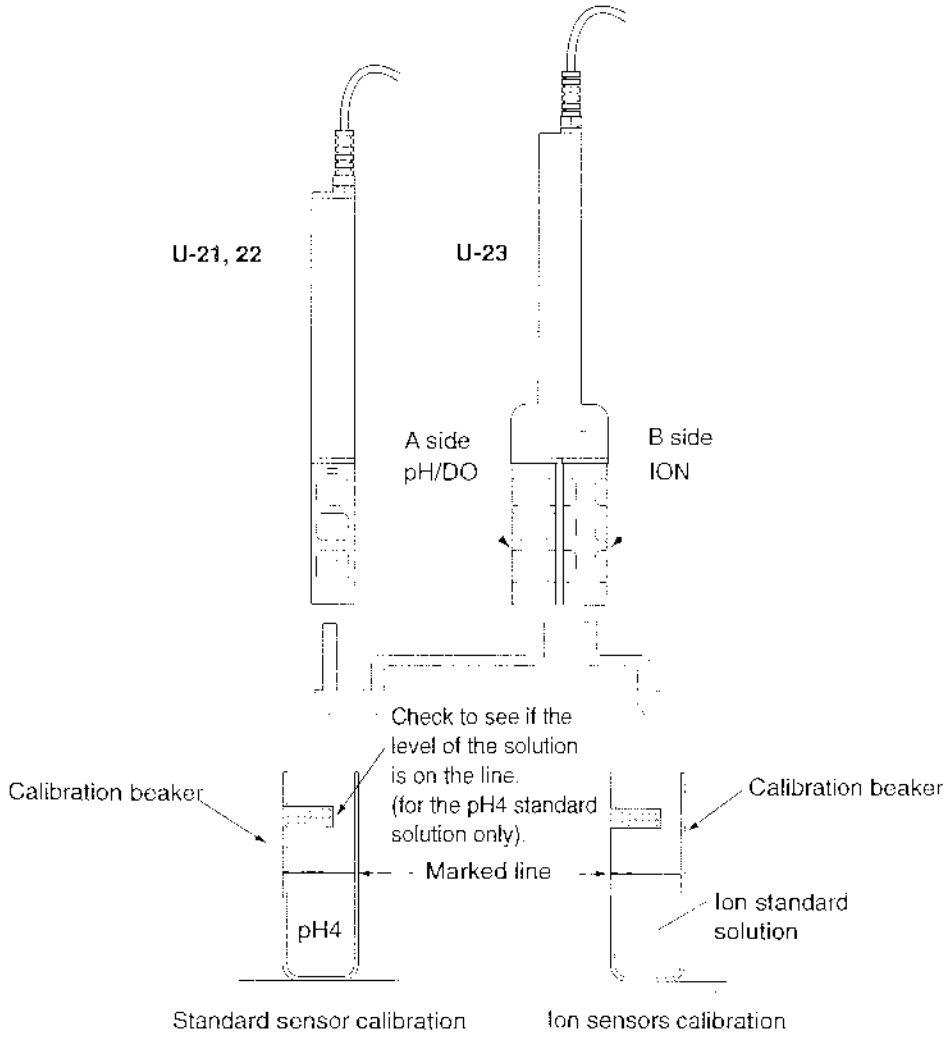


librate using the following procedure.

- Wash the sensor in distilled water a few times and put some of the pH4 standard solution into the calibration beaker to the marked line. Then immerse the sensor in it.
  For the U-23 model, immerse the sensor A side.

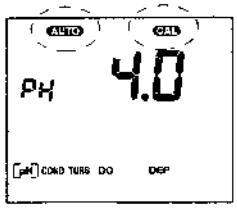
Important

Use the label on the calibration beaker and check to see if the level of the calibration solution is on the label line.



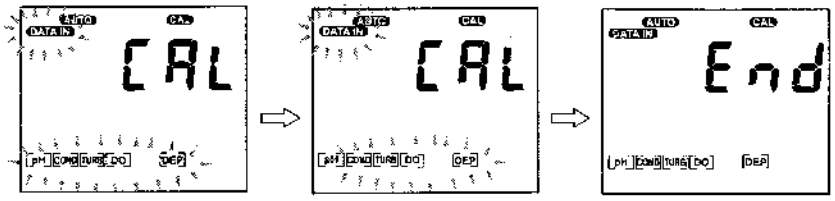
2. Press the **CAL** key in one of the Measurement modes pH, COND, TURB, DO and DEP.

AUTO and **CAL** appear and the instrument enters the AUTO Calibration mode.



3. Press the **ENT** key to start AUTO Calibration.

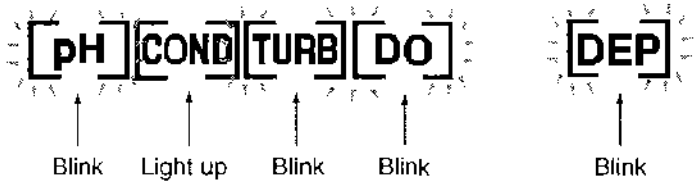
Upon completion of all of the pH, COND, TURB, DO, and DEP modes, **End** will be displayed.
During calibration, **DATA IN** and [ ] for the selected measurement item blink. [ ] light up for the item of which calibration is finished.



End of calibration

With DATA IN is blinking
To stop calibrating the sensor .... press the CAL key.
To establish the calibration ..... press the ENT key.

Example: When COND calibration is finished:
[ ] for [COND] stops blinking and light up steadily.



Note

- For the U-21 model, measurement of DEP is not performed, and therefore calibration of DEP is not performed.
- [ ] continues to blink because calibration is not performed for the item for which an error has happened. If two or more errors happen, an error with a smaller number appears. (See pages 87 to 89 for these errors and ways to solve them.) These calibration errors disappear when the sensor is calibrated properly again, or when the instrument is turned ON again.
- Calibration should be performed for three minutes. When the indications become stable, calibration should be finished.

4. Press the **MEAS** key to return to the Measurement mode.

Important

- Neutralize any basic pH 4 fluids before disposal.

AUTO calibration of the ion sensors (U-23 model only)

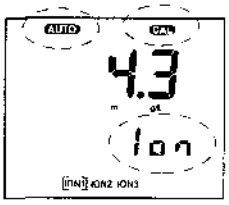
Standard supplied ion sensors (Cl^-, NO_3^-, and Ca^{2+}) allow AUTO calibration to be performed. If any optional ion sensors is used for measurements, be sure to set the ion valency described on page 72 and calibrate the optional sensor manually.

Important

Ion sensors take time to give stable indications. Therefore, immerse the ion sensors in the sample for approximately one hour. Then calibrate the ion sensors and perfom measurements.

- Wash the sensor in distilled water a few times and put some of the supplied ion standard solution (#130) into the calibration beaker to the marked line. Then immerse the B side of the sensor in it.
- Enter ion measurement mode 1, 2 or 3.
- Press the **CAL** key.

AUTO, **CAL**, and "Ion" below them appear. The instrument then enters the AUTO Calibration mode.

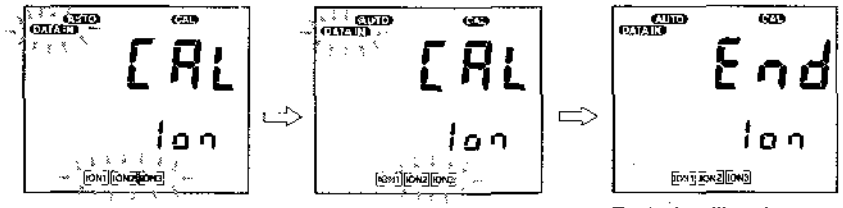


Important

Only the standard supplied ion sensors (Cl^-, NO_3^-, and Ca^{2+}) can be calibrated automatically in the supplied ion standard solution (#130).

- Press the **ENT** key to start AUTO calibration.

Upon completion of the AUTO calibration of all the ion sensors ION1, ION2, and ION3, End will be displayed.



End of calibration

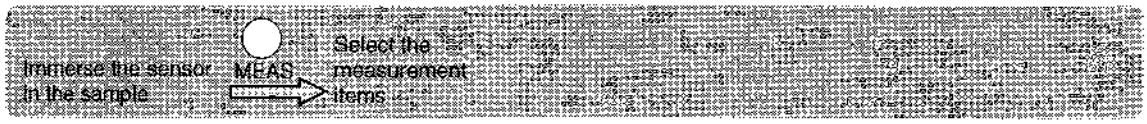
With DATA IN is blinking
To stop calibrating the sensor .... press the CAL key.
To establish the calibration ..... press the ENT key.

- Press the **MEAS** key to return to the Measurement mode.

Important

When the AUTO calibration is performed on the ion sensors, the data for the ion sensor calibrated manually is erased.

3.2.3 Measurement



1. Immerse the sensor in the sample.

2. Select the measurement item.

Use the MEAS key to switch measurement items in the following order:

For model U-21

pH → COND → TURB → DO → TEMP → SAL → TDS → σ_t → TIME ... then back to pH.

For model U-22

pH → COND → TURB → DO → TEMP → DEP → SAL → TDS → σ_t → ORP → TIME ... then back to pH.

For model U-23

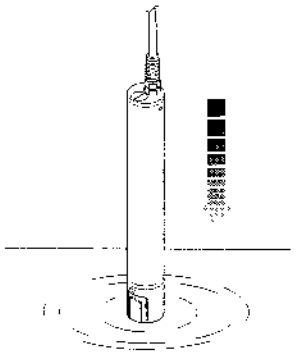
pH → COND → TURB → DO → TEMP → DEP → SAL → TDS → σ_t → ORP → ION1 → ION2 → ION3 → TIME ... then back to pH.

Note

- [GPS] lights up when the optional G.P.S. sensor is connected to the instrument and position information is received from the G.P.S. sensor.
- The above measurement items can be changed by setting "*Measurement item setting*" described on page 77.

Important

- When immersing the sensor probe in the sample, slowly lower the sensor probe into the sample.



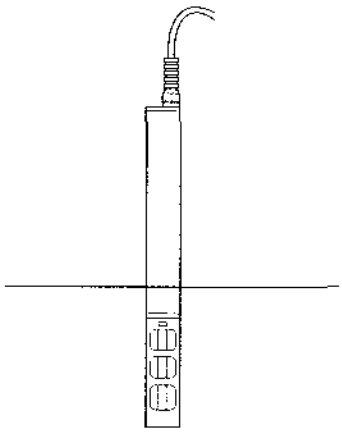
Two useful uses of the U-20 Series models

Making measurements

. Manually storing the measurement data after checking the indication becomes stable

Example: After switching measurement items with the MEAS key, you can then store the measurement data after checking the indication becomes stable.

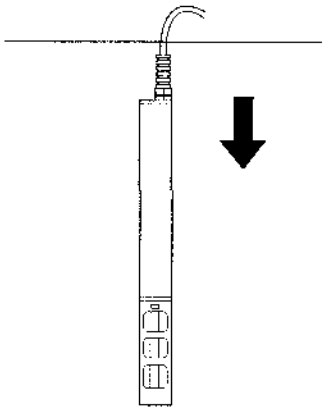
(☞ 4.1 *Manual storage of data while monitoring the measurement data*, page 32.)



. Storing data

Example: Data can be stored continuously at constant intervals from the start of the automatic data storage. This function is useful in obtaining data in depth direction and in storing data continuously.

(☞ 4.2 *Automatic data storage*, page 34.)



Notes in obtaining data on depth

When the instrument is placed at a depth of 100 m or more, the instrument may be broken.

In measurements on the model U-23, the Ca^{2+} and NH_4^+ ion sensors can be used only at depth up to 15 m, and the K^+ ion sensor only at depth up to 3 m. This is because of the properties of the responsive membrane.

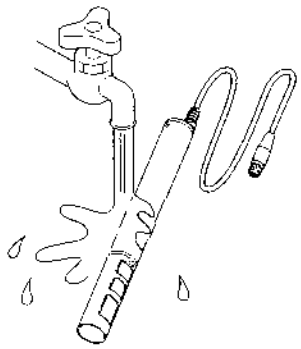
Notes for reliable measurements

Any sensor contamination may affect measurements. Use the AUTO calibration mode to check for contamination on sensors about once a week.

3.2.4 After completion of measurement

1. Turn the power to the instrument off.

2. Use tap water to completely wash off the sample on the sensor and then wipe waterdrops.



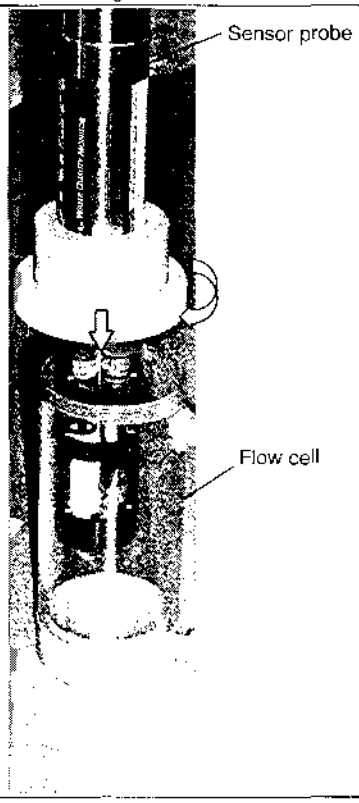
3. Put distilled water into the calibration beaker to the marked line. Then attach the calibration beaker to the sensor probe and store the probe assembly in the carrying case.

Important

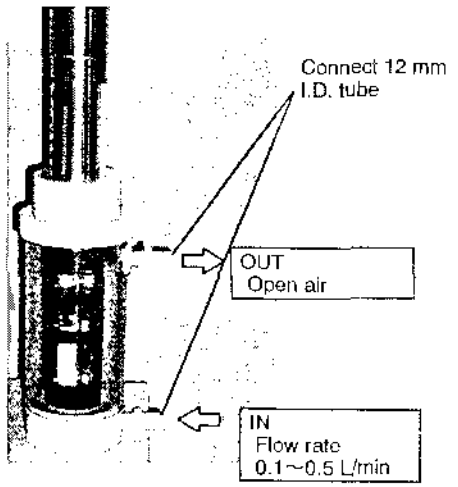
- Do not put water in the calibration beaker before attaching it to the ion sensor end (B side) of U-23.

Now you have read the description for performing measurements. For further information on how to use the instrument, refer to the chapters hereafter.

5. Put the sensor probe to the flow cell, and tightn the retaining nut.



6. Connect 12 mm I.D. tubes to the hose joints. then setting is completed.



Note▸▸▸ The setting sensor probe is movable vertically for about 20 mm. Use at the position where a gap is largest between the tip of protective tube and the bottom of flow cell.

- **Confirmation of Flow rate**

  1. Flow the sample through the flow cell.
  2. Spill effluent into your graduated cylinder for determinated time.
  3. Convert the amount of the effluent into the flow rate per 1 minute.

For any question regarding this product, please contact your local agency, or inquire from the Customer Registration website (www.horiba.co.jp/register).

HORIBA, Ltd.

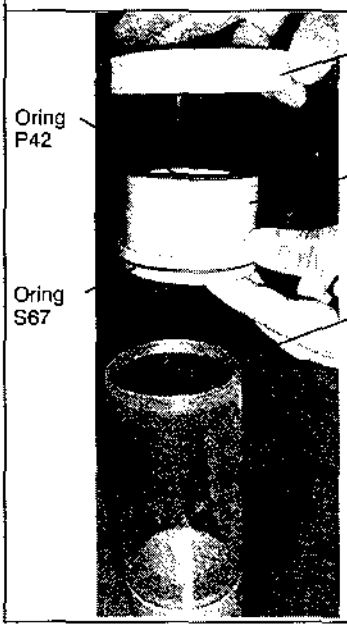
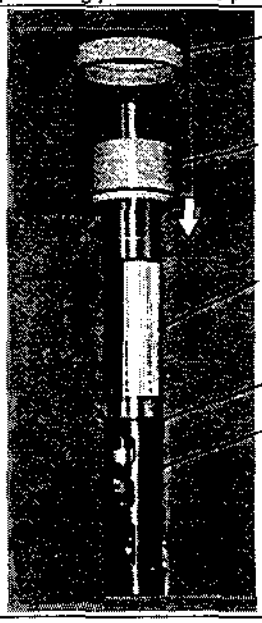
HORIBA

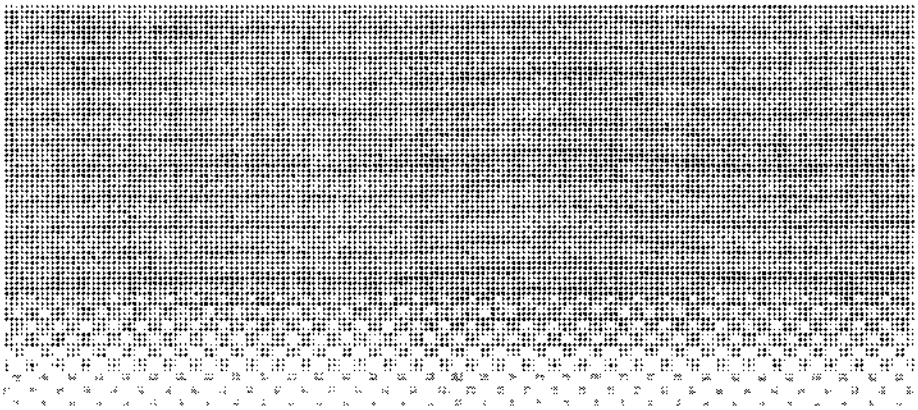
U-20, W-20 Series Flow cell INSTRUCTION MANUAL

Model: W-2100

- **CAUTION IN HANDLING**
  - This flow cell is applicable to the sensor probe of W-22, U-21 and U-22.
  - Connect the 12 mm I.D. tubes to the inlet and outlet hose joints .
  - Do not to bend tubes and not to put things on to avoid causing clog.
  - It is necessary to keep the sensor probe standing vertically on measuring.
  - Flow rate: 0.1 L/min to 0.5 L/min. Sample outlet is to be open air.
  - Sample temperature: 0°C to 45°C
  - Sample is to be including no solvent(Material: PVC).
    Clean the flow cell with water, do not use organic solvent.
  - All the while setting up and measuring, the protective tube should be attached with the sensor probe. Do not hold the protective tube when attaching with the sensor probe or removing it.

- **SET UP**

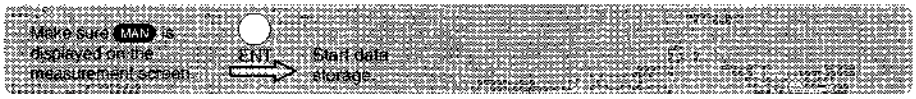
1. Roosen the retaining nut, and remove the block from the flow cell.	2. Disconnect the cable plug from the meter. 3. Feed the cable through the block and the retaining nut. 4. Slide the top of the sensor probe into the block until the block bottom reaches to the protruding portion on the protective tube.
	



4. Using the data memory function

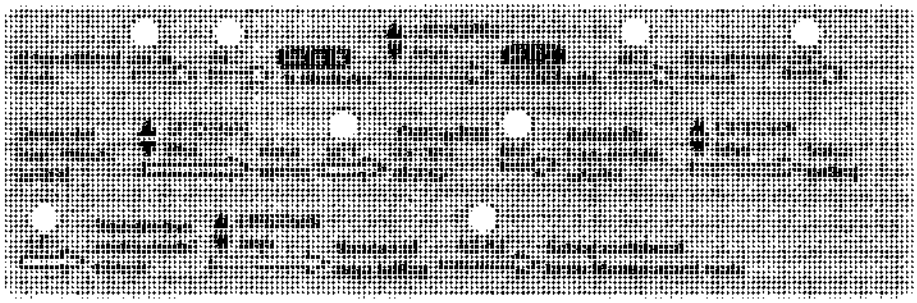
The data memory function can be used to store manualy measurement values with associated data numbers and to store automatically measurement values at fixed intervals (data logger).

4.1 Manual storage of data while monitoring the measurement data 32



4.2 Automatic data storage (U-22, U-23 models) 34

4.2.1 Data memory conditions settings (U-22, U-23 models) 34

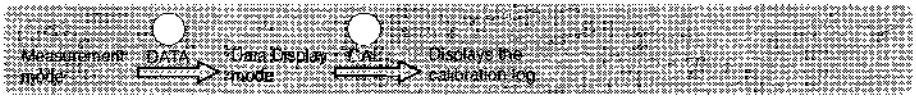


4.2.2 Start of automatic data storage (U-22, U-23 models) 37

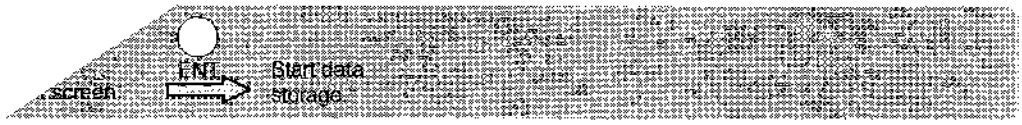
4.3 Calling up data from the memory 39

4.3.1 Calling up measurement data 39

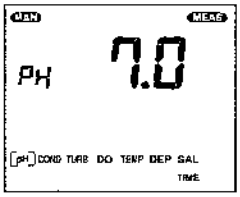
4.3.2 Calling up the calibration log 41



ɔrage of data while monitoring the
ɔt data

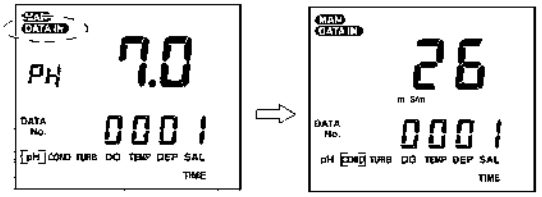


1. Make sure that **MAN** is displayed on the Measurement mode.



2. Press the **ENT** key.

Data storage starts, **DATA IN** and the data No. are displayed on the screen, and the measured value to be stored and the measurement item are displayed in order at approximately 0.5 second intervals.



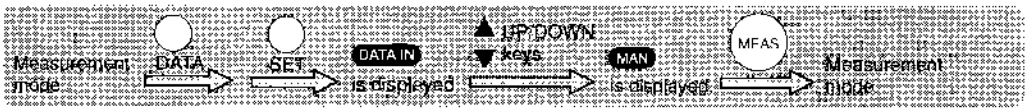
All measurement items and times are stored in sequence.

After the data is stored in memory, the screen returns to the original Measurement mode.

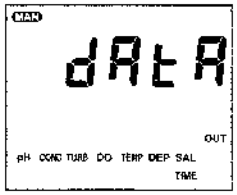
Note

- Up to 2880 sets of data can be stored in the memory.
  When 2880 sets of data have been stored in the memory, ERR 9 appears and no more data can be stored. In this case, "*Data memory clear*" while referring to page 79, and you can store new data in the memory.

- If **MAN** is not displayed (**AUTO** is displayed) in the Measurement mode. (U-22, U-23 models)

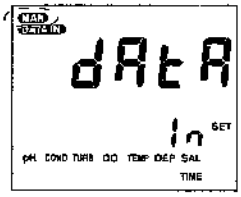


(1) Press the **DATA** key in the Measurement mode.



(2) Press the **SET** key.
DATA IN is displayed.

(3) Press the **UP/DOWN** (▲ ▼) keys to display MAN.

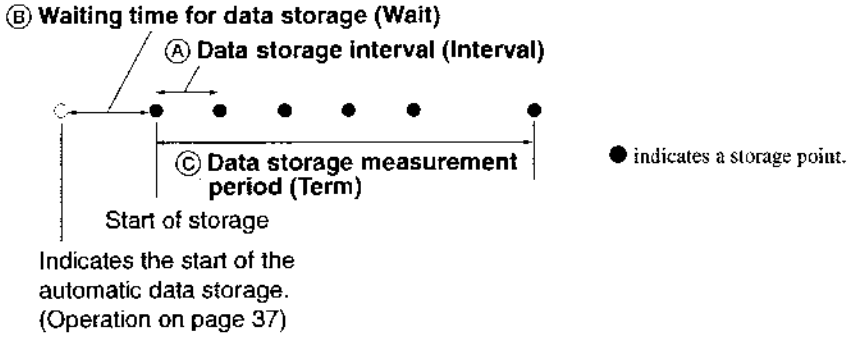


(4) Press the **MEAS** key to return to the Measurement mode.

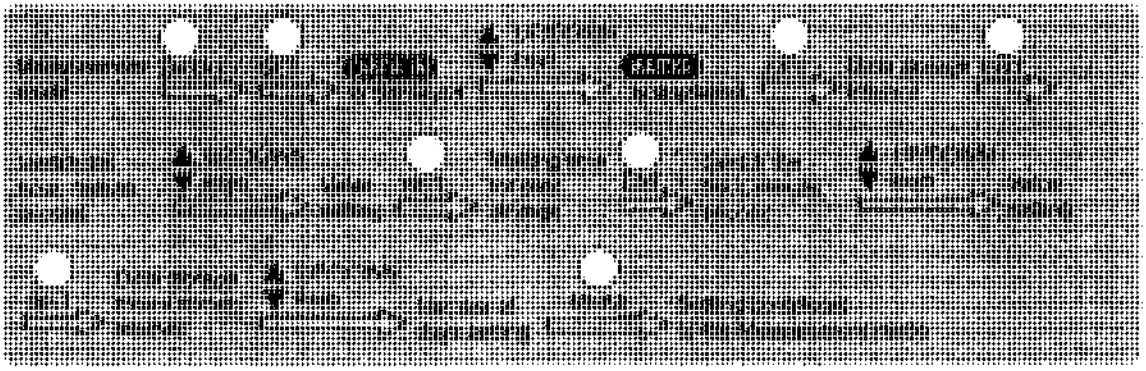
4.2 Automatic data storage (U-22, U-23 models)

Measured values are stored automatically at constant time intervals. Before using the automatic storage, the following condition settings are required:

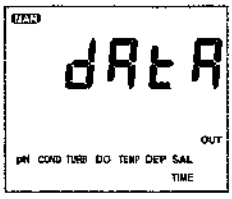
- Setting of data storage interval (4.2.1, step 4)
- Setting of waiting time for data storage (4.2.1, step 6)
- Setting of the data storage measurement period (4.2.1, step 8)



4.2.1 Data memory conditions settings (U-22, U-23 models)



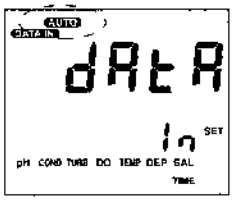
1. Press the **DATA** key in the Measurement mode.



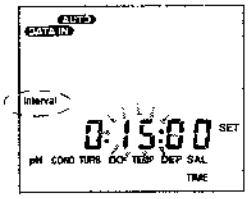
2. Press the **SET** key.

DATA IN is displayed.

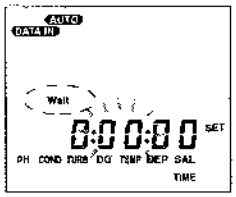
3. Press the **UP/DOWN** (▲ ▼) keys to display **AUTO**.



- Press the **SET** key to display the screen for setting the data storage interval Ⓐ.
  "**Interval**" is displayed.
- Press the **ENT** key to switch the among "hour", "minute" and "second" and set the value using the **UP/DOWN** (▲ ▼) keys.
  (Data storage intervals can be set to 2 seconds to 24 hours.)
  The current setting location will blink.

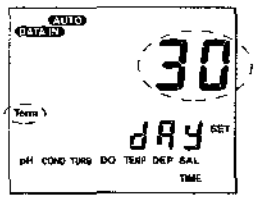


- Press the **SET** key to display the screen for setting the waiting time for data storage Ⓑ.
  "**Wait**" is displayed.
- Press the **ENT** key to switch among "hour", "minute" and "second" and set the value using the **UP/DOWN** (▲ ▼) keys.
  (The waiting time for data storage can be set to 0 second to 24 hours.)
  The current setting location will blink.



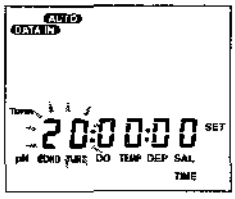
- Press the **SET** key to display the screen for setting the data storage measurement period Ⓒ (number of days).
  "**Term**" is displayed.

9. Use the **UP/DOWN** (▲ ▼) keys to set the value (number of days).



Setting of less than 24 hours

First set the number of days to zero then press ENT key to select the "hour/minute/second" setting. Use the UP/DOWN (▲ ▼) keys to set the hour, the minute and second. During setting, the number to be set blinks.

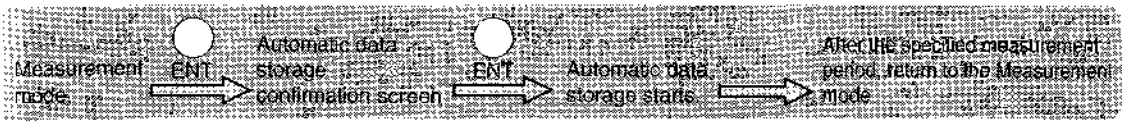


Note

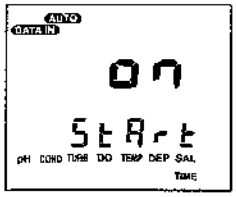
- Press the SET key to return to step 4.

10. When the **MEAS** key is pressed, setting will be completed and the instrument will return to the Measurement mode.

4.2.2 Start of automatic data storage (U-22,U-23 models)



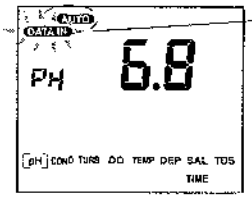
1. Make sure that **AUTO** is displayed on the Measurement mode.
2. Press the **ENT** key. A confirmation screen will be displayed asking if you wish to start automatic data storage.



Note

If you do not wish to proceed with automatic data storage, press the CAL key to return to the Masurement mode.

3. Press the **ENT** key to start automatic data storage.



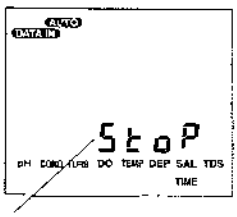
While **DATA IN** is blinking, the automatic data storage is being executed.

Note

During the automatic data storage, measurement items can be switched by pressing the MEAS key.

Important

During the automatic data storage, the ENT, SET, and DATA keys do not function and therefore calibration, setting change and stored data display cannot be performed.
To stop automatic data storage, press the CAL key.



Confirmation display for canceling automatic data storage appears.
To stop the automatic data storage ..... Press the ENT key.
To return to the screen for the automatic data storage ... Press the DATA key.

4. After the specified measurement period, DATA IN disappears and the instrument returns to the normal Measurement mode.

Note

- When the instrument is turned on, AUTO lights up and DATA IN blink if automatic data storage is being performed with the sensor probe.

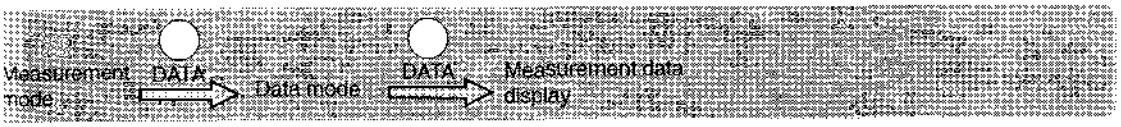
Notes for automatic data storage

- For long-term data storage, replace the sensor probe battery with a new one.
- You can remove the connector from the main unit. It can still be used for up to 60 hours at room temperature with the battery in the sensor probe (alkaline battery). Life is reduced by approximately one half when manganese batteries are used.
- If the sensor probe is connected to the instrument for monitoring, the instrument battery is first consumed to protect the memory of the sensor.
- When 2880 sets of data have been stored in the memory, ERR 9 appears and no more data can be stored. The automatic data storage is automatically ended and the instrument returns to the normal Measurement mode.
- Because ion sensors need to be calibrated once a week in measurements on the U-23 model, do not automatic data storage in the memory for more than seven consecutive days.

.3 Calling up data from the memory

.3.1 Calling up measurement data

:ading out data that has been stored manually or automatically.

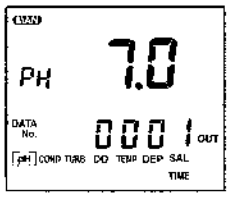


- Press the **DATA** key in the Measurement mode.

  The instrument goes to the DATA mode.

- Press the **DATA** key.

  The measurement data is displayed.
  Data you want to call can be displayed by selecting a measurement item and data No.



DATA key ..... Selects switching of measurement item or memory data No.
When switching measurement items: Measurement item blinks.
When switching data No. : Data No. blinks.

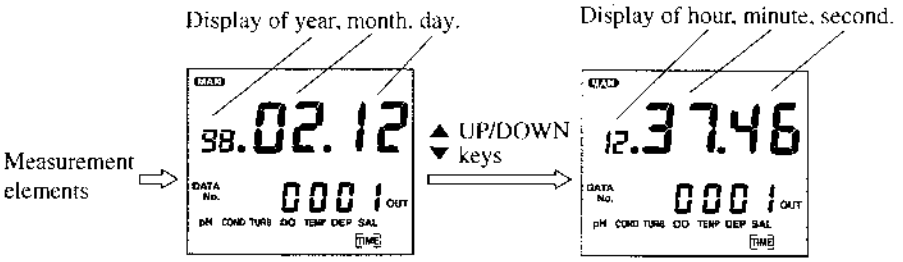
UP/DOWN (▲▼) keys ....Switch measurement item or No. which has been selected with the DATA key.

Note

If you push the CAL key, only the data numbers will be displayed, allowing rapid changing of the numbers. Push the UP/DOWN (▲▼)keys to find the number, then press the SET key to display the data.

TIME data

Use the UP/DOWN (▲▼) keys to switch between "Yer, Month, Day" and "Hour, Minute, Second".

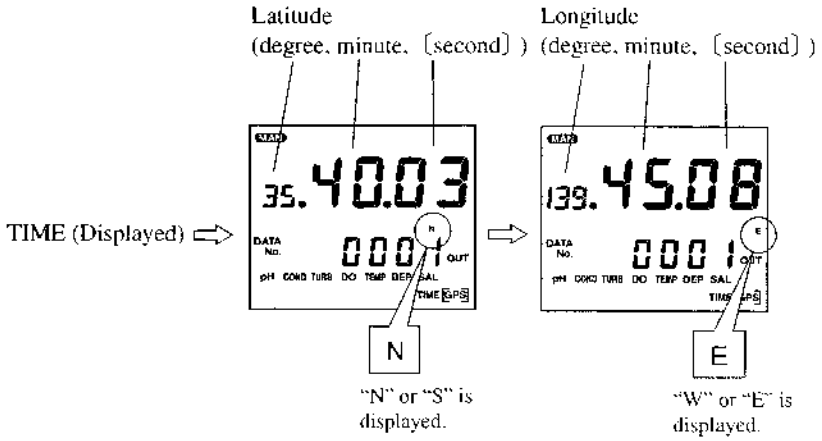


Note

- The time in the automatic memory can be out by about 2 seconds.

G.P.S. data (only when G.P.S. data is present)

Use the UP/DOWN (▲ ▼) keys to switch between "Latitude" and "Longitude".



Latitude N → The North latitude S → The South latitude
Longitude E → The East longitude W → The West longitude

ENT key ..... Prints all measurement data for the displayed memory data item.
(when the printer is connected to the instrument)

Useful uses of keys in automatic storage

SET + UP (▲) key ..... Displays the first part of the next data automatically stored.
SET + DOWN (▼) key ..... Displays the first part of the previous data automatically stored.
If there is manual data, then the previous or next manual data is shown.

Display for automatic storage

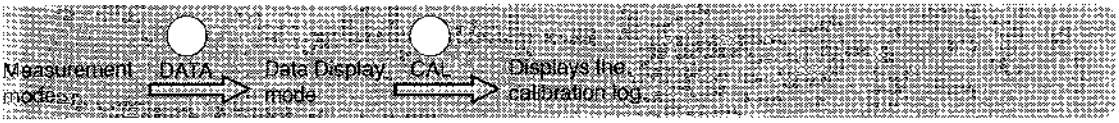
For the first and last data in one session of automatic storage the following identification marks are displayed in front of the values representing the data Nos.:
[: displayed for the first data in automatic storage.
] : displayed for the last data in automatic storage.

Note

- When the MEAS key is pressed, data calling is stopped and the instrument returns to the Measurement mode.
- Data is called from the sensor probe so to get one piece of data takes about one second.

.3.2 Calling up the calibration log

calibration log is a record containing the "year, month, day" and "hour and minute" of the last calibration of dividual measurement items and their calibration method. The instrument automatically stores the calibration log.

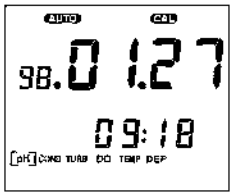


- Press the **DATA** key in the Measurement mode.

  The instrument goes to the DATA Display mode.

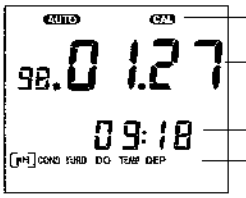
- Press the **CAL** key.

  The calibration log is displayed.



UP/DOWN (▲▼) keys: Switch the measurement item.
ENT key: Prints the entire calibration log. (when the printer is connected to the instrument)

Calibration log.



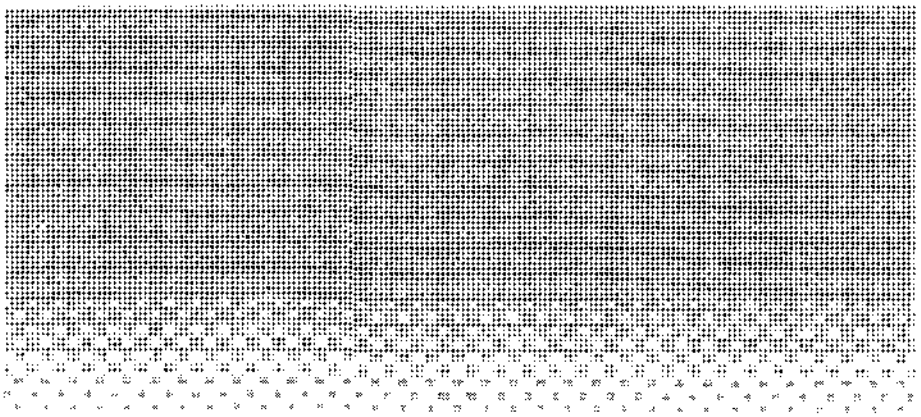
Calibration method

AUTO CAL	: AUTO calibration
MAN ZERO CAL	: Manual zero calibration
MAN SPAN CAL	: Manual span calibration
MAN ZERO SPAN CAL	: Manual zero calibration and span calibration

Note

Press the MEAS key to abort the data calling and return to the Measurement mode.

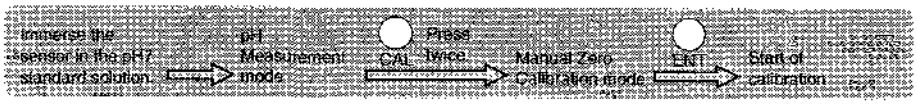
MEMO



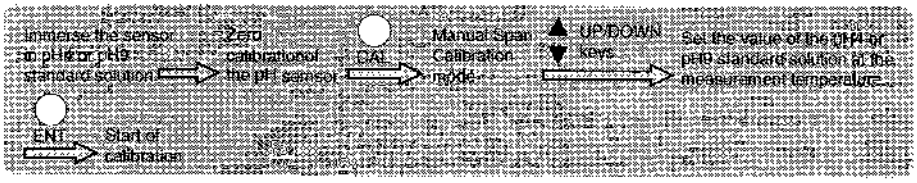
5. Techniques for more accurate measurement

In normal operation, calibration using the AUTO Calibration mode described earlier in the basic operation section provides sufficient accuracy. However, for more accurate measurement, manual calibration is effective. When measurement with high-accuracy extended display is needed, be sure to perform manual calibration.
Attention: The extended display mode is entered automatically when manual calibration is selected.

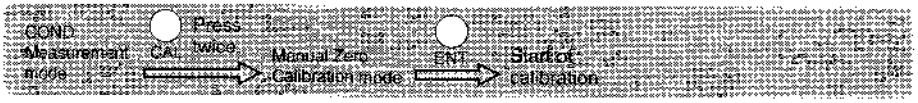
5.1 Manual pH calibration 45
5.1.1 Zero calibration 45



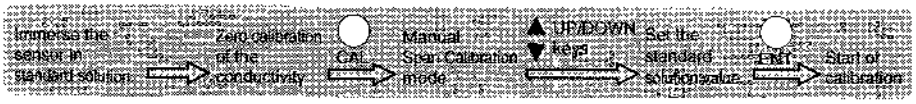
5.1.2 Span calibration 46



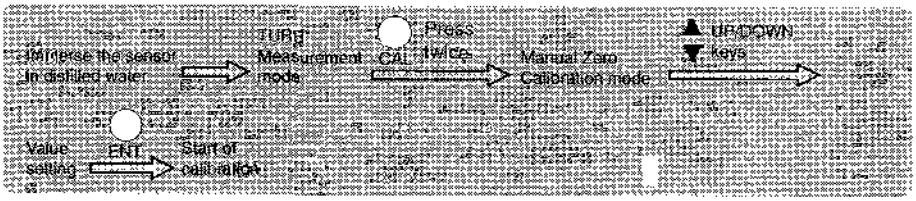
5.2 Manual conductivity (COND) calibration 47
5.2.1 Zero calibration 47



5.2.2 Span calibration 48

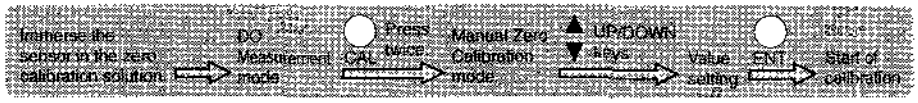


5.3 Manual turbidity (TURB) calibration 50
5.3.1 Zero calibration 50



5.3.2 Span calibration 51

5.4 Manual Dissolved-Oxygen (DO) calibration 52
5.4.1 Zero calibration 52



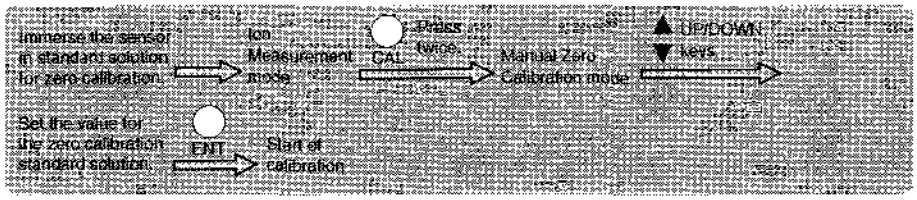
5.4.2 Span calibration 53

5.5 Water depth (DEP) calibration (U-22, U-23 models) 55
5.5.1 Zero calibration 55

5.5.2 Span calibration 56

5.6 Temperature (TEMP) calibration 57

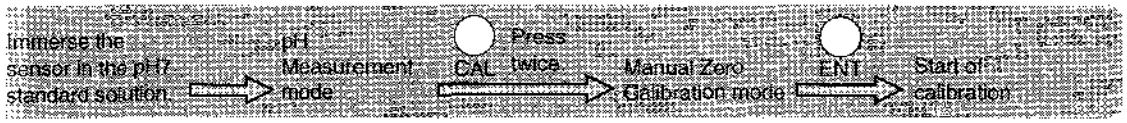
5.7 Manual ion calibration (U-23 model) 58
5.7.1 Preparation of calibration solution 58
5.7.2 Zero calibration 59



5.7.3 Span calibration 60

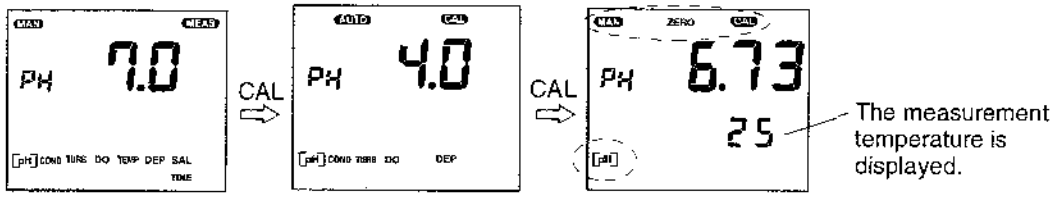
5.1 Manual pH calibration

5.1.1 Zero calibration



- Wash the sensor two or three times using distilled water, then pour some pH7 standard solution into the calibration beaker, and immerse the sensor in it. (For the U-23, immerse the sensor A side.)
- Press the **CAL** key twice in the pH Measurement mode.

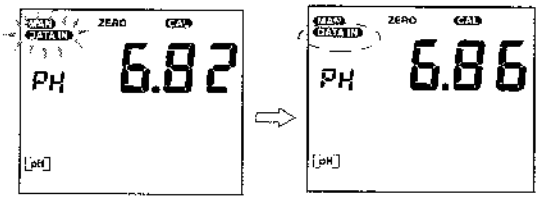
  When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.



Manual Zero Calibration mode

- Use the **UP/DOWN** (▲ ▼) keys to input the value for the pH7 standard solution at the measurement temperature.
  (☞ *8. Reference data* page 96.)
- Press the **ENT** key.

  The manual zero calibration starts.



End of calibration

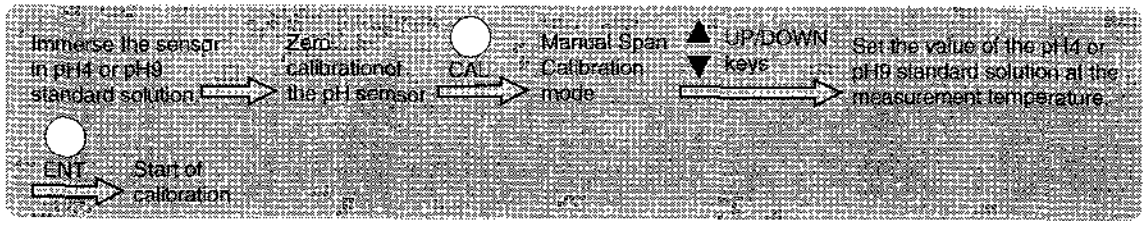
The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

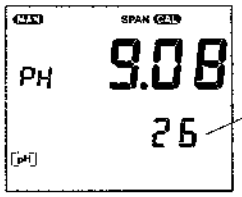
5.1.2 Span calibration



1. Wash the sensor two or three times using distilled water, then pour some pH4 or pH9 standard solution into the calibration beaker, and immerse the sensor in it. (For the U-23, immerse the sensor A side.)

2. After the zero calibration of the pH sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

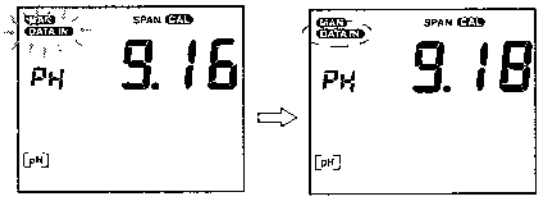
3. Use the **UP/DOWN** (▲ ▼) keys to set the value for the pH4 or pH9 standard solution at the measurement temperature.



The measurement temperature is displayed.

4. Press the **ENT** key.

The manual span calibration starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

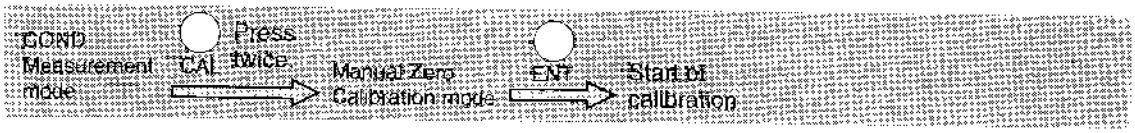
Note

- When the SET and CAL keys are pressed during the manual pH calibration mode, the calibration data for the pH sensor can be deleted.

5.2 Manual conductivity (COND) calibration

The U-20 series models can measure conductivity (COND) in the range from 0 to 10 S/m. Depending on the concentration of the sample, these models automatically select the most suitable measuring range from three ranges: 0 to 100 mS/m, 0.1 to 1 S/m, and 1 to 10 S/m. The zero point is common to the three measuring ranges.

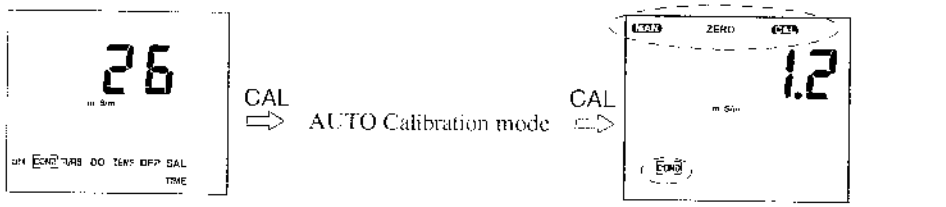
5.2.1 Zero calibration



1. Wash the conductivity (COND) sensor two or three times using distilled water. Completely remove the water on the sensor and calibrate the instrument in the atmosphere.

2. Press the **CAL** key twice in the Conductivity (COND) Measurement mode.

When the instrument enters the Manual Zero Calibration mode. **MAN**, **ZERO** and **CAL** light up.

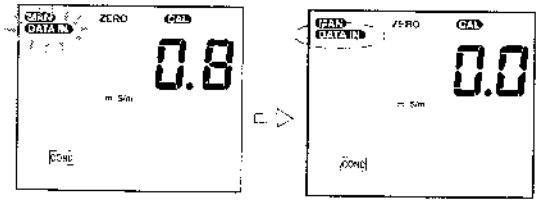


Manual Zero Calibration mode

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5.2.2 Span calibration

Preparation of calibration solution (Potassium chloride (KCl) standard solution)

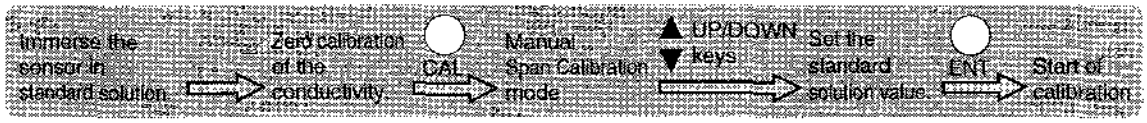
Dry Potassium chloride (KCl) powder (high-grade commercially available) at 105°C for two hours, and leave it to cool in a desiccator.
Consult the following table and measure a portion of potassium chloride (KCl), then prepare standard potassium chloride (KCl) solution following the procedure below.

Potassium chloride (KCL) standard solution	Conductivity (COND) value	Potassium chloride (KCl) weight (g) at solution temperature of 25°C	Calibration range
0.005 mol/L	71.8 mS/m	0.373	0.0 to 99.9 mS/m
0.050 mol/L	0.667 S/m	3.73	0.090 to 0.999 S/m
0.500 mol/L	5.87 S/m	37.2	0.90 to 9.99 S/m

1. Dissolve the weighed Potassium Chloride (KCl) in distilled water.
2. Put the dissolved Potassium Chloride (KCl) into a 1L measuring flask, and fill to the 1L mark with distilled water.

Calibration procedure

Perform the span calibration using the three types of standard solution as follows.

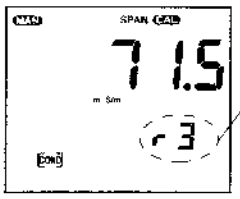


Important

- Set the temperature of the span standard solution to 25 ± 5°C.
- The sensor should be calibrated in the three standard solutions in the order of increasing concentration.

1. Wash the sensor two or three times using distilled water, then pour some standard solution into the calibration beaker, and immerse the sensor in it. (In the case of the U-23 model, immerse the sensor A side.)
2. After the zero calibration of the conductivity (COND) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
   MAN, **SPAN** and **CAL** light up.

4. Use the **UP/DOWN** (▲ ▼) keys to set the standard solution value.



The calibration range is displayed.

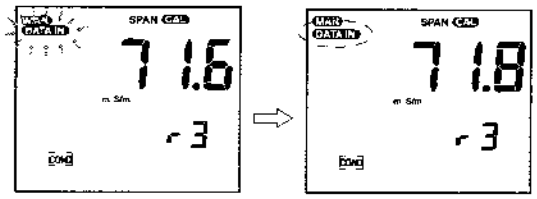
Note

The sensor automatically identifies the calibration solution and the relevant calibration range is displayed.

r 1 : 0.90 to 9.99 S/m
r 2 : 0.090 to 0.999 S/m
r 3 : 0.0 to 99.9 mS/m

5. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

6. Press the **CAL** key and use each standard solution and perform steps 1 to 4 above for calibration.

7. Press the **MEAS** key to return to the Measurement mode.

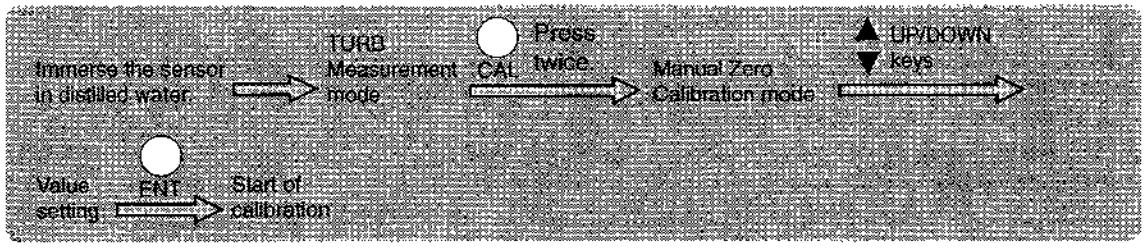
Note

When the SET and CAL keys are pressed during the manual Conductivity (COND) Calibration mode, the calibration data for the conductivity (COND) sensor can be deleted.

5.3 Manual turbidity (TURB) calibration

5.3.1 Zero calibration

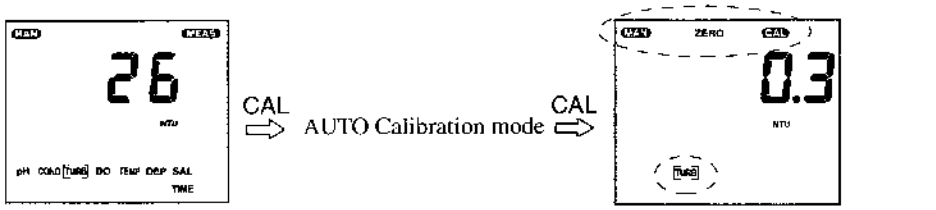
In zero calibration, distilled water is used as a calibration solution. If you cannot obtain distilled water, you may use ion exchange water, which can be considered to have a turbidity of zero.
When the turbidity (TURB) sensor is calibrated, it is particularly important that the probe is completely contamination-free. Do not use a contaminated probe. Otherwise unreliable calibration will result.



1. Wash the sensor two or three times using distilled water, then place some distilled water into the calibration beaker, and immerse the sensor in it. (For the U-23, immerse the sensor A side.)

2. Press the **CAL** key twice in the Turbidity (TURB) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

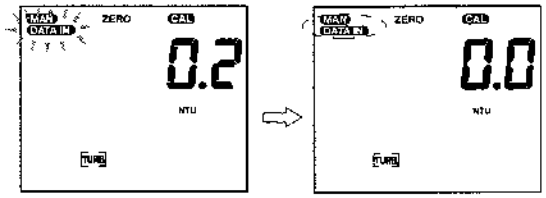


Manual Zero Calibration mode

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is started.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

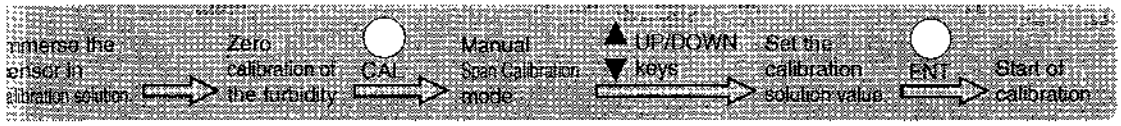
With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

.3.2 Span calibration

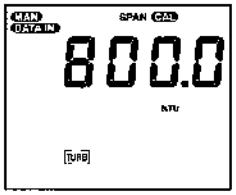
reparation of calibration solution

igh out 5.0 g of hydrazine sulfate, and dissolve it in 400 mL of distilled water. Next dissolve 50 g of hexamethylene ramine in 400 mL of distilled water, and mix the two solutions together. Finally add distilled water until the total ution volume is 1000 mL, and mix well. Store this solution at a temperature of 25 – 3°C for 48 hours. The turbidity lue (TURB) of this solution is equivalent to 4000NTU.
e the solution as span calibration solution for turbidity (TURB) of 800NTU by diluting this solution by a tor of 5 (use a pipette to measure 50 mL of the 4000NTU solution and pour it into a 250 mL measuring flask, d add 200 mL of distilled water).

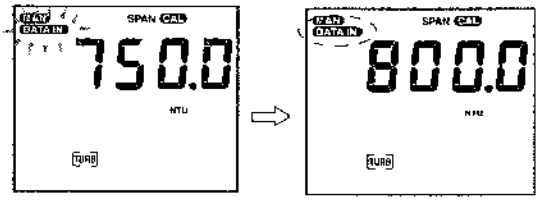
alibration procedure



- Wash the sensor two or three times using distilled water, then pour standard solution into a calibration beaker, and immerse the sensor in it. (For the U-23, immerse the sensor A side.)
- After the zero calibration of the turbidity (TURB) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
  MAN, **SPAN** and **CAL** light up.
- Use the **UP/DOWN** (▲ ▼) keys to set the value to 800.0.



- Press the **ENT** key.
  The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

- Press the **MEAS** key to return to the Measurement mode.

Important

When it is known beforehand that the solution for measurement has a low turbidity (0 to 100 NTU), calibrate the sensor in the span calibration solution of 80 NTU. To prepare an 80 NTU calibration solution, dilute the 4,000NTU calibration solution with distilled water by a factor of 50.

Note

When the SET and CAL keys are pressed during the manual Turbidity (TURB) Calibration mode, the calibration data for the turbidity (TURB) sensor can be deleted.

5.4 Manual Dissolved-Oxygen (DO) calibration

It is necessary to prepare a new calibration solution each time directly before calibration of the Dissolved-Oxygen (DO) sensor.

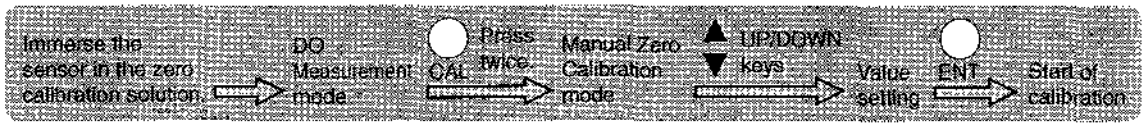
5.4.1 Zero calibration

Use ion exchange water or tap water with sodium sulfite dissolved in it.

Preparation of calibration solution

Add approximately 50 g of sodium sulfite to 1,000 ml of water (either ion exchange water or tap water) and stir the mixture to dissolve the sodium sulfite in it.

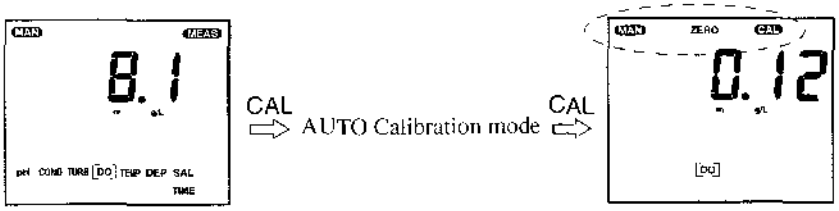
Calibration procedure



1. Use distilled water to wash the sensor a few times. Then fill the calibration beaker (above the marked line) with the zero calibration solution until the DO sensor can be immersed in the solution. Then immerse the sensor (the A side for the U-23 model) in the solution.

2. Press the **CAL** key twice in the Dissolved-Oxygen (DO) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

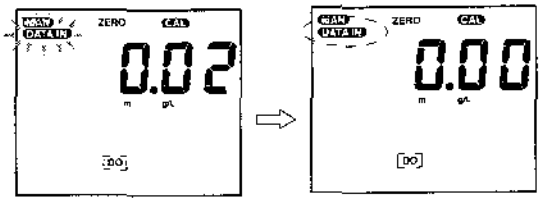


Manual Zero Caliburation mode

3. After the display has stabilized, use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

Important

- After calibration, use tap water to clean the sensor.

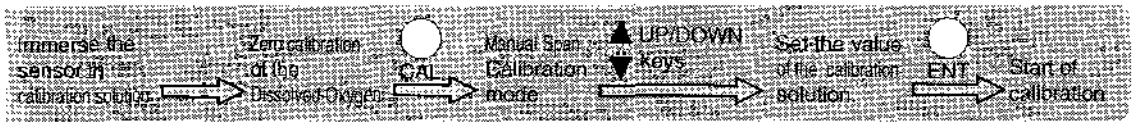
5.4.2 Span calibration

Use ion exchange water or tap water with saturated dissolved oxygen as the span calibration liquid.

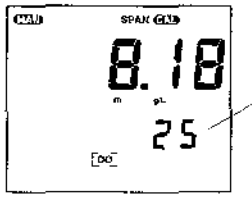
Preparation of standard solution for span calibration

Pour 1 to 2 liters of water into a container (either ion exchange water or tap water). Using a pneumatic pump, feed air into the water and froth up the solution until oxygen is saturated.

Calibration procedure



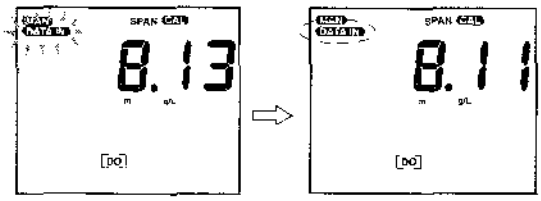
1. Wash the sensor twice or three times and immerse the sensor (the A side for the U-23 model) in the span calibration solution.
2. After the zero calibration of the Dissolved-Oxygen (DO) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
   MAN, **SPAN** and **CAL** light up.
3. After the display has stabilized, use the **UP/DOWN** (▲ ▼) keys to set the amount of saturated dissolved oxygen in water at the temperature.



The temperature setting is displayed.
Refer to the table given on page 54 and set a value equivalent to the amount of saturated dissolved oxygen at the temperature.

4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

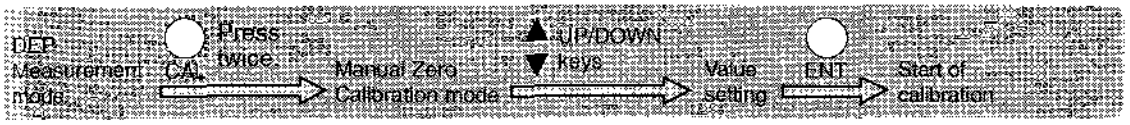
When the SET and CAL keys are pressed during the manual Dissolved-Oxygen (DO) calibration mode, the calibration data for the dissolved-oxygen (DO) sensor can be deleted.

Amounts of saturated dissolved oxygen in water at various temperatures (salinity=0.0%)

Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)
0	14.16						
1	13.77	11	10.67	21	8.68	31	7.42
2	13.40	12	10.43	22	8.53	32	7.32
3	13.04	13	10.20	23	8.39	33	7.22
4	12.70	14	9.97	24	8.25	34	7.13
5	12.37	15	9.76	25	8.11	35	7.04
6	12.06	16	9.56	26	7.99	36	6.94
7	11.75	17	9.37	27	7.87	37	6.86
8	11.47	18	9.18	28	7.75	38	6.76
9	11.19	19	9.01	29	7.64	39	6.68
10	10.92	20	8.84	30	7.53	40	6.59

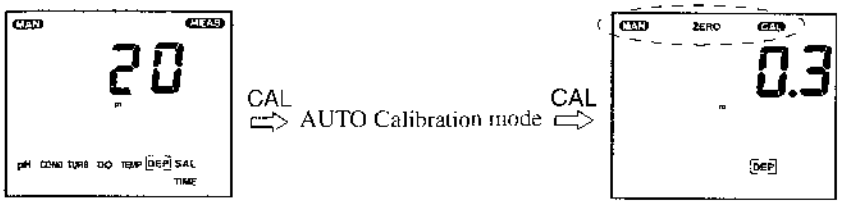
5.5 Water depth (DEP) calibration (U-22, U-23 models)

5.5.1 Zero calibration



1. Immerse the sensor in the sample water for approximately 30 minutes so that sensor probe and sample temperatures become the same.
2. Press the **CAL** key twice in the Water Depth (DEP) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

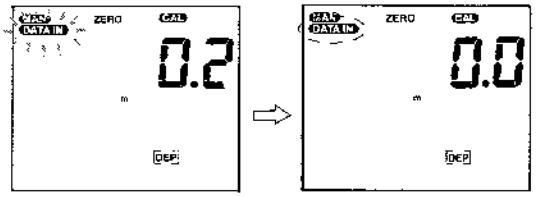


Manual Zero Calibration mode

Immerse the sensor probe so that the storage lid for a memory backup battery is at the water surface.
The water surface level is considered as depth 0 m.

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.
4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

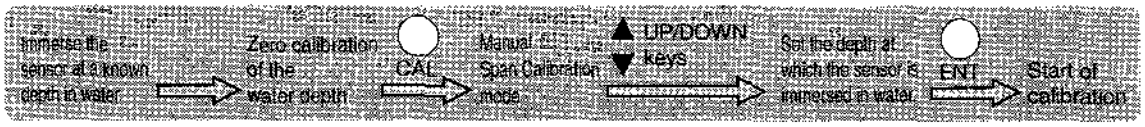
The measured value is displayed during calibration. and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

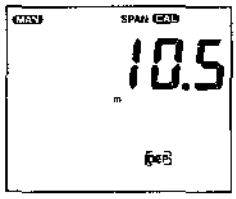
Important

Since the water depth (DEP) sensor depends greatly on temperature, calibrate the sensor at the same temperature as the sample for more accurate measurement.
Use the AUTO Calibration mode because calibration error becomes large when using in a place with flow velocity or where it is shallow.

5.5.2 Span calibration

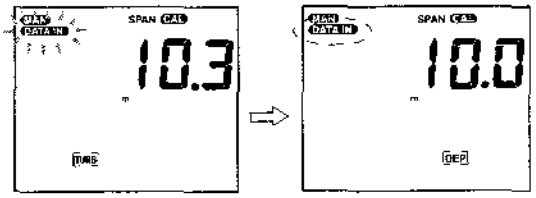


1. Immerse the sensor at a known depth in water. (Set the depth of the lid for memory backup battery as the depth setting.)
2. After the zero calibration of the water depth (DEP) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
   MAN, **SPAN** and **CAL** light up.
3. Use the **UP/DOWN** (▲ ▼) keys to set the depth at which the sensor is immersed in water.



4. Press the **ENT** key.

   The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

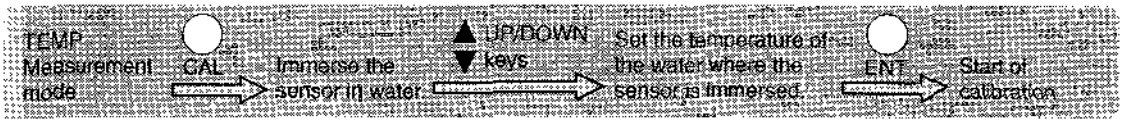
With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

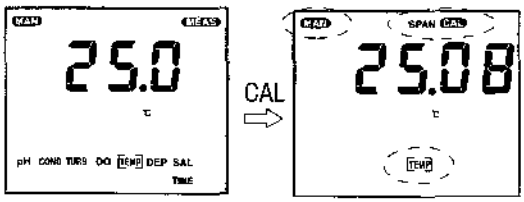
- When the SET and CAL keys are pressed during the manual Water depth (DEP) Calibration mode, the calibration data for the water depth (DEP) sensor can be deleted.

5.6 Temperature (TEMP) calibration



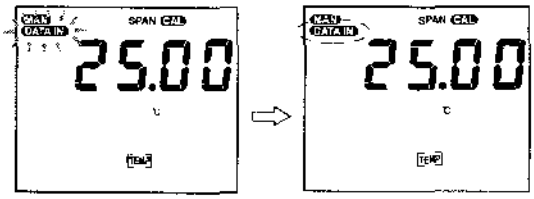
- Press the **CAL** key in the Temperature (TEMP) Measurement mode.

  Select the manual calibration mode.



- Immerse the sensor in water at a known temperature.
- Use the **UP/DOWN** (▲ ▼) keys to set the temperature of the water where the sensor is immersed as a calibration value.
- Press the **ENT** key.

  The manual calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

- Press the **MEAS** key to return to the Measurement mode.

Note

When the SET and CAL keys are pressed during the manual Temperature (TEMP) calibration mode, the calibration data for the temperature (TEMP) sensor can be deleted.

5.7 Manual ion calibration (U-23 model)

It is necessary to prepare a zero calibration solution and a span calibration solution according to the ion sensor to be calibrated. When the supplied standard ion sensors (Cl^-, NO_3^-, and Ca^{2+}) are used, the supplied ion standard solution can be used in common. When other sensors are used, it is necessary to set the ion valency first.
(☞ 6.7 *Changing the ion valency setting*)

5.7.1 Preparation of calibration solution

For standard ion sensors

To prepare a zero calibration solution, dilute the supplied ion standard solution (#130) by a factor of 10 with distilled water. The supplied ion standard solution (#130) is used without dilution as a span calibration solution. The supplied ion standard solution (#130) is used without dilution as a span calibration solution. The zero and span calibration values for the individual ion sensors are as follows:

Meter indication	ION	Zero calibration value	Span calibration value
ION1	Cl^-	3.55 mg/L	35.5 mg/L
	Chloride	(0.1m mol/L)	(1m mol/L)
ION2	NO_3^-	3.10 mg/L	31.0 mg/L
	Nitric acid	(50μ mol/L)	(0.5m mol/L)
ION3	Ca^{2+}	2.01 mg/L	20.1 mg/L
	Calcium	(50μ mol/L)	(0.5m mol/L)

For optional ion sensors

When calibrating any optional sensor, prepare a 0.1 mol/L standard solution first. Then dilute the standard solution to prepare a zero and a span calibration solution.

Preparing a 0.1 mol/L standard solution

Weigh out the individual reagents listed below for each ion type and dissolve each reagent in distilled water to obtain one liter of solution.

Ion type	Ion valency	Reagent (Special grade, commercial)	Weight	Concentration (mg/L)
Fluoride F^-	−1	Potassium fluoride	5.81 g	1900 mg/L
Potassium K^+	+1	Potassium chloride	7.46 g	3910 mg/L
Ammonia NH_3	+1	Ammonium chloride	5.35 g	1800 mg/L

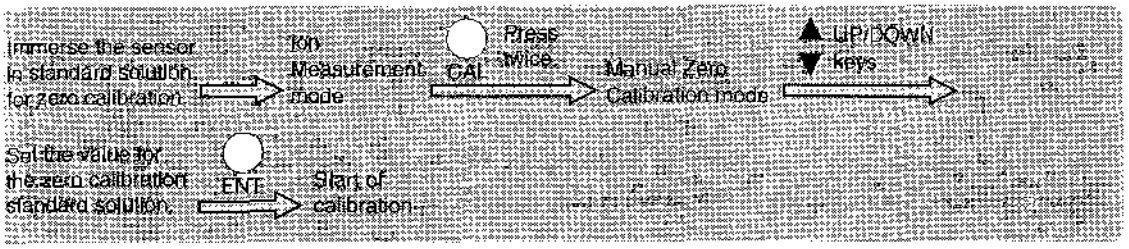
Preparing a calibration solution

To prepare a zero calibration solution and a span calibration solution , dilute the 0.1 mol/L standard solution for each ion type by a factor of 1000 and 100, respectively.

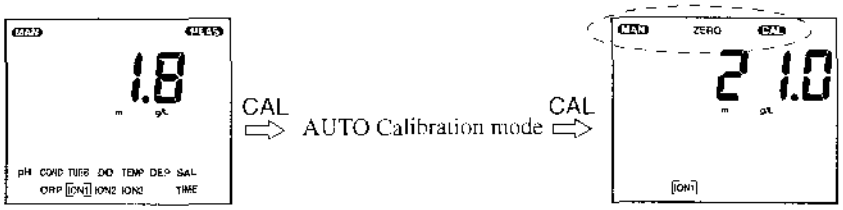
Ion type	Zero calibration solution	Span calibration solution
Fluoride F^-	1.9 mg/L (0.1m mol/L)	19 mg/L (1m mol/L)
Potassium K^+	3.9 mg/L (0.1m mol/L)	39 mg/L (1m mol/L)
Ammonia NH_3	1.8 mg/L (0.1m mol/L) + Sodium hydride*	18 mg/L (1m mol/L) + Sodium hydride*

* To prepare a standard solution for the ammonia ion sensor, place sodium hydride (0.4 g per 100 ml) into a standard solution for ammonium chloride directly before calibrating the ion sensor. A standard solution with sodium hydride changes with time because all of the ammonia content exists as ammonia gas. It is important to use the solution immediately.

5.7.2 Zero calibration

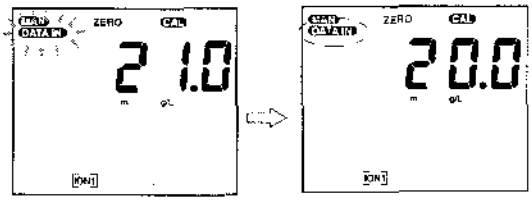


- Wash the sensor with distilled water a few times. Then pour a zero calibration solution into the calibration beaker and immerse the sensor (the B side) into the solution.
- Select the measurement mode for each measurement item (ION1, 2, and 3) and press the **CAL** key twice.
  When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.



Manual Zero Calibration mode

- Use the **UP/DOWN** (▲ ▼) keys to set the value for the zero calibration standard solution.
- Press the **ENT** key.
  The manual zero calibration is starts.



End of calibration

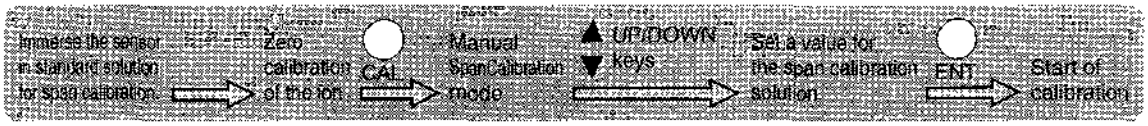
The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

Important

For standard ion sensors, the common zero calibration solution is used. It is necessary to calibrate each of these ion sensors in the solution by performing the above procedure.
For optional sensors, it is necessary to calibrate each of these ion sensors in the zero calibration solution for each ion sensor.

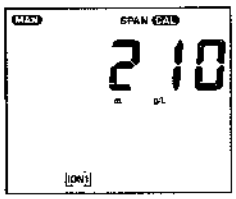
5.7.3 Span calibration



1. Pour the span calibration solution into the calibration beaker. Then wash the sensor with distilled water a few times and immerse the sensor (the B side) into the solution.

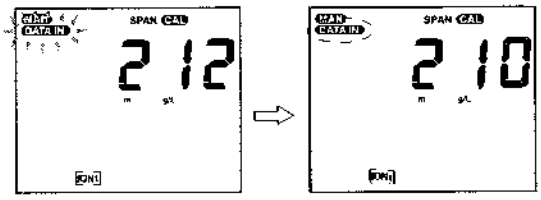
2. After the zero calibration of the ion sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

3. Use the **UP/DOWN** (▲ ▼) keys to set a value for the span calibration solution according to the ion type.



4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Important

- For standard ion sensors, the common span calibration solution is used. It is necessary to calibrate each of these ion sensors in the solution by performing the above procedure.
- For optional sensors, it is necessary to calibrate each of these ion sensors in the span calibration solution for each ion sensor.

Note

- When the SET and CAL keys are pressed during the relevant manual Ion calibration mode, the calibration data for the relevant ion sensor can be deleted.

6. Using the various functions

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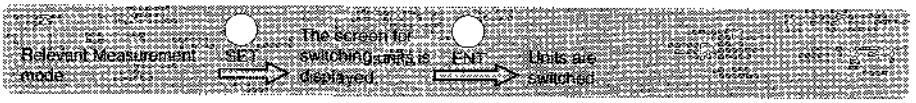
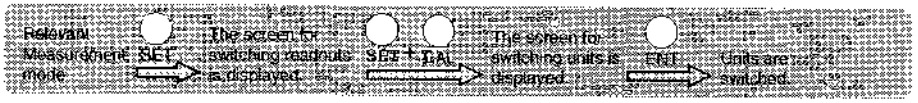
6.2 Switching measurement units 64

In the case of pH, ION, TURB, DO 64



In the case of COND and DEP 65



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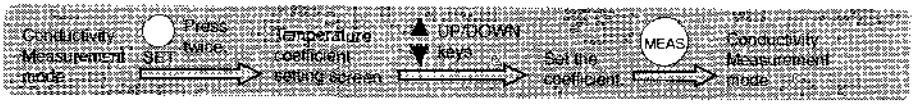
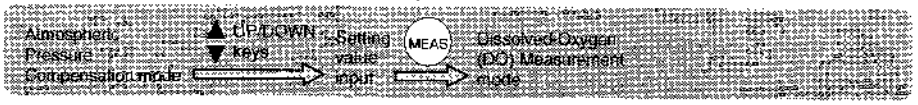


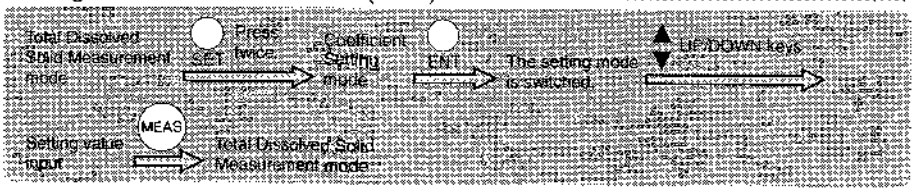
6.4 Dissolved-Oxygen (DO) environmental influence compensation 68

6.4.1 Salinity compensation 68

6.4.2 Atmospheric pressure compensation 69



6.5 Setting a total dissolved solid (TDS) coefficient 70



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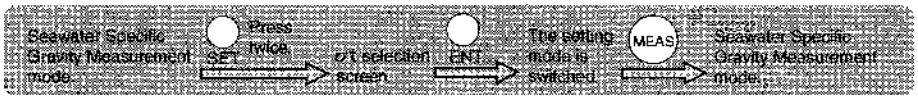
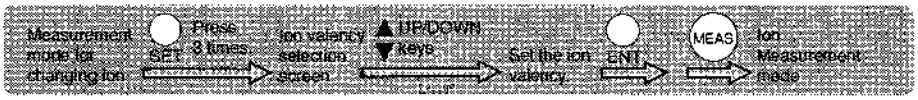
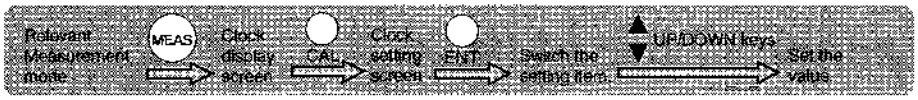


6.7 Changing the ion valency setting (U-23 model) 72



6.8 Setting the clock 73



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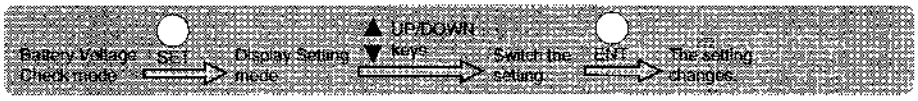

6.10.4 Remaining memory 78

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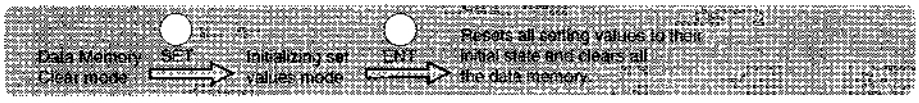
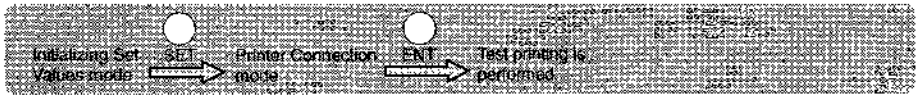
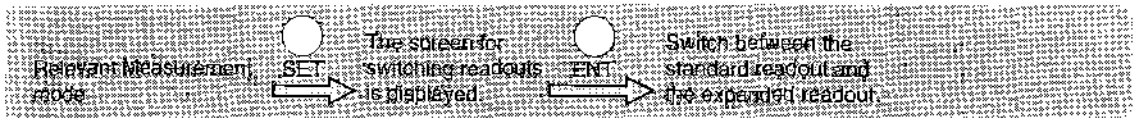


6.10.7 Printer connection, test print 82



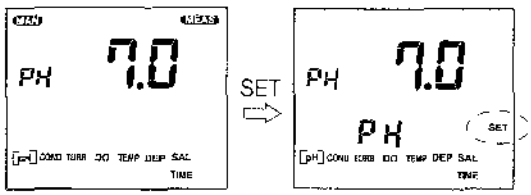
6.1 Switching to Expanded readout (High-accuracy display)

With the exception of oxidation-reduction potential (ORP), it is possible to switch between the Standard readout and the Expanded readout for the measurement value.



1. Press the **SET** key in the relevant Measurement mode.

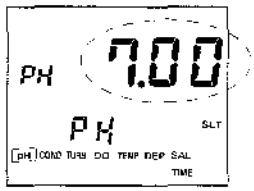
The screen for switching readouts is displayed.



Standard readout

2. Press the **ENT** key.

The screen can be switched between the standard readout and the expanded readout (High-accuracy display).



Expanded readout (High-accuracy display)

Note

Switch readouts for each measurement items.

Use the manual 2-point calibration (zero and span) when high accuracy is required for expanded readout (High-accuracy display).

The expanded readout mode is automatically activated when the manual 2-point calibration mode is chosen.

3. Press the **MEAS** key to return to the Measurement mode.

6.2 Switching measurement units

It is possible to switch between measurement units.

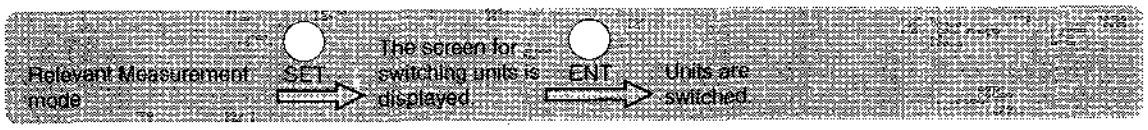
The units which can be switched are as follows:

pH ..... pH or mV
Conductivity (COND) ..... S/m or mS/cm
Water depth (DEP) ..... m or ft
Ion concentration (ION) ..... g/L or mol/L
Turbidity (TURB) ..... NTU or mg/L
Dissolved Oxygen (DO) ..... mg/L or % (Oxygen saturation ratio)

Note

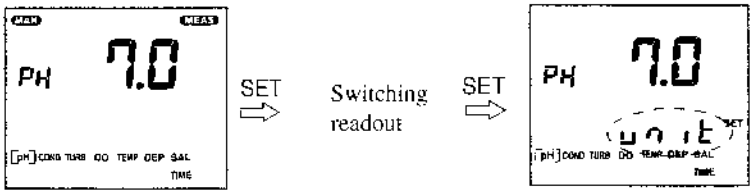
- When the measurement unit for ion is switched, the calibration value returns to the initial value.

In the case of pH, ION, TURB, DO



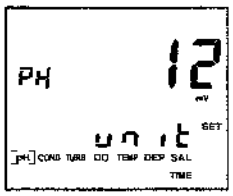
1. Press the **SET** key twice in the relevant Measurement mode.

Confirm that unit is displayed on the screen for switching units.



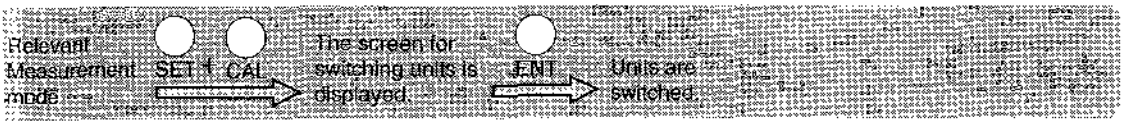
2. Press the **ENT** key.

Units are switched.



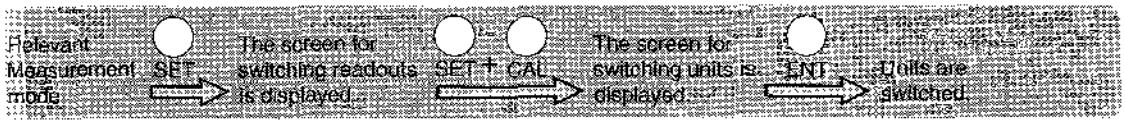
3. Press the **MEAS** key to return to the Measurement mode.

ı the case of COND



- Press the **SET** key and **CAL** key simultaneously in the Relevant Masrement mode.
  Confirm that unit is displayed on the screen for switching units.
- Press the **ENT** key.
  Units are switched.
- Press the **MEAS** key to return to the Measurement mode.

ı the case of DEP



- Press the **SET** key in the Relevant Measurement mode.
  The screen for switching readout is displayed.
- The **SET** key and **CAL** key press simultaneously.
  Confirm that unit is displayed on the screen for switching units.
- Press the **ENT** key.
  Units are switched.
- Press the **MEAS** key to return to the Measurement mode.

Display and units

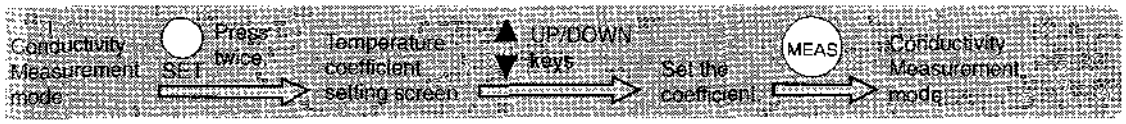
Measurement item		Readout range		Measurement units
		Expanded	Standard	
pH		0.00 to 14.00	0.0 to 14.0	pH
		–	-1999 to 1999	mV in pH measurement
Conductivity (COND)	Range 1	0.90 to 9.99	0.9 to 9.9	S/m
		9.0 to 99.9	9 to 99	mS/cm
	Range 2	0.090 to 0.999	0.09 to 0.99	S/m
		0.90 to 9.99	0.9 to 9.9	mS/cm
	Range 3	0.0 to 99.9	0 to 99	mS/m
		0.000 to 0.999	0.00 to 0.99	mS/cm
Turbidity (TURB) *1		0.0 to 800.0	0 to 800	NTU (nephelometric turbidity units) or mg/L
Dissolved-oxygen (DO)		0.00 to 19.99	0.0 to 19.9	mg/L
		0.0 to 199.9	0 to 199	%
Temperature (TEMP)		-5.00 to 60.00	-5.0 to 60.0	C
Water depth (DEP)		0.0 to 100.0	0 to 100	m
		0.0 to 330.0	0 to 330	ft
Salinity (SAL)		0.00 to 4.00	0.0 to 4.0	‰
Total dissolved solids	Range 1	5.5 to 65.0	5 to 65	g/L
(TDS) *2	Range 2	0.55 to 6.50	0.5 to 6.5	g/L
	Range 3	0.000 to 0.650	0.00 to 0.65	g/L
Seawater specific gravity (s_t)		0.0 to 50.0	0 to 50	–
Oxygen-reduction potential (ORP)		–	-1999 to 1999	mV
Ions 1, 2, and 3	Range 1	0.100 to 0.999	0.10 to 0.99	g/L, mg/L, μ g/L
		0.100 to 0.999	0.10 to 0.99	mol/L, mmol/L, μ mol/L
	Range 2	1.00 to 9.99	1.0 to 9.9	g/L, mg/L, μ g/L
		1.00 to 9.99	1.0 to 9.9	mol/L, mmol/L, μ mol/L
	Range 3	10.0 to 99.9	10 to 99	g/L, mg/L, μ g/L
		10.0 to 99.9	10 to 99	mol/L, mmol/L, μ mol/L

*1: Depending on the concentration range, the minimum turbidity is displayed as follows:
0 to 100 NTU ..... 1 NTU for standard readout, 0.1 NTU for expanded readout.
100 to 800 NTU ... 10 NTU for standard readout, 1 NTU for expanded readout.

*2: The TDS range depends on the TDS factor settings.

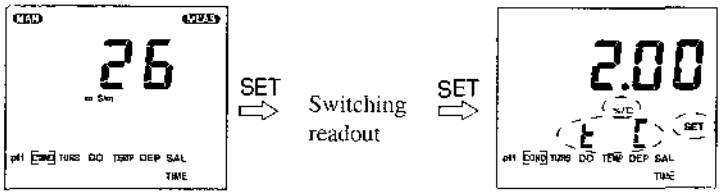
5.3 Temperature conversion for conductivity (COND)

Sample conductivity (COND) varies with temperature, and this instrument uses a temperature conversion coefficient to automatically standardize the conductivity (COND) to the value at 25°C. The initial setting value is 2%/°C, which is the generally used value.



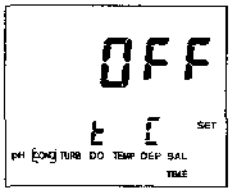
- Press the **SET** key twice in the Conductivity (COND) Measurement mode.

  The screen for setting temperature coefficients is displayed.



- Use the **UP/DOWN** (▲ ▼) keys to set the coefficient.

  The setting range is 0.00 to 3.00%/°C.



  With the ENT key, the temperature conversion is switched between ON and OFF.

- Press the **MEAS** key.

  The instrument returns to the Conductivity (COND) Measurement mode.

Note

For temperature coefficients, refer to *Reference data*, page 98 to 99.

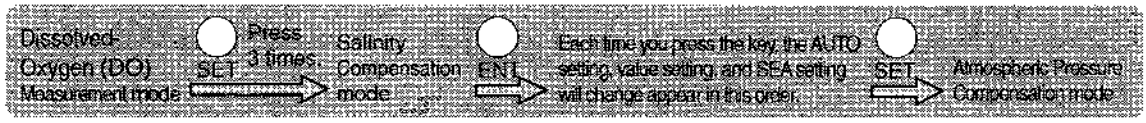
6.4 Dissolved-Oxygen (DO) environmental influence compensation

6.4.1 Salinity compensation

The indicated dissolved oxygen (DO) value can go over the actual value if salinity compensation isn't added because of the increase in salinity in the sample. To obtain a correct measured value for dissolved oxygen (DO) in the sample containing salinity, therefore, salinity compensation is needed. The following modes are available for calculation of salinity compensation.

AUTO..... Salinity compensation is performed automatically with salinity converted from a measured value for conductivity.

SEA ..... Compensation value appropriate for normal seawater is used.



1. Press the **SET** key 3 times in the Dissolved-Oxygen (DO) Measurement mode.

The salinity compensation mode currently set is displayed.

Important

- If you do not change the salinity compensation mode currently set, press the MEAS key to return to the Dissolved-Oxygen (DO) Measurement mode or press the SET key to select the Pressure Compensation mode.

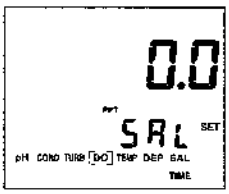
2. Press the **ENT** key.

The following screens are displayed in sequence each time the ENT key is pressed: AUTO setting, value setting, SEA setting and AUTO setting.

3. From the screen on which the value is displayed, use the **UP/DOWN** (▲ ▼) keys to enter the setting value if the salinity is known.

For AUTO and SEA setting, this step need not be performed.

The setting range is 0.0 to 40.0 PPT (parts per thousand).

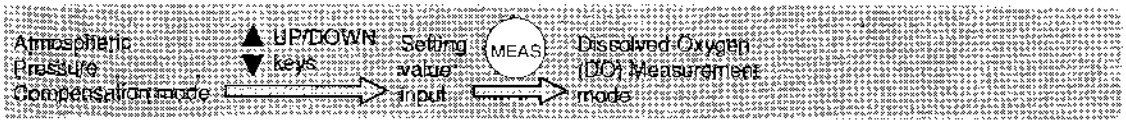


4. When the **SET** key is pressed, setting will be completed and the instrument will enter the Pressure Compensation mode.

5. Press the **MEAS** key to return to the Dissolved-Oxygen (DO) Measurement mode.

5.4.2 Atmospheric pressure compensation

Differences in the atmospheric pressure of the measurement location influence the Dissolved-Oxygen (DO) measurement. By setting (input) the actual atmospheric pressure of the measurement location into the instrument, it is possible to standardize the measured Dissolved-Oxygen (DO) value to a value at the standard atmospheric pressure (1013 hPa).



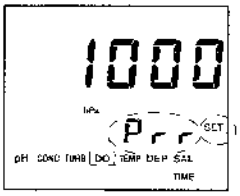
- When the **SET** key is pressed on the salinity compensation screen, setting will be completed and the instrument will enter the Pressure Compensation mode.

Important

If you do not change the Pressure Compensation mode currently set, press the MEAS key to enter the Dissolved-Oxygen (DO) Measurement mode.

- Use the **UP/DOWN** (▲ ▼) keys to input a setting value.

  The setting range is 100 to 1999 hPa.



- When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Dissolved-Oxygen (DO) Measurement mode.

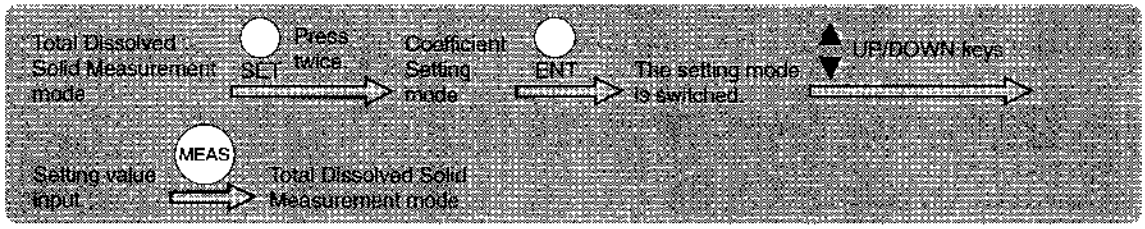
6.5 Setting a total dissolved solid (TDS) coefficient

The total dissolved solid amount (TDS) is a converted value obtained by multiplying the conductivity (COND) value by a known coefficient. Based on a conversion for KCl and $CaCO_3$ solutions, the coefficient initially set for the instrument depends on the conductivity (COND) value as shown below.

Conductivity (COND) (S/m)	Conversion coefficient
< 0.05	0.65
0.05 to 0.5	0.64
1.5 to 1	0.63
1 to 3	0.62
3 to 5	0.61
> 5	0.60

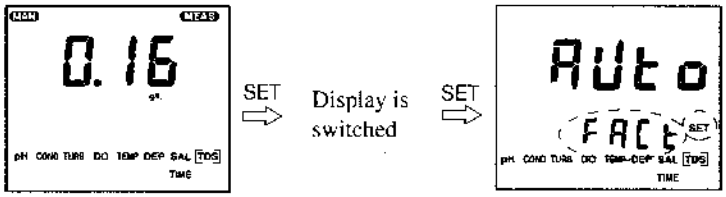
AUTO ..... Used to automatically calculate the total dissolved solid (TDS) amount with an initially set coefficient.

Setting value input ..... Used to determine the total dissolved solid (TDS) amount by setting any conversion coefficient irrespective of the conductivity (COND) value.



1. Press the **SET** key twice in the Total Dissolved Solid (TDS) Measurement mode.

The Coefficient Setting mode currently set is displayed.

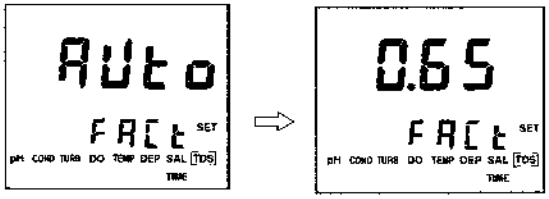


Important

- If you do not change the coefficient currently set, press the MEAS key to enter the Total Dissolved Solid (TDS) Measurement mode.

2. Press the **ENT** key.

The setting mode changes (AUTO/setting value input).



3. Use the **UP/DOWN** (▲ ▼) keys to input a setting value if required.

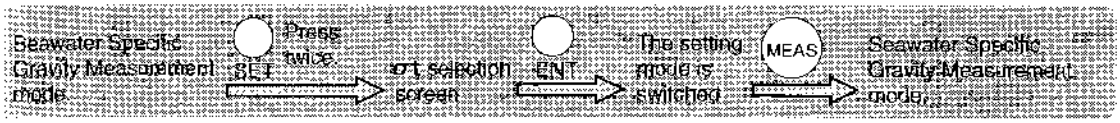
The setting range is 0.50 to 1.00.

4. When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Total Dissolved Solid (TDS) Measurement mode.

.6 Displaying seawater specific gravity (σ_0, σ_{15})

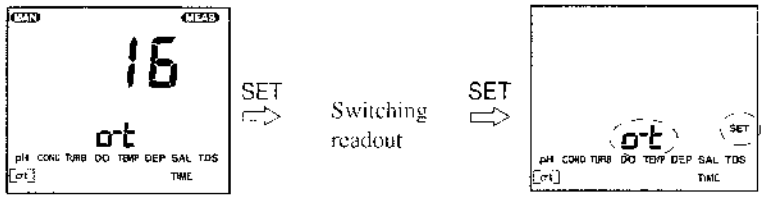
ic specific gravity of seawater varies with temperature. By converting the measured value based on the value for a ference temperature, it is possible to compare sample measurement values at different temperatures.

σ_t ..... Specific gravity of seawater at the measurement temperature.
σ_0 ..... Seawater specific gravity at 0°C.
σ_{15} ..... Seawater specific gravity at 15°C.



- Press the **SET** key twice in the Seawater Specific Gravity (σ_t) Measurement mode.

  Seawater specific gravity (σ_t) selection screen is displayed.



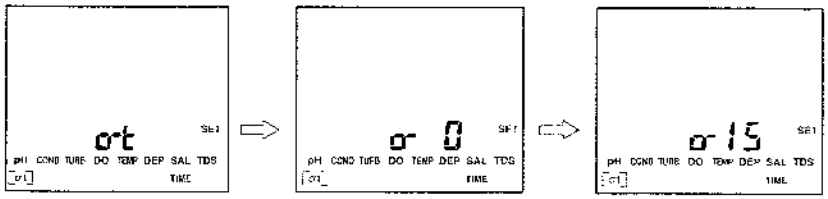
Important

If you do not change the specific gravity currently set, press the MEAS key to enter the Seawater Specific gravity (σ_t) Measurement mode.

- Press the **ENT** key.

  The setting mode is switched.

  (σ_0 → σ_{15} → σ_t → σ_0 ...)



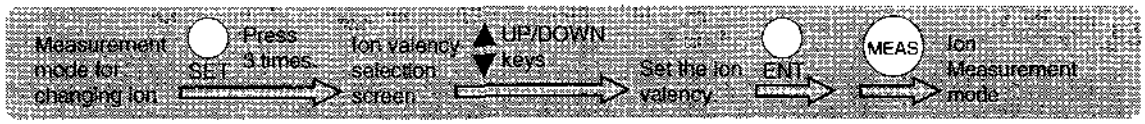
- When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Seawater Specific Gravity (σ_t) Measurement mode.

Note

See page 100 for more about seawater specific gravity.

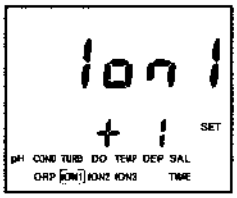
6.7 Changing the ion valency setting (U-23 model)

When you use the standard sensors (Cl^-, NO_3^- and Ca^{2+}), the ion valency has been already set in the initial settings, and no changes are required.
When other sensors than the standard ones are used, the ion valency must be set.



1. Press the **SET** key 3 times in the Ion Measurement mode for the ion to be changed.

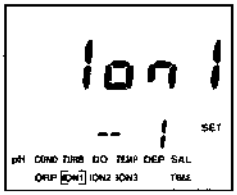
Ion valency selection screen is displayed.



Important

- If you do not change the ion valency currently set, press the MEAS key to enter the Ion Measurement mode.

2. Use the **UP/DOWN** (▲ ▼) keys to set the ion valency and press the **ENT** key.



UP (▲) : -2 → -1 → +1 → +2
DOWN (▼) : -2 ← -1 ← +1 ← +2

Important

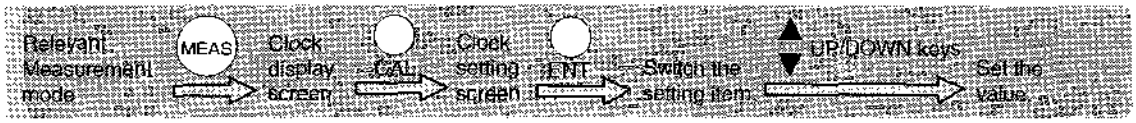
- The calibration data is cleared if the ion valency value is changed.

3. When the **MEAS** key is pressed, the setting will be completed and the instrument will enter the Ion Measurement mode.

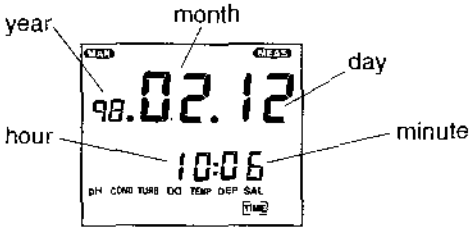
Note

- For the ion valency, refer to page 106.

5.8 Setting the clock



1. Use the **MEAS** key in the measurement mode to switch to the clock display screen.



2. Press the **CAL** key.

CAL light up and clock setting screen is displayed.

3. Press the **ENT** key to switch the measuring item.

(year → month → day → hour → minute → year ...). The setting item will blink.

- Use the **UP/DOWN** (▲ ▼) keys to set the value.
- Press **SET** key to confirm the setting.

Note

When the MEAS key is pressed, the instrument will return to the clock display.

Important

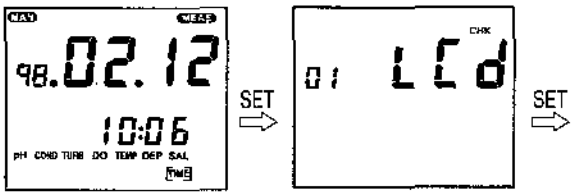
When the MEAS key is pressed without pressing the SET key and the clock display is displayed again, settings are not changed.

6.9 Key lock setting

If you press the POWER key while pressing the UP (▲) key when the power is off, the instrument is then turned ON with the key locked and the key lock function works.
With the key locked, only the POWER and MEAS keys can be used and [LOCK] is displayed on the screen. To release this function, turn the instrument OFF first and then ON again.

6.10 Check mode

When the SET key is pressed in the measurement mode from the screen where "year, month, day and time" are displayed, the instrument performs self-diagnosis check.



Each time the SET key is pressed, the check mode item is switched sequentially.

Check mode items

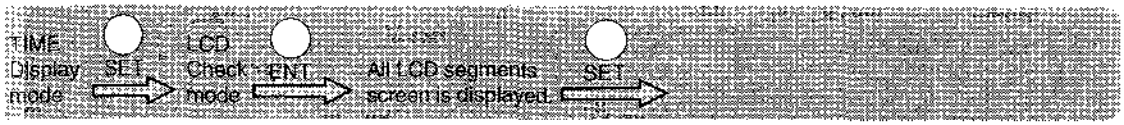
Item	Designation		Page
1	LCD check	Checks if all LCD segments will be displayed.	75
2	Battery voltage check	Performs a simple battery voltage check for the instrument and sensors.	76
3	Measurement item setting	Can set the measurement item to be stored.	77
4	Remaining memory	Displays the number of data that can be stored now.	78
5	Data memory clear	Clears the data memory.	79
6	Initializing set values	Initializes all memory settings.	80
7	Printer test	Performs a test print.	82

Note

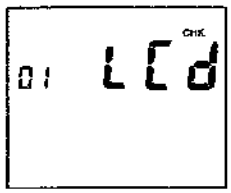
- In the check mode, it is possible to return to the Measurement mode by pressing the MEAS key.

.10.1 LCD check

ll LCD segments are displayed.

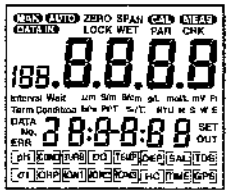


1. Press the **SET** key in the Clock Display mode.
   LCD check mode screen is displayed.

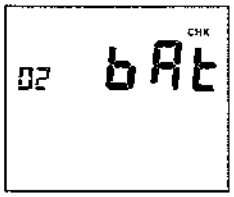


2. Press the **ENT** key.

3. Check to see if all LCD segments are displayed.



4. When the **SET** key is pressed, the instrument goes to the battery voltage check.

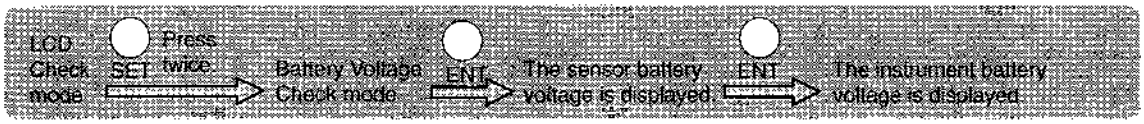


Note

When the MEAS key is pressed, the instrument returns to the Clock Display mode.

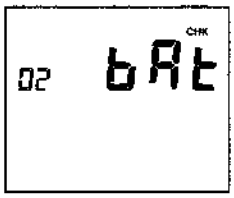
6.10.2 Battery voltage check

The battery voltage in use is displayed.



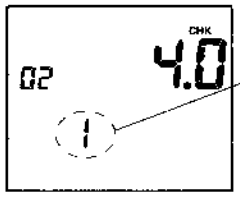
1. Press the **SET** key twice in the LCD Check mode.

Battery Voltage Check mode screen is displayed.



2. Press the **ENT** key.

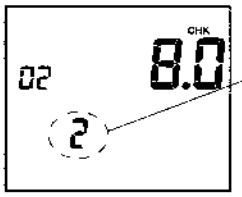
The sensor battery voltage is displayed.



1: Sensor battery voltage
Criteria
3 to 5.0 V : Normal
Less than 3 V: Replace the battery.
(Error No. 2 is ON.)

3. Press the **ENT** key.

The instrument battery voltage is displayed.



2: Instrument battery voltage
Criteria
5.5 to 11.0 V : Normal
Less than 5.5V: Replace the battery.
(Error No.3 is ON.)

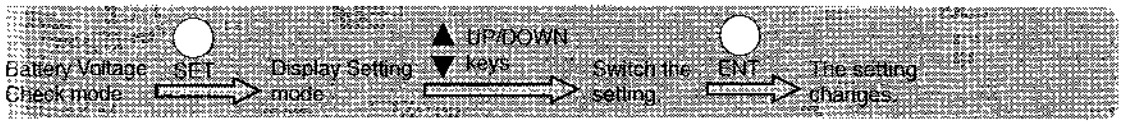
4. When the **SET** key is pressed, the instrument goes to the battery voltage check.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

.10.3 Measurement item setting

easuring items can be set.

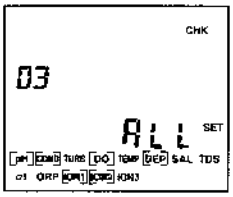


- Press the **SET** key in the Battery Voltage Check mode.

  Display setting mode screen is displayed.

- Use the **UP/DOWN** (▲ ▼) keys to switch the measurement item.

  The selected item blinks.



- Press the **ENT** key to switch between [set/ not set] for the blinking item.

  An item for which "set" is selected is indicated with [ ].

Note

If the temperature is "not set" data for each component is not temperature-compensated and is displayed as data at 25°C.

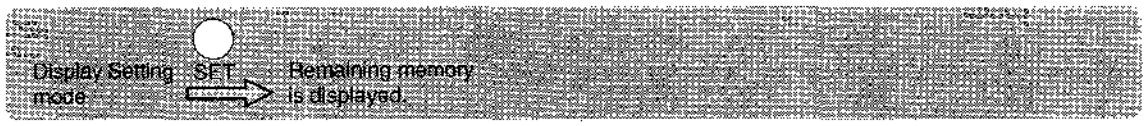
- When the **SET** key is pressed, the instrument goes to the remaining memory display.

Note

When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.10.4 Remaining memory

The number of date that can be stored can be displayed.



1. Press the **SET** key four times in the Display Setting mode.

Remaining memory is displayed.

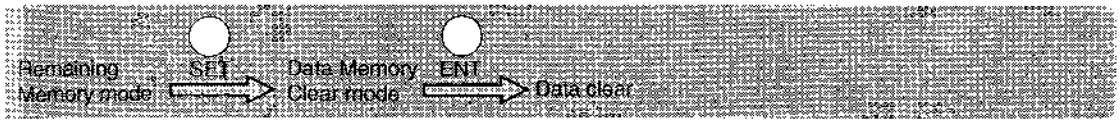
2. When the **SET** key is pressed, the instrument goes to the Data Memory Clear mode.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

.10.5 Data memory clear

I the data memory is cleared.

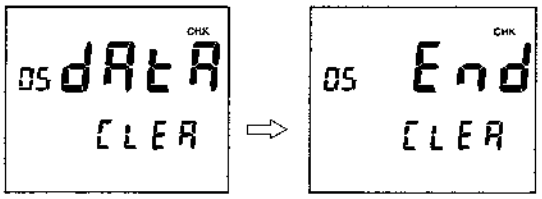


- Press the **SET** key in the Remaining Memory mode.

  Data memory clear mode screen is displayed.

- Press the **ENT** key.

  The data is cleared.



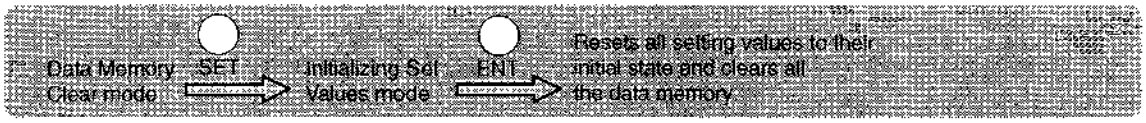
- When the **SET** key is pressed, the instrument goes to the Memory Initialization mode.

Note

When the MEAS key is pressed, the instrument returns to the Clock Display mode.

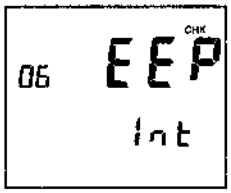
6.10.6 Initializing set values

All setting values are reset to their initial state.



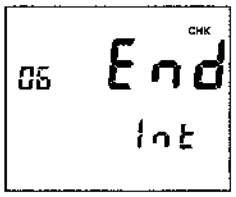
1. Press the **SET** key in the Data Memory Clear mode.

Initializing Set Values mode screen is displayed.



2. Press the **ENT** key.

All setting values are reset to their initial state.



3. When the **SET** key is pressed, the instrument goes to the Printer Connection mode.

Note

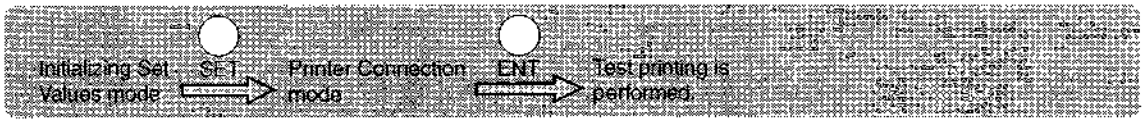
- When the MEAS key is pressed, the instrument return to the Clock Display mode.
- Data stored in the memory remains.

nitial setting

Item	Description	Initial value
Common	Display setting	Standard
	Data storage	Manual
pH	Unit	pH
COND	Unit	S/m
	Temperature coefficient	2.0 %/°C
DO	Salinity setting	AUTO
	Atmospheric pressure setting	1013 hPa
	Unit	mg/L
TURB	Unit	NTU
DEP	Unit	m
TDS	Coefficient	AUTO
σ_t	Unit	σ_t
ION	Unit	g/L
ION1	Ion type	-1
ION2	Ion type	-1
ION3	Ion type	+2

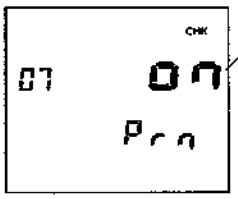
6.10.7 Printer connection, test print

This mode only operates when the function expansion unit is connected. A test print is performed if a printer is connected.



1. Press the **SET** key in the Initializing Set Values mode.

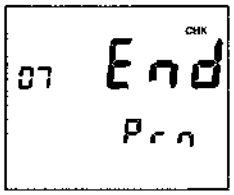
Printer Connection mode screen is displayed.



Displayed as OFF when not connected.

2. Press the **ENT** key to start printing.

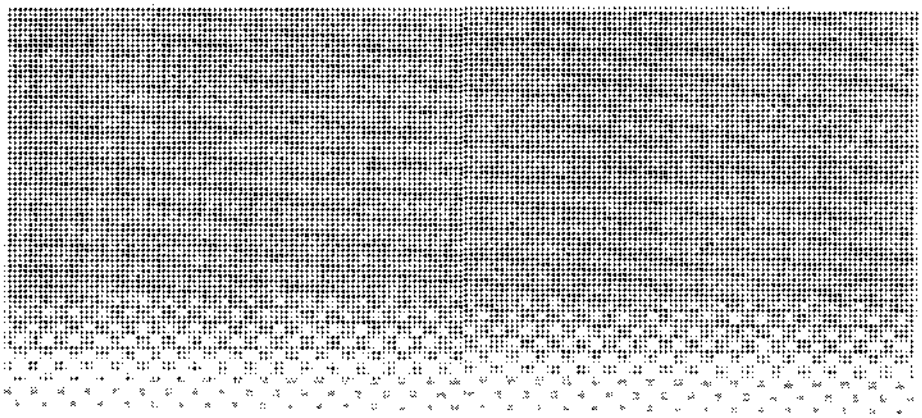
Normally, "End" is displayed. If an error has occurred, "Err" is displayed.



3. When the **SET** key is pressed, the instrument will return to the first LCD check mode.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.



7 ■ Instrument specifications

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7.4 Spare parts 92
7.5 Option 93

7.1 Daily maintenance

Sensor probe

● Storage

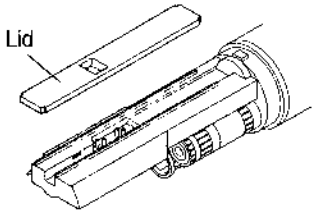
After use, clean the sensor probe in tap water and wipe off contamination. Next put distilled water into the calibration beaker to the marked line. Then attach the calibration beaker to the sensor probe and store the probe assembly in the carrying case.
Do not put water in the calibration beaker before attaching it to the ion sensor end (B side) of U-23.

For a long use
Wipe off contamination from the cable, sensor probe, and sensor before storage.

TEMP/COND/TURB units

● To remove contamination

1. Remove the lid from the cell.
2. Clean the unit in tap water. If the unit is severely contaminated, use an absorbent cotton to remove contamination.
3. Attach the lid to the cell block before storage.



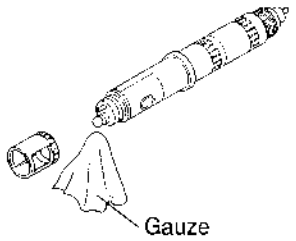
Important

- The cell has a window for turbidity measurement. Be careful to avoid damage to the window. In case of measurements, attach the lid to the cell in the correct direction.

pH/ORP sensors

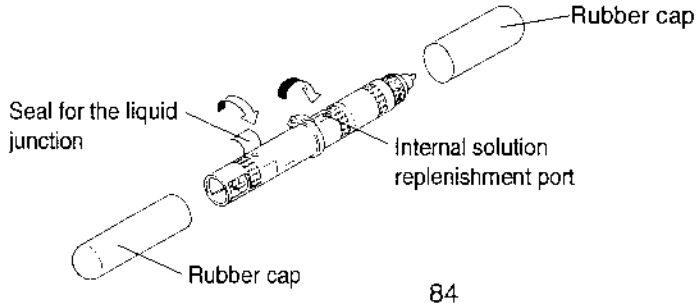
● To remove contamination

Use a piece of gauze dampened with detergent and wipe off contamination.



● Long-term storage

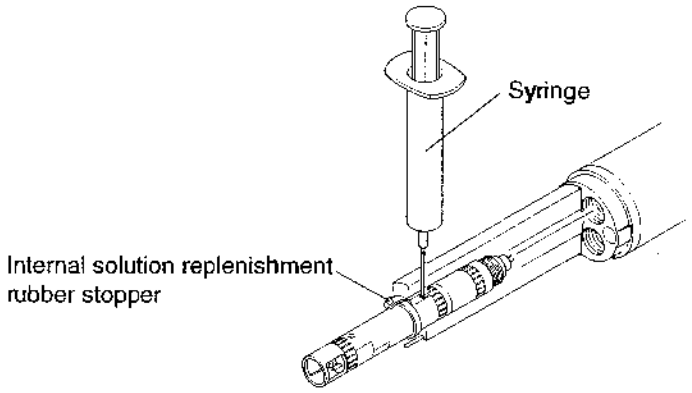
Remove the sensor from the sensor probe and close the internal solution replenishment port. Then attach a seal to the liquid junction and attach the rubber caps before storage.



Monthly maintenance

Replace the internal solution as described below:

1. Remove the sensor from the sensor probe using a sensor spanner.
2. Open the internal solution replenishment rubber stopper and remove the internal solution with a syringe.
3. Pour new internal solution (#330) to the level near rubber stopper. Be careful to avoid air bubbles from coming in the solition.
4. Attach the sensor to the sensor probe.



O sensor

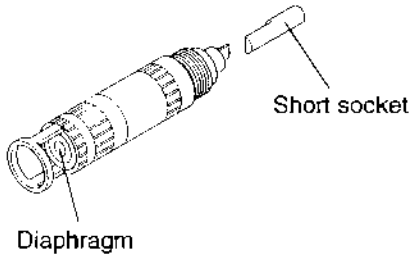
To remove contamination

Wipe off contamination with gauze to avoid damage to the diaphragm.

Long-term storage

Remove the sensor from the sensor probe using a sensor spanner. Set the supplied short socket and store the sensor in a cool, dark place.

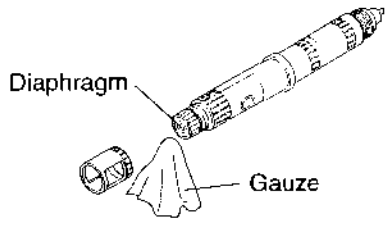
If the sensor probe is stored without a short socket, stable indications may not be obtained.



ION sensors (for the U-23 model only)

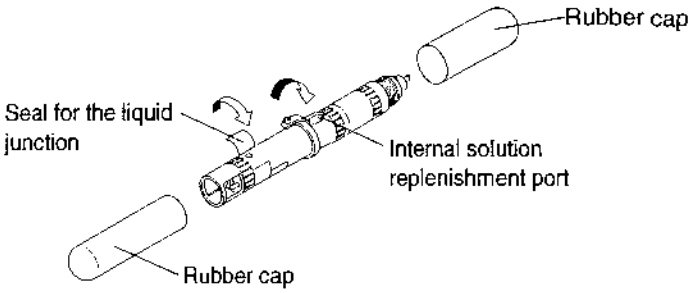
● To remove contamination

Use a piece of gauze dampened with water to wipe off contamination, being careful not to scratch it.



● Long-term storage

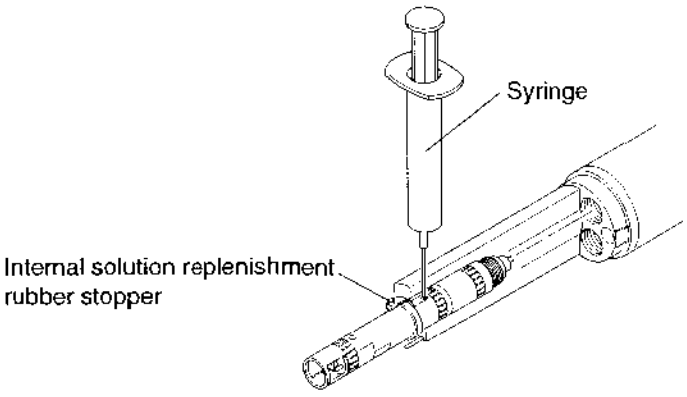
Use a sensor spanner to remove the sensor from the sensor probe and close the internal solution replenishment port. Attach a seal for the liquid junction and attach the rubber caps to the sensor before storage.



● Maintenance at intervals of one week to one month

Replace the internal solution as described below:

1. Remove the sensor from the sensor probe using a sensor spanner.
2. Open the internal solution replenishment rubber stoper and remove the internal solution with a syringe.
3. Pour a new internal solution described in the sensor manual to the level near the rubber stopper. Be careful to avoid air bubbles from coming in the solution.
4. Attach the sensor to the sensor probe.



7.2 Troubleshooting

The instrument has a simple error message that informs users of operational errors and failure. Err No. is displayed at the bottom of the screen.

Error message list

Err No.	Designation	Err No.	Designation
1	Sensor memory failure	6	Span calibration error
2	Sensor battery voltage drop	7	Calibration stability error
3	Instrument battery voltage drop	8	Printer error
4	Communications error	9	DATA IN error
5	Zero calibration error		

Error and remedy

Important

For err Nos. 5 to 7, the calibration err display disappears when a proper calibration is performed after the following action, or when the instrument is turned on again. For the other err Nos., the err display disappears after any of the following actions is taken.

Err NO.	Problem	Cause	Remedy
1	No data can be read from or written into the sensor probe memory.	Internal IC failure	Call your nearest store for sensor probe repair.
2	Sensor probe battery voltage drop	① Battery voltage drop ② Improper installation of the battery	① Replace the sensor probe battery. ② Set the batteries (LR03) in the correct direction.
3	Instrument battery voltage drop	① Battery voltage drop ② Improper installation of the battery	① Replace the instrument battery. ② Set the battery (6LR61) in the correct direction.
4	No communications possible between the instrument and the sensor probe	① Improper connection of the connector to the instrument ② Cable disconnection	① Connect the connector to the instrument properly and turn on the instrument again. ② Call your nearest store for cable repair.
5	No zero calibration possible	pH • Contamination on the pH glass membrane • Change in concentration of the internal solution for the reference electrode • Cracks in the pH glass electrode COND • The standard solution is contaminated. • The sensor is dirty. • The COND sensor is broken.	pH • Clean the pH glass membrane. • Replace the internal solution for the reference electrode. • Replace the sensor. COND • Change the standard solution. • Clean the sensor. • Contact your nearest store.

Err NO.	Problem	Cause	Remedy
5	Zero calibration not possible	TURB	TURB
		• Air bubbles in the cell	• Swing the sensor probe while drawing a large arc.
		• Cell contamination	• Clean the cell.
		DO	DO
		• Damage to the diaphragm of the DO sensor	• Check the sensor and replace it if damaged.
		ION (For the U-23 model only)	ION
		• Contamination on the ion sensor	• Clean the ion sensor.
		• Improper internal solution for the comparison electrode	• Fill the reference electrode with proper internal solution.
		• End of the service life of the ion cartridge	• Replace the ion cartridge.
		DEP (For the U-22, U-23 models)	DEP
		• Contamination on the DEP sensor	• Clean the DEP sensor.
		• Damage to the DEP sensor	• Contact your nearest store.
6	Span calibration not possible	pH	pH
		• Contamination on the pH glass membrane	• Clean the pH glass membrane.
		• Change in concentration of the internal solution for the reference electrode	• Replace the internal solution for the reference electrode.
		• Cracks in the pH glass electrode	• Replace the sensor.
		COND	COND
		• The standard solution isn't correct.	• Calibrate with correct standard solution.
		• The COND sensor is broken.	• Contact your nearest store.
		TURB	TURB
		• Air bubbles in the cell	• Swing the sensor probe while drawing a large arc.
		• Cell contamination	• Clean the cell.
		DO	DO
		• Damage to DO sensor diaphragm	• Check the DO sensor and replace it if damaged.
		ION (For the U-23 model only)	ION
		• Contamination on the ion sensor	• Clean the ion sensor.
		• Improper internal solution for the reference electrode	• Fill the comparison electrode with proper internal solution.
		• End of the service life of the ION cartridge	• Replace the ion cartridge.
		DEP (For the U-22, U-23 models)	
		• Contamination on the DEP sensor	• Clean the DEP sensor.
		• Damage to the DEP sensor	• Contact your nearest store.
		TEMP	
		• Damage to the TEMP sensor	• Contact your nearest store.
7	The calibration value does not become stable within approximately three minutes.	① Sensor contamination	① Clean each sensor.
		② Dry sensor surface	② Pour the standard solution into the calibration beaker. Calibrate the sensor again 1 to 2 hours later.
		③ Severe temperature change	③ Calibrate the sensor in a place at a stable temperature or in a thermostatic oven.

Err NO.	Problem	Cause	Remedy
8	Printer unit failure		Turn OFF the instrument and use the remedy described below. Then turn ON the printer again.
		① Paper has jammed in the printer	① Remove the jammed sheet of paper
		② Improper printer unit connection	② Check to see if the printer is properl connected to the instrument.
		③ Printer failure	③ Replace the printer.
			* Contact your nearest store if the instrument does not recover after replacement of the printer.
9	Data cannot be stored because the memory is full.	No free space in the memory	Delete the data stored in the memory. (☞ Page 79)

Other troubles

emedies for various trouble with no Err No. displayed are described below.

Problem	Cause	Remedy
o data display with the ower on	• No batteries	• Set new batteries.
	• Improper position of the positive and negative poles	• Set the batteries properly while paying attention to the positive and negative poles.
	• Battery voltage drop	• Replace the batteries with new ones.
	• Improper instrument battery contact	• Use radio pliers to narrow the positive terminal of the battery snap.
o setting change possible	• Automatic data storage is under way	• Press the CAL key to stop the automatic data storage.
o key operation possible	• The key lock function is working	• Turn OFF the instrument. Then turn ON the instrument again. (☞ Page 36)
	• Failure to calibrate the sensor or wrong calibration.	• Calibrate the sensor properly.
linking measured value	• Improper measurement sample	• Use a sample that is in the measurement range.
	• Sensor contamination	• Clean each sensor.
	• Poor calibration is possible. (The standard solution is contaminated.)	• Carry out correct calibration.
:YPE Err o Err is displayed and the eration cannot be performed.	• Improper connection of the cable connector to the instrument	• Connect the connector to the instrument properly and turn on the instrument again.
	• Cable disconnection	• Contact your nearest store.
	• Instrument inside failure	• Contact your nearest store.

7.3 Specifications of U-20 series models

NOTE ○: Applicable
—: Unapplicable

		U-21	U-22	U-23
Instrument	Water-proof construction	IP67	IP67	IP67
	Mass	Approximately 475g (including the grip holder)	Approximately 475g (including the grip holder)	Approximately 475g (including the grip holder)
Sensor	Use in 2-inch well	○	○	-
	Measurement temperature	0 to 55°C	0 to 55°C	0 to 55°C
	Storage temperature	-5 to 60°C	-5 to 60°C	-5 to 60°C
	Measurement depth	to 100m	to 100m	to 100m (except ion)
	Maximum sensor outside diameter	46mm	46mm	95mm
	Sensor length	380mm	380mm	430mm
	Continuous use available	30 days	30 days	30 days (except ion)
	Automatic data gathering at set time	○	○	○
	Mass (Cable 2m)	Approximately 1kg	-	-
	(Cable10m)	Approximately 1.5kg	Approximately 1.5kg	Approximately 1.8kg
pH	Measuring principle	Glass electrode method	Glass electrode method	Glass electrode method
● Two-point calibration	Range	pH0 to 14	pH0 to 14	pH0 to 14
● Automatic temperature compensation	Resolution	0.01pH	0.01pH	0.01pH
	Repeatability	±0.05pH	±0.05pH	±0.05pH
	Accuracy	±0.1pH	±0.1pH	±0.1pH
Dissolved-Oxygen	Measuring principle	Diaphragm galvanic battery method	Diaphragm galvanic battery method	Diaphragm galvanic battery method
● Salinity conversion (0 to 40ppt/Auto)	Range	0 to 19.99mg/L	0 to 19.99mg/L	0 to 19.99mg/L
	Resolution	0.01mg/L	0.01mg/L	0.01mg/L
● Automatic temperature compensation	Repeatability	±0.1mg/L	±0.1mg/L	±0.1mg/L
	Accuracy	±0.2mg/L	±0.2mg/L	±0.2mg/L
Conductivity	Measuring principle	4 AC electrode method	4 AC electrode method	4 AC electrode method
● Auto range	Range	0 to 9.99S/m	0 to 9.99S/m	0 to 9.99S/m
● Automatic temperature conversion (25°C)	Resolution	0.1%F.S	0.1%F.S	0.1%F.S
	Repeatability	±1%	±1%	±1%
	Accuracy	±3%	±3%	±3%
Salinity	Measuring principle	Conductivity conversion	Conductivity conversion	Conductivity conversion
	Range	0 to 4%	0 to 4%	0 to 4%
	Resolution	0.01%	0.01%	0.01%
	Repeatability	±0.1%	±0.1%	±0.1%
	Accuracy	±0.3%	±0.3%	±0.3%
Total Dissolved Solid (TDS)	Measuring principle	Conductivity conversion	Conductivity conversion	Conductivity conversion
	Range	0 to 100 g/L	0 to 100 g/L	0 to 100 g/L
● Conversion factor setting	Resolution	0.1%F.S	0.1%F.S	0.1%F.S
	Repeatability	±2 g/L	±2 g/L	±2 g/L
	Accuracy	±5 g/L	±5 g/L	±5 g/L
Seawater specific gravity	Measuring principle	Conductivity conversion	Conductivity conversion	Conductivity conversion
	Range	0 to 50 σ_t	0 to 50 σ_t	0 to 50 σ_t
● Display σ_t, σ_0, σ_{15}	Resolution	0.1 σ_t	0.1 σ_t	0.1 σ_t
	Repeatability	±2 σ_t	±2 σ_t	±2 σ_t
	Accuracy	±5 σ_t	±5 σ_t	±5 σ_t
Temperature	Measuring principle	Thermistor method	Thermistor method	Thermistor method
	Range	0 to 55°C	0 to 55°C	0 to 55°C
	Resolution	0.01°C	0.01°C	0.01°C
	Repeatability	±0.3°C	±0.3°C	±0.3°C
	Accuracy	±1.0°C	±1.0°C	±1.0°C

		U-21	U-22	U-23
nductivity (COND)	Measuring principle	Penetration and scattering method	Penetration and scattering method	Penetration and scattering method
Unit selection	Range (NTU or mg/L)	0 to 800NTU	0 to 800NTU	0 to 800NTU
	Resolution	0.1NTU	0.1NTU	0.1NTU
	Repetability	±3%	±3%	±3%
	Accuracy	±5%	±5%	±5%
ater depth	Measuring principle		Pressure method	Pressure method
	Range	-	0 to 100m	0 to 100m
	Resolution	-	0.1m	0.1m
	Repetability	-	±3%	±3%
	Accuracy	-	±5%	±5%
idation-reduction	Measuring principle		Platinum electrode method	Platinum electrode method
tential (ORP)	Range	-	±1999mV	±1999mV
	Resolution	-	1mV	1mV
	Repetability	-	±5mV	±5mV
	Accuracy	-	±15mV	±15mV
ric acid ion	Measuring principle			Ion electrode method
Auto range	Range		-	NO_3^-: 0.62 to 62000mg/L (pH3 to 7)
	Resolution		-	0.1%F.S
	Repetability		-	±5%
	Accuracy	-	-	±10%
loride ion	Measuring principle			Ion electrode method
Auto range	Range	-	-	Cl^-: 0.4 to 35000mg/L (pH3 to 11)
	Resolution	-	-	0.1%F.S
	Repetability	-	-	±5%
	Accuracy	-		±10%
lcium ion	Measuring principle			Ion electrode method
Auto range	Range	-	-	Ca^{2+}: 0.4 to 40080mg/L (pH5 to 11)
	Resolution	-	-	0.1%F.S
	Repetability	-	-	±5%
	Accuracy	-	-	±10%
ultaneity measurement items		7	10	13

otes: Accuracy is indicated for the intermediate point measured in a standard solution after 2-point calibration.
The percentage for repetability and accuracy is for F.S. (except salinity)

7.4 Spare parts

Spare parts list

Sensors

Sensor	Model	Spare part number	Compatible probe
pH sensor	6230	9037-0056-00	U-21/22/23
pH/ORP sensor	6280	9037-0057-00	U-22/23
DO sensor	5460	9037-0058-00	U-21/22/23
Nitric acid ion sensor	6531	9037-0059-00	U-23
Chloride ion sensor	6522	9037-0060-00	U-23
Calcium ion sensor	6533	9037-0061-00	U-23
Ammonia gas sensor	5012	9037-0062-00	U-23
Fluoride ion sensor	6530	9037-0063-00	U-23
Potassium ion sensor	6532	9037-0064-00	U-23

* A chip for ion electrode replacement is also included in the ion sensors.

Cartridges for ion sensor replacement

Chip	Model	Spare part number
Nitric acid ion cartridge	7681	9003-0152-00
Chloride ion cartridge	7660	9003-0150-00
Calcium ion cartridge	7683	9003-0154-00
Potassium ion cartridge	7682	9003-0153-00
Fluoride ion cartridge	7661	9003-0151-00

Standard and internal solutions

Solution	Model	Spare part number
pH 4 standard solution (500 mL)	100-4	9003-0016-00
pH 7 standard solution (500 mL)	100-7	9003-0017-00
pH 9 standard solution (500 mL)	100-9	9003-0018-00
Powder for ORP standard solution (250 mL × 10)	160-51	9003-0031-00
Powder for ORP standard solution (250 mL × 10)	160-22	9003-0030-00
pH reference internal solution (250 mL)	330	9037-0052-00
Ion one-point standard solution (250 mL)	130	9037-0065-00
Nitric acid ion sensor reference internal solution (50 mL)	302	9037-0066-00
Chloride ion sensor reference internal solution (50 mL)	301	9037-0067-00
Calcium and fluoride ion sensor reference internal solution (50 mL)	300	9037-0068-00
Potassium ion sensor reference internal solution (50mL)	303	9037-0069-00
Ammonia gas sensor reference internal solution (50mL)	370	9012-0009-00

hers

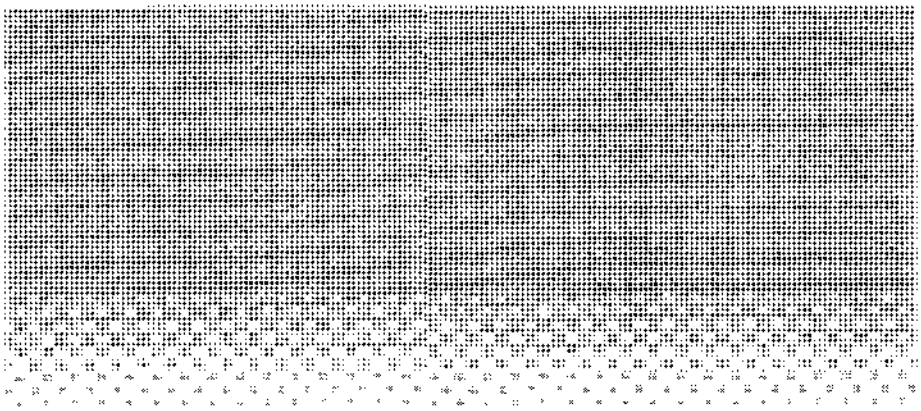
	Model	Spare part number
Diaphragm for ammonia gas sensor (6 pcs.)	–	9037-0070-00
Calibration beaker	–	9037-0073-00
Connector plug for the prove	–	9037-0071-00
Sensor spanner	–	9037-0072-00
DO diaphragm replacement kit		9037-0074-00
Battery cover packing		9096-0013-00
System unit cover O-ring		9096-0014-00

The spare parts above are prepared by dealers.
Order the part by designating the parts name, model and spare parts number.

.5 Option

'arts name	Model
:xpansion adaptor	U-2001
)ystem unit	U-2002
\C adaptor (for 100V)	AC-10

MEMO



8. Reference Data

● pH measurement

1. Principle of pH measurement

U-20 series use the glass electrode method for pH measurements. The glass electrode method measures a potential difference between the glass film for pH and the comparison electrode. For more information, refer to JIS Z 8802 pH measurement method.

2. Temperature compensation

The electromotive force generated by the glass electrode changes depending on the temperature of the solution. Temperature compensation is used to compensate for the change in electromotive force caused by temperature.
This function does not compensate the change in pH caused by the temperature of the solution. When pH is to be measured, the temperature of the solution when the pH is measured must be recorded along with that pH value, even if a meter that has automatic temperature compensation is used. If the solution temperature is not recorded, the results of the pH measurement may be meaningless.

3. Types of standard solutions

When measuring pH, the pH meter must be calibrated using a standard solution. There are five kinds of standard solutions specified in "JIS 28802 pH measurement". For normal measurement, two of standard solutions with a pH of 4, 7, and 9 are sufficient to accurately calibrate the meter.
For standard solutions, refer to "JIS Z 8802 pH measurement".

pH 4 standard solution ..... 0.05 mol/L potassium hydrogen phthalate aqueous solution
(Phthalate)
pH 7 standard solution ..... 0.025 mol/L potassium dihydrogenphosphate, 0.025 mol/L sodium phospate aqueous solution
(Neutral phosphate)
pH 9 standard solution ..... 0.01 mol/L tetra-sodium boric acid aqueous solution
(Borate)

pH values of pH standard solutions at various temperatures settings.

Temp.	pH 4 standard solution	pH 7 standard solution	pH 9 standard solution
(°C)	Phthalate	Neutral phosphate	Borate
0	4.01	6.98	9.46
5	4.01	6.95	9.39
10	4.00	6.92	9.33
15	4.00	6.90	9.27
20	4.00	6.88	9.22
25	4.01	6.86	9.18
30	4.01	6.85	9.14
35	4.02	6.84	9.10
40	4.03	6.84	9.07
45	4.04	6.84	9.04

4. Supplements for pH measurement

Pressure compensation diaphragm

U-20 series can measure pH with high accuracy through the pressure compensation diaphragm without being affected by hydraulic pressure. Attention should be paid to the following points so that the diaphragm may function fully.
Before measurement, use a syringe and fill the reference electrode up to the replenish port with the internal solution.
When injecting the polarity reference internal solution, be carefnl that air bubbles do not get into the solution.

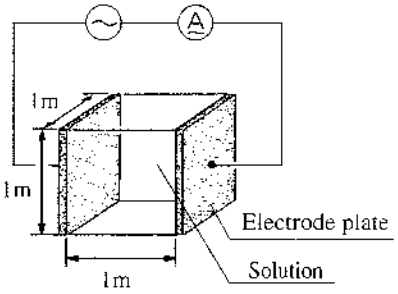
COND measurement

Four-AC-electrode method

nductivity is an index of the flow of electrical current in a substance.

lts dissolved in water are separated into cations and anions. Such solution is called electrolytic solution. Electrolytic lution has the property of allowing the flow of current according to Ohm's law. This property is referred to as ionic nductivity, since current flow is caused by ion movement in electrolytic solution. Metals, on the other hand, allow : flow of current by means of electrons. This property is called electronic conductivity, which is distinguished from nic conductivity.

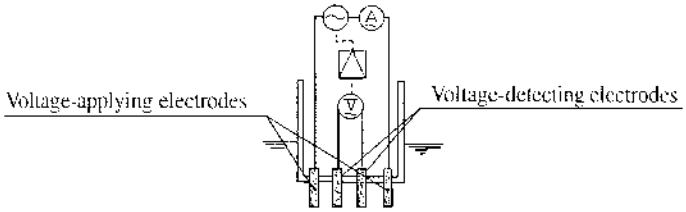
cube with 1 cm on each side, as shown in Fig. 1, is used to demonstrate an electrolytic solution. Two electrode plates : placed on opposite sides, and the cube is filled with a solution. If the resistance between these two electrode plates represented by r (W), the conductivity of the solution L ($S.m^{-1}$) is represented as L=1/r. S stands for Siemens, a unit measurement of conductance.



(Fig. 1 Definition of conductivity)

e most general method for measuring conductivity is based on the above principle, and is called the 2-electrode ethod. In the 2-electrode method the influence of polarization cannot be ignored for solutions with high conductivity d conductivity cannot be measure accurately. In addition, contamination on the surface of the electrode increases parent resistance, resulting in inaccurate measurement of conductivity.

e U-20 series has adopted the 4-electrode method to overcome these disadvantages of the the 2-electrode method. shown in Fig. 2, the U-20 series uses two voltage-detecting electrodes and two voltage-applying electrodes, for a al of four electrodes. The voltage-detecting electrodes are for detecting AC voltage, and the voltage-applying ctrodes are for applying AC voltage.



(Fig. 2 Principle of the 4-electrode method)

r us assume that the current, I (A), flows in a sample of conductivity L – under automatic control of the voltage-plying electrodes – so that the voltage at the voltage detecting-electrodes, E (V), remains constant at all times. en, the resistance of the sample, R (Ω), across the voltage-detecting electrodes is represented as R=E/I. The istance, R, of the sample is inversely proportional to its conductivity, L. Accordingly, a measurement of conductivity, of a standard solution of known conductivity, Ls, enables calculation of conductivity of a sample according to the mula L = Ls (I/Is) from the ratio L : Ls = I : Is.

en in the 4-electrode method, polarization occurs, since AC current flows in the voltage-applying electrodes. The ltage-detecting electrodes are, however, free from the effects of polarization, since they are separated from the ltage-applying electrodes, and furthermore, current flow is negligible. Therefore, the 4-electrode method is an cellent method to enable measurement of conductivity covering a very high range.

2. SI units

New measurement units, called SI units, have been in use from 1996. Accordingly, the U-20 series also uses SI units. The following conversion table is provided for people who use the conventional kind of conductivity meter. Note that along with the change in unit systems, the measnrement values and cell counts have also changed.

	Former nnits →	SI units
Measurement	0.1mS/cm →	0.01S/m
value	1mS/cm →	0.1S/m
	100mS/cm →	10S/m

3. Temperature coeffcient

In general, the conductivity of a solution varies largely with its temperature. The conductivity of a solution depends on the ionic conductivity, described earlier. As the temperature rises, conductivity becomes higher since the movement of the ions becomes more active. The temperature coefficient shows the change in % of conductivity per °C, with a certain temperature taken as the reference temperature. This is expressed in units of %/°C. The temperature coefficient assumes the premise that the conductivity of a sample changes linearly according to temperature. Strictly speaking, with actual samples, however, conductivity changes along a curve. Furthermore, the cuve varies with the type of sample. In the ranges of smaller temperature changes, however, samples are said to have the temperature coefficient of 2 %/°C (at reference tempreture 25°C) this holds for most samples, except in certain special cases.
(The temperature coefficients for various types of solutions are listed on the next page.)
The U-20 series uses an automatic temperature conversion function to calculate conductivity at 25°C at a temperature coefficient of 2 %/°C, based on the measured value of the temperature. Results are displayed on the readout.
The U-20 series's temperature conversion function is based on the following formula.

$$L_{25} = L_t / \{ 1 + K (t - 25) \}$$

L_{25} : Conductivity of solution converted to 25°C
(value displayed on U-20)
t : Temperature of solution at time of measurement (°C)
L_t : Conductivity of solution at t (°C)
K : Temperature coeffcient (%/°C)

Conductivity and temperature coefficient for various types of solutions

Conductivity and related temperature coefficients of representative substances (at 25°C) are shown in the table below.

ubstance	Tempera -ture °C	Concentra -tion wt%	Conducti -vity S/m	Temperature coeffcient %/°C
aOH	15	5	19.69	2.01
		10	31.24	2.17
		15	34.63	2.49
		20	32.70	2.99
OH	15	25.2	54.03	2.09
		29.4	54.34	2.21
		33.6	52.21	2.36
		42	42.12	2.83
H_3	15	0.1	0.0251	2.46
		1.6	0.0867	2.38
		4.01	0.1095	2.50
		8.03	0.1038	2.62
Cl	18	5	39.48	1.58
		10	63.2	1.56
		20	76.15	1.54
		30	66.20	1.54
$_2SO_4$	18	5	20.85	1.21
		10	39.15	1.28
		20	65.27	1.45
		40	68.00	1.78
		50	54.05	1.93
		60	37.26	2.13
		100.14	1.87	0.30
NO_3	18	6.2	31.23	1.47
		12.4	54.18	1.42
		31	78.19	1.39
		49.6	63.41	1.57
$_3PO_4$	15	10	5.68	1.04
		20	11.29	1.14
		40	20.70	1.50
		45	20.87	1.61
		50	20.73	1.74
aCl	18	5	6.72	2.17
		10	12.11	2.14
		15	16.42	2.12
		20	19.57	2.16
		25	21.5	2.27

Substance	Tempera -ture °C	Concentra -tion wt%	Conducti -vity S/m	Temperature coeffcient %/°C
Na_2SO_4	18	5	4.09	2.36
		10	6.87	2.49
		15	8.86	2.56
Na_2CO_3	18	5	4.56	2.52
		10	7.05	2.71
		15	8.36	2.94
KCl	18	5	6.90	2.01
		10	13.59	1.88
		15	20.20	1.79
		20	26.77	1.68
		21	28.10	1.66
KBr	15	5	4.65	2.06
		10	9.28	1.94
		20	19.07	1.77
KCN	15	3.25	5.07	2.07
		6.5	10.26	1.93
NH_4Cl	18	5	9.18	1.98
		10	17.76	1.86
		15	25.86	1.71
		20	33.65	1.61
		25	40.25	1.54
NH_4NO_3	15	5	5.90	2.03
		10	11.17	1.94
		30	28.41	1.68
		50	36.22	1.56
$CuSO_4$	18	2.5	10.90	2.13
		5	18.90	2.16
		10	32.00	2.18
		15	42.10	2.31
CH_3COOH	18	10	15.26	1.69
		15	16.19	1.74
		20	16.05	1.79
		30	14.01	1.86
		40	10.81	1.96
		60	4.56	2.06

● SAL conversion

The U-20 series is designed to measure salinity as well as the other parameters.
Note that the "salinity" referred to here is the salinity of sea water. There is a constant relation between conductivity and salinity at certain temperatures.
Therefore, if data on the conductivity and temperature are available, the corresponding salinity is known. In other words, the salinity measurement of the U-20 series is based on the principle of calculating the salt content, making use of the measured values of conductivity and temperature.
Note therefore, that measured results of all substances whose conductivity is detected are displayed as salinity. For example, the measured result is displayed as NaCl concentration, even if in fact the sample component is, for example, hydrochloric acid (HCl).

● TDS conversion

TDS is short for Total Dissolved Solids and means the total dissolved solid amount.
The conductivity of a solution is affected by the amount of salinity, minerals, and dissolved gases. That is, conductivity is an index that shows the total amount of all substances in the solution. Of these substances, TDS indicates only the amount of dissolved solids.
TDS can be used for a comparison of the state of substances composed of a single component such as NaCl. However, the use of TDS for the comparison of solutions of different types causes serious errors.
Conductivity and TDS are expressed by the following formulas:

Conductivity in SI units (S/m) ..... TDS(g/L) = L (S/m) × K × 10
TDS(g/L) = L (mS/m) × K ÷ 100
Conductivity in the old units (mS/cm) ..... TDS(g/L) = L (mS/cm) × K
K = TDS coefficient

Initial settings use the values listed in the table (☞ Page 70) that generally uses TDS coefficients.
For accurate TDS comparisons, find the TDS coefficient from measured conductivity values. Then set the value thus obtained and make measurements.

● σ_t conversion

Specific gravity of seawater

The density and specific gravity of seawater are equal numerically and generally are not distinguished strictly. Since seawater density ρ is between 1.000 and 1.031, 1 is subtracted from ρ and σ is obtained by multiplying the value by 1000.
The resultant value is used as the specific gravity of seawater.

$$\sigma = (\rho - 1) \cdot 1000$$

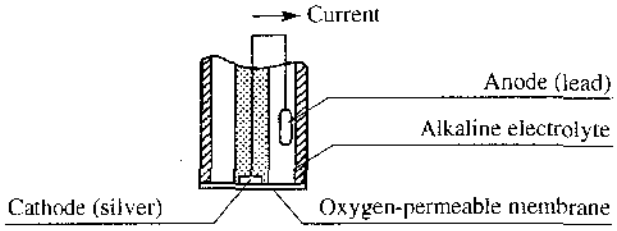
The density of seawater ρ is expressed by temperature, hydraulic pressure, and salinity functions. The density of seawater σ under the atmospheric pressure is expressed as σ_t. The density of seawater under the atmospheric pressure is determined by temperature and salinity.
The U-20 Series models make salinity measurement through temperature measurements and conductivity conversion and findst through calculations.
In Japan σ_{15} at 15°C is called a standard specific gravity and widely used while in foreign countries σ_0 at 0 C is employed. σ_{15} and σ_θ are determined by the function of salinity.
In ocean surveys, in particular, these values σ_t, σ_{15}, and σ_θ are more widely used than conductivity and salinity and, in the U-20 Series models, newly added as measurement components.

DO measurement

Principle of measurement

e "DO" referred to here means the concentration of oxygen dissolved in water. DO is essential to self-purification river and sea and to water creatures such as fish. DO measurement is also essential to drainage and water quality ntrol.

g. 3 shows the principle of measurement using a DO sensor.



(Fig. 3 Principle of DO sensor)

noble metal (silver) is fitted closely to an oxygen-permeable membrane to make the cathode; a base metal (lead) is ed as the anode. Both are immersed in an alkaline electrolyte with the anode-to-cathode external circuit closed. ygen diffusing through the oxygen-permeable membrane causes a reduction reaction at the cathode; this allows w of current in the external circuit:

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$$

the anode, oxidation reaction occur as follows:

$$2Pb \rightarrow 2Pb^{2+} + 4e^-$$

e current is proportional to the quantity of oxygen diffusing through the oxygen-permeable membrane. Accordingly, easurement of the current makes the DO in a sample known.

e DO measuring method based on this principle is called the membrane-electrode method. This method allows nvenient measurement of DO, especially when compared with chemical-analysis method, which needs complicated e-treatment to eliminate the effects of oxidizing or reducing substances.

DO correction for salinity

hen a solution and air are in contact and in complete equilibrium (saturated), DO: C [mg/L] in the solution, and the ygen partial-pressure: Ps [MPa] in air are in the following relation:

$$C = Ps/H$$

[MPa/ (mg/L)] is referred to as Henry's constant, which depends on the composition of the solution. In general, C comes smaller as the salinity in the solution increases, since H becomes larger.

DO sensor is intended to detect Ps in the above expression. Therefore, the DO measurement would be in error if the O sensor were immersed either in air-saturated pure water or in solution with salt. To settle this problem, it is cessary to correct the DO reading based on the salinity of the sample using salinity correction.

nventional DO meters make this salinity correction by inputting a known salinity value. This poses no problems if e salinity of the sample is known. In general, however, the salinity of the sample is usually not known, and the ethod is not practical even if the DO meters are equipped with the salinity correction function.

e U-20 series is capable of measuring the salinity of a sample and automatically correcting the using this function.

Features of the U-20 series DO sensors

conventional DO measurements, it was necessary to keep the velocity of the flow constant because the velocity of w led to fluctuation in indicated values. In our U-20 Series models, improvements in sensors have made it possible make measurements with stable indications and with little influence of the velocity of flow.

● Turbidity (TURB) measurement

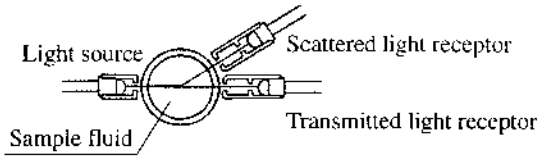
1. Principle of measurement

From among several types of turbidity-uncasuring methods available, the U-20 series uses the light-transmission-scattering method, shown in Fig. 4.

Irradiation of a beam of light onto a sample briugs about separation of the beam into (1) the light transmitted through the solution and (2) the light scattered by turbidity components in the sample. In the light-transmission-scattering method, the intensity of both transmitted light and the scattered light are measured using separate receptors, and the turbidity is obtained based on the ratio of the two.

With the U-20 series, the light source is a pulse-lighting iufrared-emission diode. The scattered light is measured at a point 60° offset from the light source. This light-absorption-scattering method has several advantages, including the fact that (1) the actual color of the sample fluid has little effect on the measurement of turbidity, (2) fluctuations in light quantity from the light source are easily compensated for, and (3) it allow the U-20 series to be operated with relatively low-power consumption.

The turbidity value differs with the structure of the cell so changes with the instrument.



(Fig. 4 Principle of the light-transmission-scattering method)

2. Standard solution

U-20 series can perform calibration using formazin (NTU) or kaolin standard solutions as a turbidity standard solution. However, units for the solution used for calibration should be displayed in measurements. Do not use more than 400 mg/L of kaolin standard solution because it increases precipitation speed, resulting in measurement error.

● DEP measurement

1. Depth (DEP) measurement

For the U-22 and U-23 models, depth measurement can be made through use of a pressure gauge. The principle of the depth measurement uses the relation between depth and pressure.

Although the measurement with the depth sensor is affected by atmospheric pressure, the depth sensor, however, makes zero-point adjustments through the automatic calibration before measurements.

2. Influence of temperature and calibration

The depth sensor depends greatly on temperature. For a wide difference between the temperature at which the sensor has been automatically calibrated and the temperature of the measurement sample, the sensor can make depth measurements with a higher accuracy by the following method:

Immerse the depth sensor of the sensor probe into the sample.

Keep the sensor immersed in the sample for approximately 30 minutes until the temperatures of the sensor and the sample are the same.

Then make the zero calibration of the sensor manually. (☞ Page 55)

) Measuring mV (oxidation-reduction potential (ORP))

RP principles

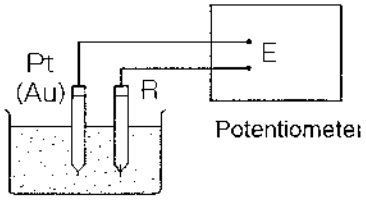
RP (or "redox potential") is an abbreviation for oxidation-reduction potential. ORP is the energy level (potential) :termined according to the state of equilibrium between the oxidants (M^{Z+}) and reductants $M^{(Z-N)+}$) that coexist within solution.

$M^{Z+} + ne^- \Leftrightarrow M^{(Z-n)+}$ .....①

only ① exists within a solution, a metal electrode (platinum, gold, etc.) and a reference electrode are inserted into e solution, forming the ORP measuring system shown in Fig. 5. Measuring the potential (ORP) that exists between e two electrodes enables the potential to generally be expressed by the following equation.

$$E = E_0 - \frac{RT}{nF} \ln \frac{a_{M^{(Z-n)+}}}{a_{M^{Z+}}} \quad \text{..... ②}$$

E: Electric potential E_0 : Constant R: Gas constant T: Absolute temperature
n: Electron count F : Faraday constant a : Activity



(Fig. 5 Measuring mV)

r example, for a solution in which trivalent iron ions coexist with bivalent iron ions, equations ① and ② would be follows.

$Fe^{3+} + e^- \Leftrightarrow Fe^{2+}$ .....①

$$E = E_0' - \frac{RT}{F} \ln \frac{a_{Fe^{2+}}}{a_{Fe^{3+}}} \quad \text{.....②}$$

hen only one type of state of equilibrium 1 exists in the solution, the ORP of the solution can be determined iquely by equation 2. What is important here is that ORP is determined by the ratio of activity between the oxidant e^{3+}) and the reductant (Fe^{2+}) (using the equation $a_{Fe^{2+}}/a_{Fe^{3+}}$). Actually, however many kinds of states of equilibrium ist simultaneously between various kinds of ions, in most solutions. This means that under actual circumstances, RP cannot be expressed using the simple equation shown above and that the physical and chemical significance with spect to the solution is not very clear.

this respect, the value of ORP must be understood to be only one indicator of the property of a solution. The asurement of ORP is widely used, however, as an important index in the analysis of solutions (potentiametric ration) and in the disposal and treatment of solutions.

cently, there have appeared various claims regarding this matter, such as that a high degree of ORP is effective in rilization or that drinking water that has a low ORP reduces the chance of illness by reacting with the activated ygen in the cells of the body. ORP is used as an index for alkaline drinking water.

● Ion measurement

1. Ion concentration measurement

When certain ions exist within the solution that is to be measured, the responsive ion sensor membrane generates an electric potential corresponding to the concentration of the ions. The potential that is generated is measured by the ion meter as potential, using the reference electrode as the standard. With ion sensors, the measured potential and the logarithm of the ion activity within the solution being measured are generally proportional to each other and are expressed in the following way.

$$E = Eo + (2.303\ RT/nF)\ log\ [\ \gamma\ C]$$

E : Measured electric potential (V)

Eo : Standard potential (V), determined according to the system. This includes the standard potential of the reference electrode and the liquid junction potential.

F : Faraday constant (96.485 $Cmol^{-1}$)

R : General gas constant (8.314 $JK^{-1}\ mol^{-1}$)

T : Absolute temperature (K)

n : Ion valency

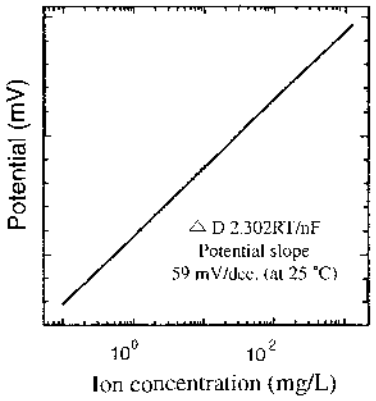
γ : Activity coefficient

C : Ion concentration (mol/L)

The above formula is called "Nernst's equation" and is the basis for measuring ion concentration using an ion sensor. The part of the above Nernst's equation that reads "2.303 RT/nF" is the change in potential generated when the ion concentration changes by a factor of 10.

This change in potential is called the "potential slope," "incline," "slope," or "Nernst's factor." If the above equation is adhered to when calibrating with standard solution and determining the value of the potential slope and E_0, finding the potential E of the ion sensor inside the solution being measured will enable the ion concentration to be determined. When actual measurement is performed, the ion sensor measures the ion concentration, so a linear relationship forms between the value of the ion concentration and the electrode potential, if the concentration is plotted on a logarithmic axis, as shown in Fig.6. Conducting quantitative analysis using an ion sensor requires either an ion meter that has an logarithm calculation function or the creation of a calibration curve using semi-logarithmic graph paper.

Calibration curve for univalent positive ion electrode



(Fig. 6 Relationship between ion concentration and electric potential)

Standard solution

ding the ion concentration of the solution being measured requires prior calibration of the ion meter using a pared standard solution with a known ion concentration. The number of times the meter is to be calibrated depends the accuracy desired. Calibration is usually performed once a day or prior to making measurements. Calibrating meter when the standard solution has been mixed using a stirrer or other utensil will improve the electrode ponsiveness and measurement stability.

lasically, at least two standard solutions of different concentrations should be used to calibrate this meter. If the pproximate ion concentration of the liquid to be measured is known, standard solutions having lower and higher oncentrations than that liquid should be used for calibration. In such cases, the standard solution with the lower ion oncentration should have 1/10 the concentration of the standard solution with the higher concentration.

f the approximate ion concentration of the liquid to be measured is unknown, choose low and high-concentration tandard solutions with a larger differential than the 1/10 used in the above example. However, be sure not to xceed the limits of the linear ion sensor detection capabilities.

3. Notes on use of ion sensors

<Measuring range>

Use measured values in the ion concentration range for individual ion sensors.

<pH range>

There is a pH range suitable for measurements for each ion sensor. Make ion measurements in the pH range.

<Hindering ion>

Some ion sensors respond to other ions than ions to be measured. Smaller permissible coexistence limits in the table below cause more serious errors. For ion sensors that cannot withstand hindering ions, the responsive membrane will be broken. Handle ion sensors with care.

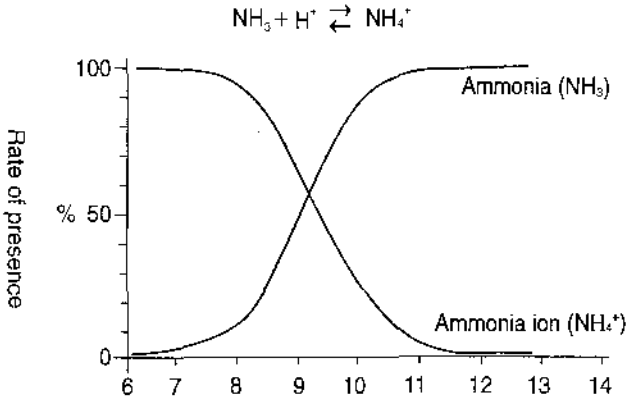
Model Ion type	Sensor type (Quantity)	① Measurement range ② pH range	Influence of hindering ions The values in parentheses show permissible coexistence limits.	Valency
6522 Cl^-	#7660 (1 piece)	① 0.4 to 35,000mg/L Cl^- (1 to 10^{-5}mol/L Cl^-) ② pH3 to 11 (350mg/L Cl^-)	Not measurable: $S_2O_3^{2-}$, S^{2-}, I^-, Ag^+, Hg^{2+} Br^- (0.03), MnO_4^- (0.1), SCN^- (0.3) (In the10^{-3} mol/L Cl^-)	−1
6530 F^-	#7661 (1 piece)	① 0.02 to 19,000mg/L F^- (1 to 10^{-6}mol/L F^-) ② pH4 to 10 (20mg/L F^-)	OH^- (10) (In the measurement range)	−1
6531 NO_3^-	#7681 (1 piece)	① 0.62 to 62,000mg/L NO_3^- (1 to 10^{-6}mol/L NO_3^-) ② pH3 to 7 (62mg/L NO_3^-)	ClO_4^- (0.02), I^- (0.1), NO_2^- (3), Cl^- (40) F^- (200), CH_3COO^- (300) (In the10^{-3} mol/L NO_3^-)	−1
6532 K^+	#7682 (1 piece)	① 0.04 to 39,000mg/L K^+ (1 to 10^{-6}mol/L K^+) ② pH5 to 11 (3.9mg/L K^+)	Rb^+ (0.4), Cs^+ (3), NH_4^+ (70) (In the10^{-4} mol/L K^+)	+1
6533 Ca^{2+}	#7683 (1 piece)	① 0.4 to 40,080mg/L Ca^{2+} (1 to 10^{-5}mol/L Ca^{2+}) ② pH5 to 11 (3.9mg/L Ca^{2+})	Fe^{3+} (0.1), Fe^{2+}, Zn^{2+} (1), Sr^{2+} (50), Ni^{2+}, Cu^{2+} (70), Co^{2+} (350), Mn^{2+} (500) (In the10^{-4} mol/L Ca^{2+})	+2
5012 NH_3	—	① 0.1 to 1,000mg/NH_3 (0.1 to 10^{-5}mol/L NH_3) ② Some exist as NH_3 at pH 8 or more. All ammonia components exist as NH_3 at pH 12 or more.	Substance that emits acid and basic gases (Volatile amine)	−1

Important

- Because of the above properties, the NO_3^- ion sensors cannot be used in seawater.

1. Properties of the ammonia ion sensor

The optional ammonia sensor for the U-23 model measures ammonia gas (NH_3).
Ammonia gas (NH_3), which is a component in water, and ammonia ion NH_4^+ exist differently depending on pH. (Fig. 7)



(Fig. 7 The pH rates of ammonia and ammonia ions in water.)

Before making measurements of only the ammonia (NH_3) in the sample, therefore, it is necessary to only immerse the ion sensor into the sample. Before making measurements of all ammonia components, it is necessary to change ammonia ion (NH_4^+) into ammonia gas (NH_3) by pouring the sample into the calibration beaker and adding approximately 0.3 g of sodium hydride.

MEMO

LaMotte

2020 TURBIDIMETER

Instruction MANUAL

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GENERAL INFORMATION

PACKAGING & DELIVERY

Experienced packaging personnel at LaMotte Company assure adequate protection against normal hazards encountered in transportation of shipments. After the product leaves the manufacturer, all responsibility for safe delivery is assured by the transportation company. Damage claims must be filed immediately with the transportation company to receive compensation for damaged goods.

Should it be necessary to return the instrument for repair or servicing, pack the instrument carefully in a suitable container with adequate packing material. A return authorization number must be obtained from LaMotte Company by calling 1-800-344-3100 or faxing 1-410-778-6394. Attach a letter with the authorization number to the shipping carton which describes the reason for the return. This information will enable the service department to make the required repairs more efficiently.

GENERAL PRECAUTIONS

Read the instruction manual before attempting to set up or operate this instrument. Failure to do so could result in personal injury or damage to the instrument.

The 2020 Turbidimeter should not be stored or used in a wet or corrosive environment. Care should be taken to prevent water from wet turbidity tubes from entering the turbidimeter light chamber.

NEVER PUT WET TUBES IN THE TURBIDIMETER.

SAFETY PRECAUTIONS

Read the label on all reagent containers. Some labels include precautionary notices and first aid information. Certain reagents are considered hazardous substances and are designated with a * in the instruction manual. Material Safety Data Sheets (MSDS) are supplied for these reagents. Read accompanying MSDS before using these reagents. Additional emergency information for all LaMotte reagents is available 24 hours a day from the Poison Control Center listed in the front of the phone book. Be prepared to supply the name and four digit LaMotte code number found on the container label or at the top of the MSDS. LaMotte reagents are registered with a computerized poison control information system available to all local poison control centers.

LIMITS OF LIABILITY

Under no circumstances shall LaMotte Company be liable for loss of life, property, profits, or other damages incurred through the use or misuse of their products.

SPECIFICATIONS

Instrument Type	Nephelometric turbidity, calibrated in NTU
Range	0.00 -1100 NTU
Accuracy	.05 or ±2% for readings below 100 NTU, whichever is greater ±3% above 100 NTU
Resolution	*Standard Mode* 0.01 from 0.00 -10.99 NTU 0.1 from 11.0 -109.9 NTU 1 from 110 -1100 NTU *EPA Mode* NTU — Reported to the nearest NTU 0 - 1.0 — 0.05 1.0 - 10 — 0.1 10 - 40 — 1 40 - 100 — 5 100 - 400 — 10 400 - 1000 — 50 1000 — 100
Display	3½ digits
Response Time	5 seconds
Warm-up time	Not required
Automatic Shut Off	2 minutes
Lamp	Tungsten Filament bulb (approximate life 800 hours)
Sample	15 mL in capped tube
Sample Chamber	Accepts 25mm diameter flat-bottomed tubes (capped)
Power source	Battery Operation: 9 Volt Alkaline Line Operation: 120V/50Hz, 220V/60Hz*, with supplied adapter
Size (L X W X H)	8.5 x 16.2 x 6.7 cm, 3.4 X 6.4 X 2.6 inches
Shipping Weight	Meter only: 11 oz. (312g) Kit: 3 lb. 7 oz. (1560g)
Serial Interface	RS232, 8 pin mDIN, 9600b, 8, 1, n

**CE Mark: The devise complies to the product specifications for the Low Voltage Directive when furnished with the 220V AC Adapter (Code 1774). The 120V AC adapter is not CE approved.*

PARTS & ACCESSORIES

Included in the Model 2020 Turbidity Meter Kit (Code 1799 OR 1799-EX2):

Code	*Item*
26856	2020 Turbidity Meter
1726-110	AC Adapter, 9V (or 1726-220 with 1799-EX2)
1476	AMCO™ 2020 Turbidity Standard, 1.0 NTU, 60 mL
1477	AMCO™ 2020 Turbidity Standard, 10 NTU, 60 mL
0286-4	Turbidity tubes, set of 4

Optional Accessories:

1478	AMCO™ 2020 Turbidity Standard, 100 NTU, 60 mL
1479	AMCO™ 2020 Turbidity Standard, 250 NTU, 60 mL
1800	High Turbidity Dilution Kit includes: Syringe, Filter Holder, Membrane Filters
0943	Syringe
0598	Filter holder
1103-6	Membrane Filters, 0.45 micron, pkg of 6
5115PS-H	Deionized Water, 60 mL
6195-H	Formazin Turbidity Standard, 4000 NTU, 60 mL

EPA COMPLIANCE

This instrument meets or exceeds EPA design specifications for NPDWR and NPDES turbidity monitoring programs as specified by the USEPA method 180.1. There is also a compliance reading mode which rounds the reading to meet EPA reporting requirements.

WARRANTY

This instrument is guaranteed to be free from defects in material and workmanship for one year from original purchase date. If within that time the instrument is found to be defective, it will be repaired without charge except for transportation costs. The guarantee does not cover batteries.

CE COMPLIANCE

The 2020 Turbidimeter has been independently tested and has earned the European CE Mark of compliance for electromagnetic compatibility and safety.

DECLARATION OF CONFORMITY

Application of Council Directives:	89/336/EEC
Standards to which Conformity Declared:	EN55022, EN50082-1, En600950
Manufacturer's Name:	LaMotte Company
Manufacturer's Address:	802 Washington Avenue PO Box 329 Chestertown, MD 21620
Importer's Name:	Reagecon Diagnostics Ltd
Importer's Address:	13 A/D Shannon Free Zone Shannon, Co. Clase. Ireland
Type of Equipment:	Water Quality Meters
Model Number:	2020/1200
Year of Manufacture:	1997

I, the undersigned, hereby declare that the equipment specified above conforms to the above Directive and Standards.

Chestertown, Maryland
Place

James K. Trumbauer
Signature

3-19-97
Date

James K. Trumbauer
Name

V.P., Director of Research & Development
Position

NOTE: The device complies to the product specifications for the Low Voltage Directive when furnished with the 220V AC Adapter (Code 1774).

WHAT IS TURBIDITY?

Turbidity, cloudiness in water, can be interpreted as an absence of clarity or brilliance. It is caused by suspended and colloidal matter such as clay, silt, organic and inorganic matter and microscopic organisms. Turbidity should not be confused with color since a darkly colored water can still be clear and not turbid.

Turbid water is often an indicator of conditions that could cause damage to manufacturing equipment. Water clarity is especially important to the producers of consumer products such as beverage producers, food processors and water treatment plants. The particulates that cause turbidity may not always be harmful to human health, but are considered an undesirable characteristic.

Turbidity in industrial water used for boiler and cooling systems should be as low as possible. In boilers, the particles may become concentrated and settle out as a sludge that will damage equipment and cause foaming. In cooling water systems, particles can interfere with corrosion inhibitors. Water clarity is improved with fluid-particle separation processes such as sedimentation, coagulation and filtration.

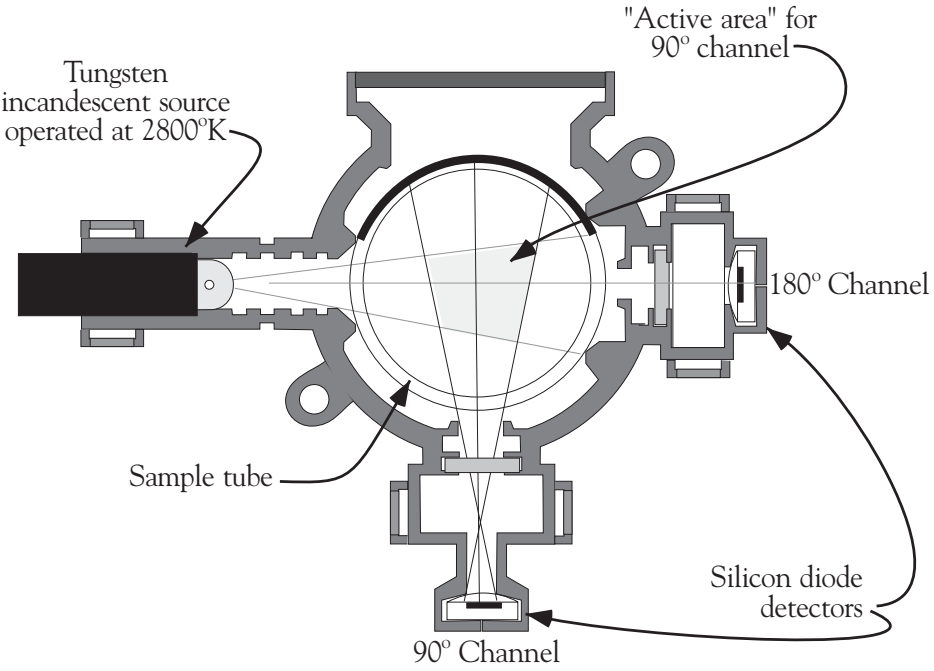
In swimming pools, cloudy water is a common problem. The usual causes for poor water clarity are corrosion, improper filtration and/or improperly balanced water. An algae condition or severe chloramine condition can also cloud pool water.

In natural waters, turbidity is an indicator of quality and productivity and can be used to monitor the health of streams and lakes. Turbid water may indicate runoff from construction, agriculture or other types of pollution. Suspended sediment can carry nutrients and pesticides throughout the water system. Suspended particles near the surface absorb additional heat from sunlight, raising the water temperature and blocking out the light needed by submerged aquatic vegetation and bottom dwelling creatures.

HOW IS TURBIDITY MEASURED?

Light passing through clear water will travel in a straight line. Particles in turbid water will cause the light to scatter giving it a "cloudy" appearance. The turbidity of a sample is determined by measuring the amount of scatter when a light is passed through a sample. The higher the turbidity, the greater the amount of scatter.

Turbidity can be measured in many ways. Visual methods include, the comparative methods, the Secchi disk method and the Jackson Candle method. Comparative methods are used in shallow water and determine turbidity by matching the turbidity of a water sample to a standard of known turbidity either with a "target" at the bottom of a tube or with a turbidity comparator. In the deeper waters of lakes, ponds, rivers and estuaries the Secchi disk is often used to measure turbidity. The Secchi disk is a disk about eight inches in diameter that is either white or is marked with black and white quadrants. The disk is lowered into the water on a calibrated line and the depth is noted where the disk just disappears from sight. The disk is then raised until it is visible. The average of these two distances is known as the "Secchi depth".



2020 Nephelometer

At waterworks and wastewater treatment plants the Jackson Candle apparatus was a standard instrument for measuring turbidities of incoming raw waters and treated wastewater effluents for many years. The equipment was modified over time but originally it consisted of a long glass tube supported over a "standard candle." Water was added to or removed from the tube until the image of the candle flame became indistinct. The depth of the water in the tube was read off a calibrated scale etched into the side of the tube, and results were reported numerically as Jackson Turbidity Units (JTU). The lowest turbidity that can be determined with this method is 25 Nephelometric Turbidity Units (NTU). Since the EPA's Surface Water Treatment requirements state that, finish water from municipal treatment plants will have a turbidity less than 1 NTU, indirect methods were developed to measure turbidity. Turbidimeters are the preferred method.

Nephelometers, such as the 2020, are turbidimeters that measure the scattered light at 90 degrees from the light source. A reference beam passes through the sample and is measured at 180 degrees. The ratio of these two readings is electronically converted to a turbidity measurement in NTU.

GENERAL OPERATING INFORMATION

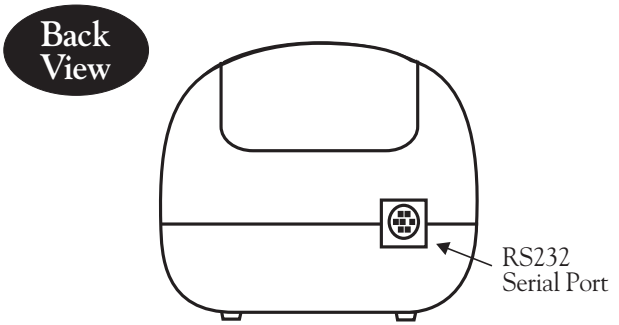
OVERVIEW

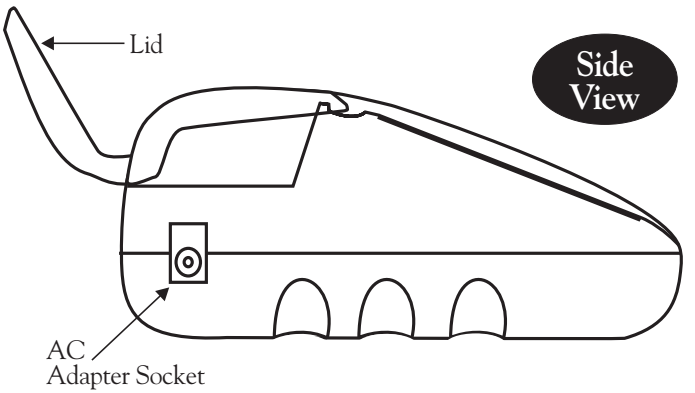
The 2020 Turbidimeter is a portable, microprocessor controlled nephelometer. A multi-detector optical configuration assures long term stability and minimizes stray light and color interferences. All readings are determined by the process of signal averaging over a 5 second period, minimizing fluctuations in readings attributed to large particles and enabling rapid, repeatable measurements. It has a sealed keypad. The microprocessor enables auto-ranging over the full range of 0 to 1100 NTU and provides direct digital readout with a resolution of 0.01 NTU for the lowest range.

The optics feature a tungsten bulb light source with a life expectancy of 800 hours. The light is detected by a silicon photo diode.

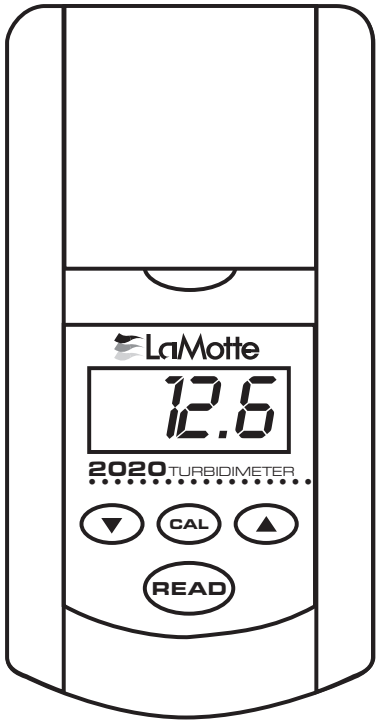
The 2020 is supplied with a 9 volt alkaline battery and an AC power adapter. A fresh battery should be installed at all times, even when using the power adapter. This will ensure that the meter will power down properly.

A RS-232 serial port on the back of the meter allows an interface of the turbidimeter with an IBM compatible computer for real time data acquisition and data storage using the PC. This port also allows an interface with a RS-232 serial printer.

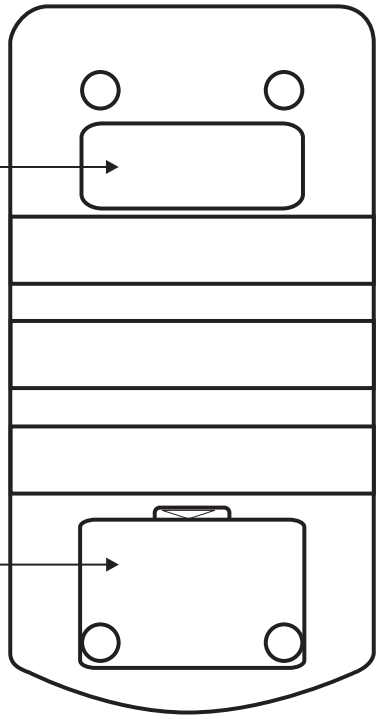




Top View



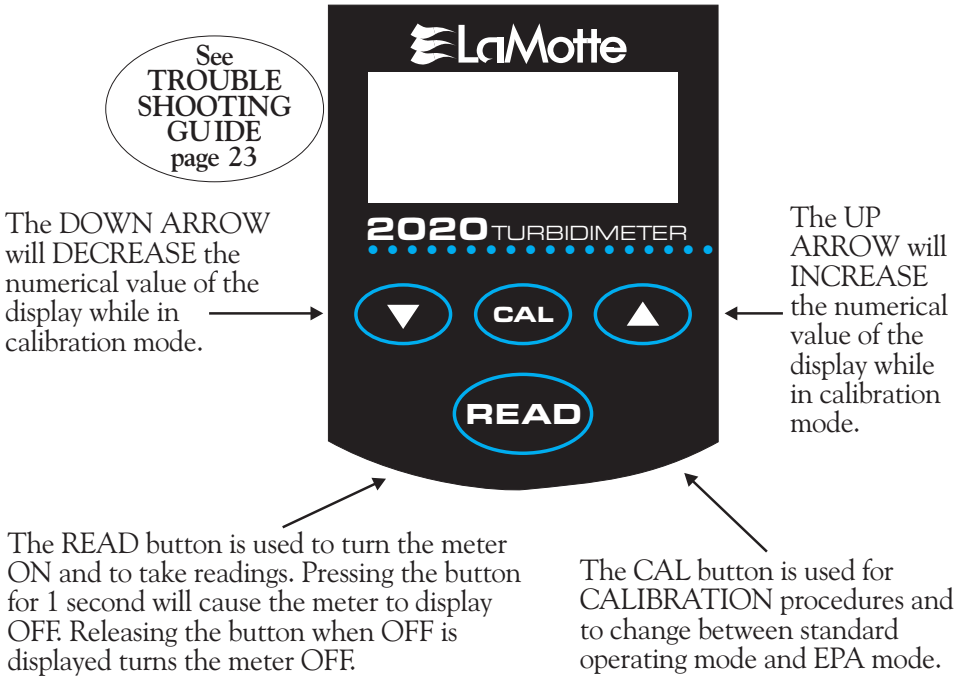
Bottom View



THE KEYPAD

The DISPLAY will display turbidity reading with the following resolution:
0.00 - 10.99 NTU; 11.0 - 109.9 NTU; 110 - 1100 NTU

- When the **READ** button is first pushed, a number will be briefly displayed that indicates the software version number.
- A walking dash "–" will be displayed when measurement is taking place.
- The display will flash after the **CAL** button has been pushed during the calibration procedure until the **CAL** button has been pushed again to enter the adjusted value.
- "*OFF*" will be displayed after the **READ** button has been held down for 1 second. The meter will turn off when the button is released.
- "*ER1*" will be displayed when the battery voltage is very low.
- "*ER2*" will be displayed when measured turbidity is over range (1100 NTU).
- "*ER3*" will be displayed when the bulb has burned out or the tube is misaligned.
- "*BAT*" will be displayed when the battery voltage is getting low. Readings are reliable. The meter should not be operated if the battery low indicator has appeared. Replace battery as soon as possible.
- "▲" will be displayed when the meter is in EPA mode.

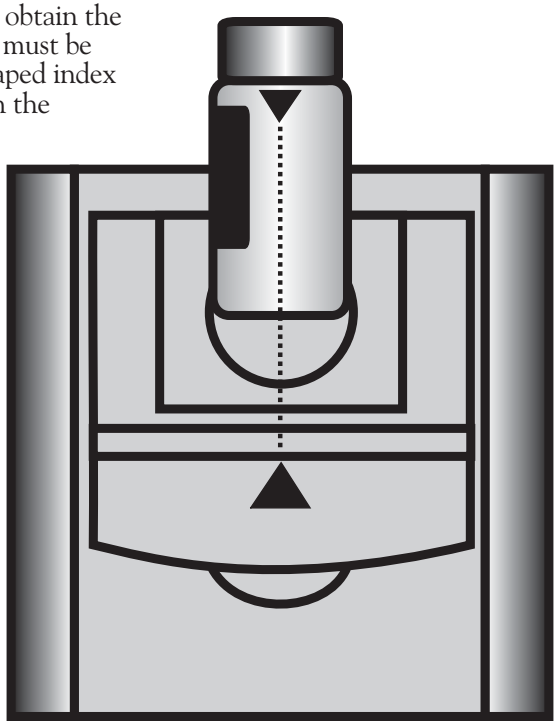


TURBIDITY TUBES

Turbidity tubes should always be washed prior to use. Use a mild detergent to remove any dirt or finger prints. Dry the outside of the turbidity tubes with a clean, lint-free cloth or disposable wipe. Allow the turbidity tubes to air-dry in an inverted position to prevent dust from entering the tube.

The handling of the turbidity tubes is of utmost importance. Scratches, fingerprints and water droplets on the turbidity tube or inside the light chamber can cause stray light interference leading to inaccurate results. It is imperative that the turbidity tubes and light chamber be clean and dry. Scratches and abrasions will permanently affect the accuracy of the readings. The inside of the tubes can be acid washed periodically and coated with special silicon oil to mask imperfections in the glass. Avoid acid contact with the black ink on the outside of the tubes. After a tube has been filled and capped, it should be held by the cap and the outside surface should be wiped with a clean, lint-free absorbent cloth until it is dry and smudge-free. Handling the tube only by the cap will avoid problems from fingerprints. Always set the clean tube aside on a clean surface that will not contaminate the tube.

Variability in the geometry and quality of the glassware is the predominate cause of variability in results. The special anti-reflective area on the 2020 tubes allows more accurate turbidity readings for low NTU samples. Only 2020 tubes should be used with the 2020 turbidimeter. Orientation of the tube in the chamber will greatly affect the test results. To obtain the most accurate results, the tubes must be positioned so that the arrow-shaped index mark on the tube is aligned with the arrow-shaped index mark molded into the housing in front of the light chamber. This will ensure that the most accurate results are obtained.

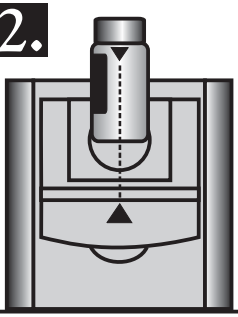


The turbidity tubes are optically selected but very small variations in the tubes may cause different readings on the same sample in low turbidity water. If greater accuracy is required, such as for drinking water requirements, the tubes supplied with the 2020 should be individually calibrated. This procedure is important for reading below 10 NTU but is not needed for samples above 10 NTU.

1.

Fill each tube (0286) with high-quality water. (Generally distilled water or drinking water is sufficient.)

2.

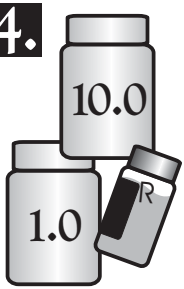


Record a turbidity reading for each tube following the instructions for turbidity measurement on page 19.

3.

Mark the tube with the lowest reading with an "R", for reference turbidity tube, near the top of the tube with a permanent marker.

4.

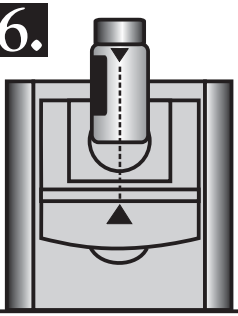


Follow the Calibration Procedure on page 17 using the reference turbidity tube, turbidity-free water, and the 1.00 NTU or 10.0 NTU AMCO™ standard.

5.

Fill the remaining turbidity tubes with the same AMCO™ turbidity standard as in the reference turbidity tube.

6.



Insert each tube into the meter with the proper orientation, Close lid and record the reading for each tube.

7.

The difference between the theoretical value (1.00 NTU or 10.0 NTU) and the actual value is the correction factor for that tube. This factor should be used when comparing results from different tubes.

CALIBRATION

STANDARD SOLUTIONS

The 2020 has been pre-calibrated in the range of 0 to 1100 NTU with AMCO™ primary standards manufactured by Advanced Polymer Systems, Inc. This allows the 2020 to be used for treated water, natural water or wastewater. Recalibration of the 2020 by the user is not required. However, a procedure to standardize the calibration should be performed to obtain the most accurate readings over a narrow range.

Two AMCO™ standards of 1.00 NTU and 10.0 NTU are supplied with the 2020. Standards of other values are available as accessories. The standards are a suspension of uniformly sized plastic "micro spheres" in ultra pure water, which require no preparation and are stable for long periods of time. These standards were manufactured specifically as a reference to calibrate the 2020. Only LaMotte specific AMCO™ standards should be used with the 2020. These standards are guaranteed to be accurate to within ±1%, if the following precautions are observed:

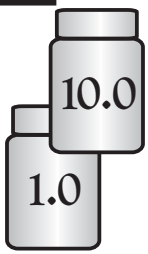
- The standards will remain stable for up to one year after the fill date.
- Never pour any unused or used standard back into the primary standard bottle.
- Do not open the bottle in a dusty or dirty environment. Dust and contaminants from the air can ruin the quality of the standard solutions.
- Before filling a tube with a standard, rinse the inside of the tube with a small amount of standard.
- Cap the standard bottle and the tube immediately after filling.

With proper preparation techniques, freshly prepared Formazin standards should be equivalent to the AMCO™ standards and can be used for meter calibration. A 4000 NTU Formazin Standard is available from LaMotte Company for use in preparing calibration standards. (See "Optional Accessories," pg. 6.) Correct procedures and approved methods for the use of Formazin standards can be found in the current edition of Standard Methods for Examination of Water and Wastewater.

Note: Change battery when ERI (very low battery) is displayed. Operating the meter with a very low battery could result in permanantly erasing the calibration.

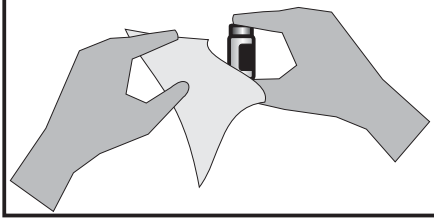
CALIBRATION PROCEDURE

1.

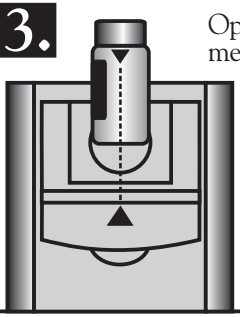


Select a LaMotte AMCO™ 2020 Standard in the range of the samples to be tested. NOTE: Only use LaMotte AMCO™ Standards specific to the 2020 Turbidimeterr. Contact LaMotte for replacement standards.

2. Fill a turbidity tube with the standard, cap, and wipe the tube clean with a lint-free cloth.

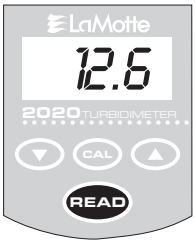


3.

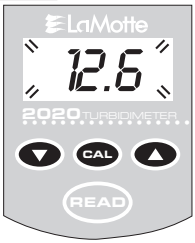


Open the lid of the meter. Align the indexing arrow mark on the tube with the indexing arrow mark on the meter, and insert the tube into the chamber.

4. Close the lid. Push the **READ** button. If the displayed value is not the same as the value of the reacted standard (within the specification limits), continue with the calibration procedure.

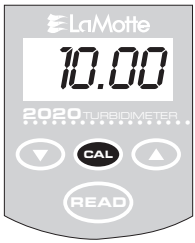


5.



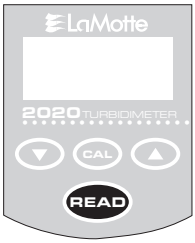
Push the **CAL** button for 5 seconds until **CAL** is displayed. Release button. The display will flash. Adjust the display with the ▼ and ▲ buttons until the value of the standard is displayed.

6.



Push the **CAL** button again to memorize the calibration. The 2020 display will stop flashing. Calibration is complete.

7.



Turn the unit off by holding the **READ** button down for at least 1 second, or proceed to measure the test samples following the procedure on page 19.

Note

The calibration procedure should be followed once a week, or more often as required by regulations and laws for compliance monitoring. The calibration of the meter is independent of the operating mode.

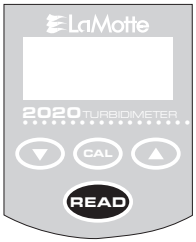
ANALYSIS PROCEDURES

SELECTING THE EPA MODE

The 2020 turbidity meter has two operating modes, the standard operating mode and the EPA mode. The meter can only be switched from one mode to the other while turning the 2020 on, from the OFF state. The 2020 will remain in which ever mode it was last used, even if the meter has been turned OFF.

To switch from one mode to the other mode:

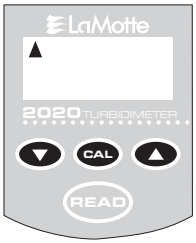
1.



Turn OFF the 2020, if it is on.

2.

Press **CAL** button and hold it down while pressing the **READ** button to turn the meter on.



The meter will come on in the opposite mode than it was in previously. (While in EPA mode the ▲ will be visible on the display).

The standard operating mode displays the measured turbidity to the full resolution of the meter. The EPA mode displays the measured turbidity rounded to the reporting requirements of the EPA and Standard Methods compliance monitoring programs. This greatly simplifies the reporting requirements by eliminating the need for the user to manually round off the results according to EPA specifications. The EPA requires these reporting requirements because it recognizes the inherent accuracy of turbidity measurements within the specified ranges.

Note: The calibration of the meter is independent of the operating mode.

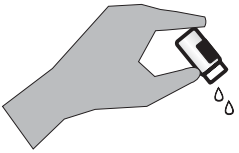
TURBIDITY MEASUREMENT

1.

Fill a clean container with at least 50 mL of sample water and cover. Set sample aside to allow sample to equilibrate to air temperature and let gases escape. Avoid contaminants. Analyze as soon as possible.

2.

Rinse an empty turbidity tube with a portion of the sample. Shake out excess water.

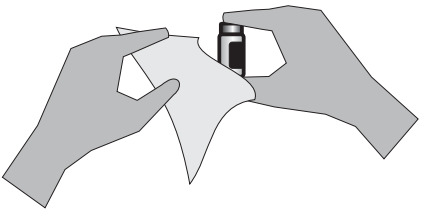


3.

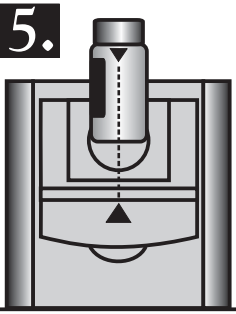
Fill the turbidity tube (0286) to the neck by carefully pouring the sample down the side of the tube to avoid creating bubbles.

4.

Cap the tube and wipe tube dry with a clean lint-free tissue.

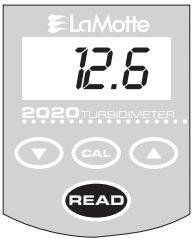


5.



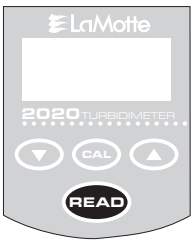
Open the 2020 lid. Align the indexing arrow on the tube with the indexing arrow on the meter. Insert the turbidity tube into chamber.

6.



Close the lid. Push the **READ** button. The turbidity in NTU units will be displayed within 5 seconds.

7.



The 2020 will turn off automatically 2 minutes after the last button push. To turn the meter OFF manually, hold the **READ** button down for at least 1 second. Release the button when OFF is displayed.

Note

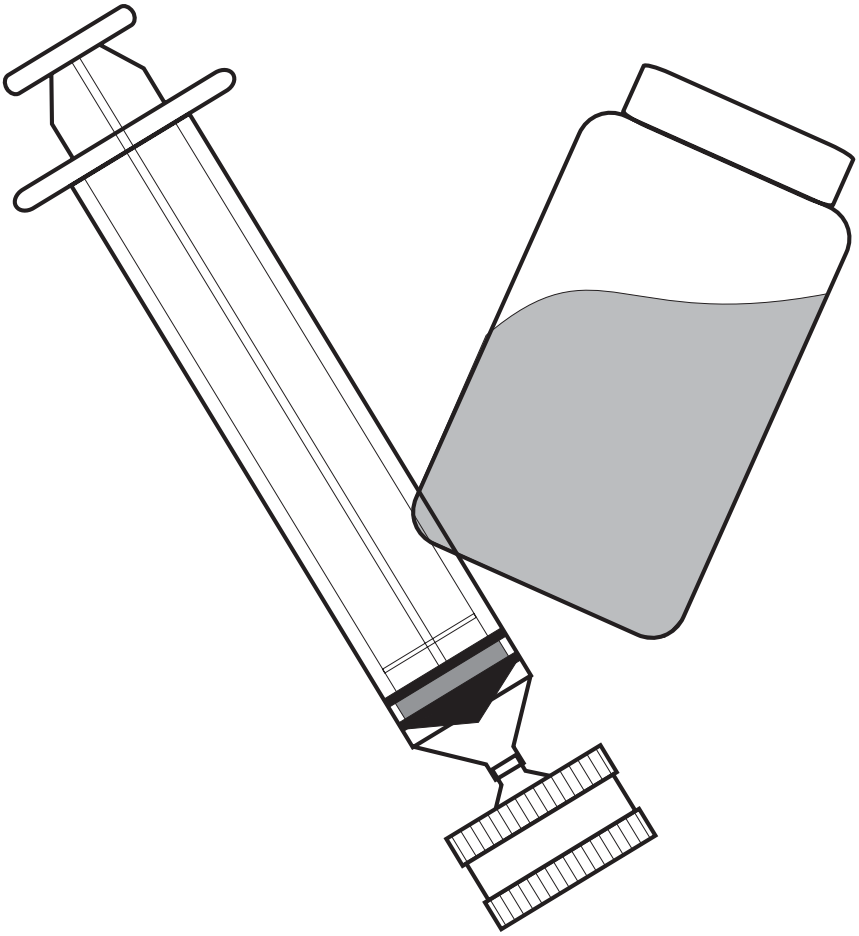
If the sample is higher than 1100 NTU, it must be diluted and retested. See pages 20-22.

PREPARATION OF TURBIDITY FREE WATER

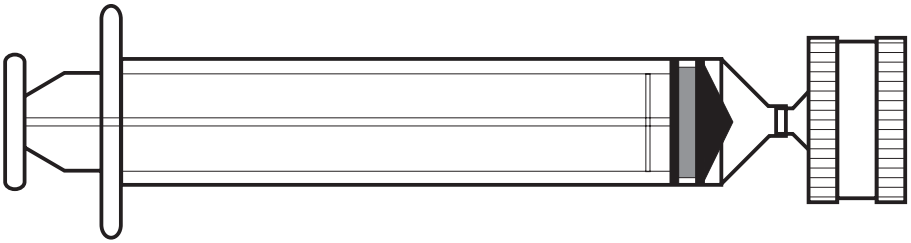
An accessory package (Code 1800, not included) is available for preparing turbidity free water for dilution of high turbidity samples.

The preparation of turbidity free water requires careful technique. Introduction of any foreign matter will affect the turbidity reading. A filtering device with a special membrane filter is used to prepare turbidity-free water. The filter, filter holder, and syringe must be conditioned by forcing at least two syringes full of deionized water through the filtering apparatus to remove foreign matter. The first and second rinses should be discarded. Turbidity-free water as prepared below may be stored in the dark at room temperature in a clean glass bottle with a screw cap and used as required. The storage container should be rinsed thoroughly with filtered deionized water before filling. The water should be periodically inspected for foreign matter in bright light.

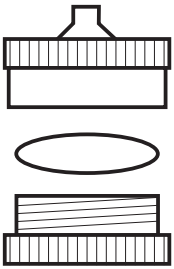
See procedure on next page...



PROCEDURE:



1.

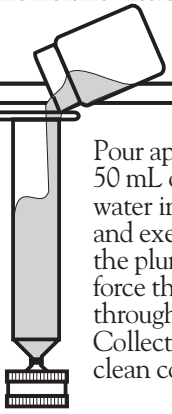


Unscrew the top of the filter holder (0598). (The black O-Ring should remain in the top part of the filter holder). Place a white membrane filter (1103) on the screen inside. Position the filter disk so that it covers the entire surface of the screen. Replace the top of the filter holder and screw on securely. Note: the membrane filters are white and packaged between two blue protective disks. Handle membrane filters with extreme care.

2.

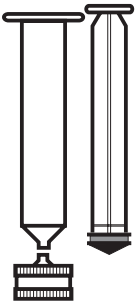
Remove the plunger from the syringe (0943). Attach filter holder to the bottom of the syringe.

3.



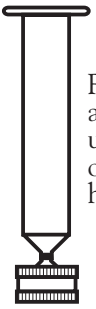
Pour approximately 50 mL of deionized water into the barrel and exert pressure on the plunger to slowly force the water through the filter. Collect water in a clean container.

4.



Remove the filter holder from the syringe, then remove the plunger from the barrel. (This step is required to prevent rupturing the membrane filter by the vacuum as the plunger is removed.)

5.



Replace the filter holder and repeat steps 3 and 4 until the desired amount of turbidity-free water has been collected.

Periodically examine the membrane filter to insure that no holes or cracks are present. Depending on the nature of the unfiltered water, it is possible to prepare a liter or more of turbidity-free water using a single filter. The membrane filter may be stored in the holder indefinitely and used as required.

DILUTION PROCEDURES

If a sample is encountered that is higher than 1100 NTU, a careful dilution will bring the sample into the acceptable range. However, there is no guarantee that halving the concentration will exactly halve the NTU values. The particulates often react in an unpredictable manner when diluted.

TESTING TIPS

1. Samples should be collected in a clean glass or polyethylene container.
2. Samples should be analyzed as soon as possible after collection.
3. Discard tubes that are badly scratched.
4. Gently mix sample by inverting before taking a reading but avoid introducing air bubbles.
5. Turbidity readings will be affected by electric fields around motors.
6. Carbon in the sample will absorb light and cause low readings.
7. Observe shelf life recommendations for turbidity standards.
8. The turbidimeter should be placed on a surface free from vibration. Vibrations can cause high readings.
9. Excessive color in a sample will absorb light and cause high readings. The user should verify if a certain level of color will cause a significant error at the level of turbidity being tested.

TROUBLESHOOTING

PROBLEM	CHECK	ACTION
No Power	Battery	Replace
	AC Adapter	Plug in
	AC Wall Outlet	Verify power source
	Contact LaMotte for Return Authorization	Return to LaMotte for repair
Suspect Calibration	Check calibration with standards	Use new standards
	Verify standards with Formazin	Run alternate test with Formazin
	Verify with another meter	Check other meter calibrations
	Check tube alignment	Re-align tube
	Check sample tubes for dirt and scratches	Check, clean and/or replace if necessary
	Check to see if internal meter components are wet	Always dry tubes before inserting. Examine chamber for visible moisture.
	Reset meter to factory calibration	With meter off, hold down ▼ and press **READ**
	Battery	A fresh battery should be installed at all times, even when using the power adapter. This will ensure that the meter will power down properly and the calibration will not be lost. Return meter for recalibration.
	Contact LaMotte for Return Authorization	Return for calibration check
ER1	Very low battery	Change battery immediately (see pg. 16)
ER2	Over range	Dilute sample
ER3	Burnt out bulb or misaligned tube	Check alignment Call LaMotte
BAT	Low Battery	Change battery

RS232 PORT

The 2020 Turbidimeter may be interfaced with any IBM compatible computer using an Interface cable (Code 1772). The meter may also be interfaced with an RS-232 serial printer, using an appropriate cable and setting the printer configuration to the output below.

Output: RS232 compatible, asynchronous serial, 9600 baud, no parity, 8 data bits, 1 stop bit.

Computer Connection: RS232 interface connection, 8 pin mDIN/9 pin F D-submin.

Pin out:

5	RS-232 TxD
3	RS-232 RxD
4, 6, 8	digital ground

MAINTENANCE

REPLACING THE BATTERY

The LaMotte 2020 uses a standard 9-volt alkaline battery that is available worldwide. A fresh battery should be installed at all times, even when using the power adapter. This will ensure that the meter will power down properly. The battery compartment is located on the bottom of the case. To replace the battery:

1. Open the battery compartment lid
2. Remove the battery and disconnect the battery from the polarized plug.
3. Carefully connect the new battery to the polarized plug and insert it into the compartment.
4. Close the battery compartment lid

REPLACING THE LAMP

The tungsten lamp included with the model 2020 has a life of approximately 800 hours. If the display becomes unstable when using LaMotte AMCO™ standards, call LaMotte Company for a return authorization number to have the lamp replaced and have the unit examined.

REPAIRS

If it is necessary to return the instrument for repair, telephone LaMotte Company at 1-800-344-3100 or fax 1-410-778-6394 for a return authorization number.

LaMOTTE COMPANY

Helping People Solve Analytical Challenges®

PO Box 329 • Chestertown • Maryland • 21620 • USA
800-344-3100 • 410-778-3100 (Outside U.S.A.) • Fax 410-778-6394
Visit us on the web at www.lamotte.com

08/04 • 61799

Maintenance Guide for Oil / Water Interface Meters
For the H.01L and Sm.Oil

This interface is designed to measure the thickness of floating product (L.N.A.P.L.). It will also detect and measure sinking layers of L.N.A.P.L.s.

This instrument has been designed and built to give years of reliable and accurate use.

Please follow the simple guidelines given in this booklet for the best performance from your unit.

Signals indicate the following:
The continuous tone and light indicates product (hydrocarbon). Intermittent tone and light indicates water.

General Hints

Cold Weather
In cold weather condensation will form on the lens as it enters the well and contacts the warmer moist air in the well. This causes the unit to sound as product. To overcome this allow the probe to allimatise in the well or lower the probe into the water, the n take readings.

A dirty or scratche probe lens aggravates this problem.

Removing The Probe
This requires two wrenches a 1/2"AF and 9/16"AF. When the knurled nut has been loosened the link may be still tight in the probe. Gently separate the link from the probe by hand. Care must be taken to avoid either breaking the wires while removing the probe or crimping the wire while replacing the probe.

Do...
- Avoid sharp edged casing
- Avoid entanglement with other equipment in boreholes and wells.
- Clean and dry probe after use
- Rewind tape onto reel after each use

Warranty is conditional upon adherence to these guide lines.

Do Not...
- Do not use to plumb borehole depths
- Do not use as guide to backfilling with sand etc. Instrument may get locked

in sand.

Remember to...

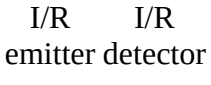
- CLEAN THE UNIT AFTER USE
- AVOID SCRATCHING THE LENS
- HANG THE UNIT ON THE CASING WHERE POSSIBLE, AND RUN THE TAPE OVER THE FRAME LEG. THIS PROTECTS THE TAPE AGAINST CUTS AND NICKS
- THIS IS A TOOL TO HELP YOU TO DO YOUR JOB WELL. TREAT IT PROPERLY AND IT WILL NOT LET YOU DOWN

IF YOU HAVE ANY OTHER QUESTIONS OR COMMENTS
CALL 1-800-331-2032 OR YOUR SUPPLIER.

Tips For Using The Oil/Water Interface Meter

THE WAY IT WORKS

IN AIR the infrared emitter sends out an I/R beam inside a 45 degree prism.



The I/R beam reflects off the internal face of the prism to the opposite face, which then reflects the beam to the detector.

In this state the instrument is silent.
IN FLUIDS which are denser than air, the SNELL principle operates.
The I/R beam is not reflected internally, but exits the prism. The detector does not detect the I/R beam.

In this state the instrument is emitts a tone and the red light is on.
At the same time the two conductivity probes are testing for conductivity.
If the fluid does not conduct electricity, the sound tone and light are solid (product) If the fluid does conduct electricity, the tone is intermittent (water).

Care of Reel

Remove electronic front panel before wash down.

Reel may be cleaned with the following:

Aliphatics: Hexane, Heptane, White Kerosene, Mineral Spirits

Alcohols: Methyl, Isopropyl, Isobutyl
1 + 3 Denatured Alcohol

Halogenated Hydrocarbons: Freons TF + TE

Others: Soap solution Naptha Alconox 10%, Fantastic®, Windex®, Joy®, Top Job®, Mr. Clean®, Formula 409®.

® Registered Trade Marks

Care of Tape and Probe

Tape and Probe may be cleaned and de-greased with the following:

Alcohols: Hexane, Heptane

Aliphatics: Hexane, Heptane, White Kerosene, Mineral Spirits

Halogenated Hydrocarbons: Fully Halogenated Freon

Others: Soap Solution, Naptha Alconox 10%, Joy detergent® 10%, Lestoil®

Do Not clean with abrasives.
The Probe lens is made from an Amorphous Thermoplastic, highly resistant to many chemicals, however, contact with the following should be avoided or approached with caution:
Chloroform I.I.2. Cresol, Iodine, Oleum, Resorcinol

Partially Halogenated Hydrocarbons such as Methylene Chloride, Phenol (saturated solution) should be approached with care. If exposure to any of the above causes damage to the instrument

English

INSTRUCTIONS
PRESS BUTTON = METER ON FOR 4 MINUTES
GREEN LIGHT ON = METER ACTIVE - BATTERY GOOD
GREEN LIGHT OFF = METER OFF OR BATTERY NO GOOD

INTERFACE METER

GROUND METER WITH LEAD PROVIDED
OIL STEADY LIGHTS AND BUZZER TONE
TAKE MEASUREMENTS GOING THROUGH THE OIL INTO THE WATER, THEN BACK FROM THE WATER INTO THE OIL - REPEAT TO GET CONSTANT READING

AFTER USE
REMOVE EXCESS MOISTURE
CLEAN PROBE AND TAPE WITH SOAP AND WATER
• DO NOT USE ABRASIVES OR SOLVENTS •
PROTECT AND MAINTAIN POLISHED PROBE TIP FOR TOTAL WASHDOWN - REMOVE ELECTRONIC PANEL FROM REEL. DO NOT WASH ELECTRONICS.
DRY REEL AND REPLACE ELECTRONIC PANEL.

Probe Electronics

1	0100-41	Tape Connector	**5**	0100-35/43	Probe Body
2	0100-34	Link/Tape Connector	**6**	0100-38 (WLM)-36(1/F)	1/F Lens.WLM Tip.
3	0100-42	Link	**7**	0100-39	Conductivity Pins
4	0100-40	Link/Probe Connector	**8**	0100-45/44	Reel Electronics

Note: Items 1 to 8 are servicable only by supplier.

Reel Electronics

1	01001A1B	Frame	**15**	0100-3	Front Plate
2	0100-9	Nut	**16**	0100-5	Cranking Handle
3	0100-23	Screw	**17**	0100-11	Spindle
4	0100-6/24	Brake	**18**	0100-18	O Ring
5	0100-18	Spacer	**19**	0100-56	Electric Disk
6	0100-8	Shaft	**20**	0100-54	Disk Handle
7	0100-13	Bearing	**21**	0100-55/68	Disk Retaining Knob

8	0100-15	Ground Ring	**22**	0100-71	Screw
9	0100-62	Screw	**23**	0100-59/60	Tape Screws
10	0100-20	Screw	**24**	0100-53	Battery Holder
11	0100-7	Frame Boot	**25**	0100-48/49	Battery L.E.D.
12	0100-2	Rear Plate	**26**	0100-46/47	Oil/Water L.E.D.
13	0100-4	Hub	**27**	0100-50/51	On/Off Button
14	0100-12	Screw	**28**	0100-14	Bearing Retainer

MODEL 580B

ORGANIC VAPOR METER (OVM) / DATALOGGER

INSTRUCTION MANUAL
P/N 16860

THIS EQUIPMENT IS SUITABLE FOR USE IN CLASS I, DIVISION 2, GROUPS (AS APPLICABLE) OR NON-HAZARDOUS LOCATIONS ONLY.

WARNING - EXPLOSION HAZARD - SUBSTITUTION OF COMPONENTS MAY IMPAIR SUITABILITY FOR CLASS I, DIVISION 2.

THERMO ENVIRONMENTAL INSTRUMENTS INC
8 WEST FORGE PARKWAY
FRANKLIN, MASSACHUSETTS 02038

TELEPHONE: (508) 520-0430
FACSIMILE: (508) 520-1460

1-9-96

INSTRUMENT WARRANTY

WARRANTY. Subject to the exceptions stated below, Thermo Environmental Instruments Inc. agrees to correct, either by repair or at our opinion, by replacement, any defects in materials or workmanship which develop within one year from the date of surface shipment, parts and labor supplied free of charge and surface transportation costs to be borne by the offeror both ways, provided that the investigation and inspection defects developed under normal and proper use.

The exceptions mentioned above are: (1) All items claimed must be returned to Thermo Environmental Instruments Inc., transportation charges collect, and will be shipped prepaid and charged to the customer unless the item is found to be defective and covered by the warranty in which case Thermo Environmental Instruments Inc. will pay all surface transportation charges; (2) Thermo Environmental Instruments Inc. agrees to extend to the customer whatever warranty is given to Thermo Environmental Instruments Inc. and incorporated into products sold to the customer; (3) Thermo Environmental Instruments Inc. shall be released from all obligations under this warranty in the event repairs or modifications are made by persons other than its own authorized service personnel, or service personnel from an authorized representative, unless such repair is minor, merely the installation of a new plug-in component; (4) If any model or sample was shown to the Purchaser, such model or sample was shown merely to illustrate the article and not to represent that any article delivered hereunder would conform to the model or sample; and (5) Spare parts are warranted for ninety (90) days.

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580B USER MANUAL
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SECTION I

INTRODUCTION

1 INTRODUCTION

The 580B is a portable Organic Vapor Meter (OVM), which detects and quantitates most organic vapors with a highly sensitive photoionization detector (PID). The 580B has an operating range of 0-2000 parts per million (ppm) with a minimum detectable of 0.1 ppm. No support gases are required.

The 580B is controlled by a microprocessor which provides many features that were not previously available. Maximum signal hold, detector linearization, overrange lockout, IBM PC (or compatible) interface, extensive data logging capabilities and much more. With the many features provided by the 580B leak detection, head space measurements, and field survey are all easily accomplished. Completely portable, the 580B operates from internal batteries for eight hours in the field.

1.1 ABOUT THIS MANUAL

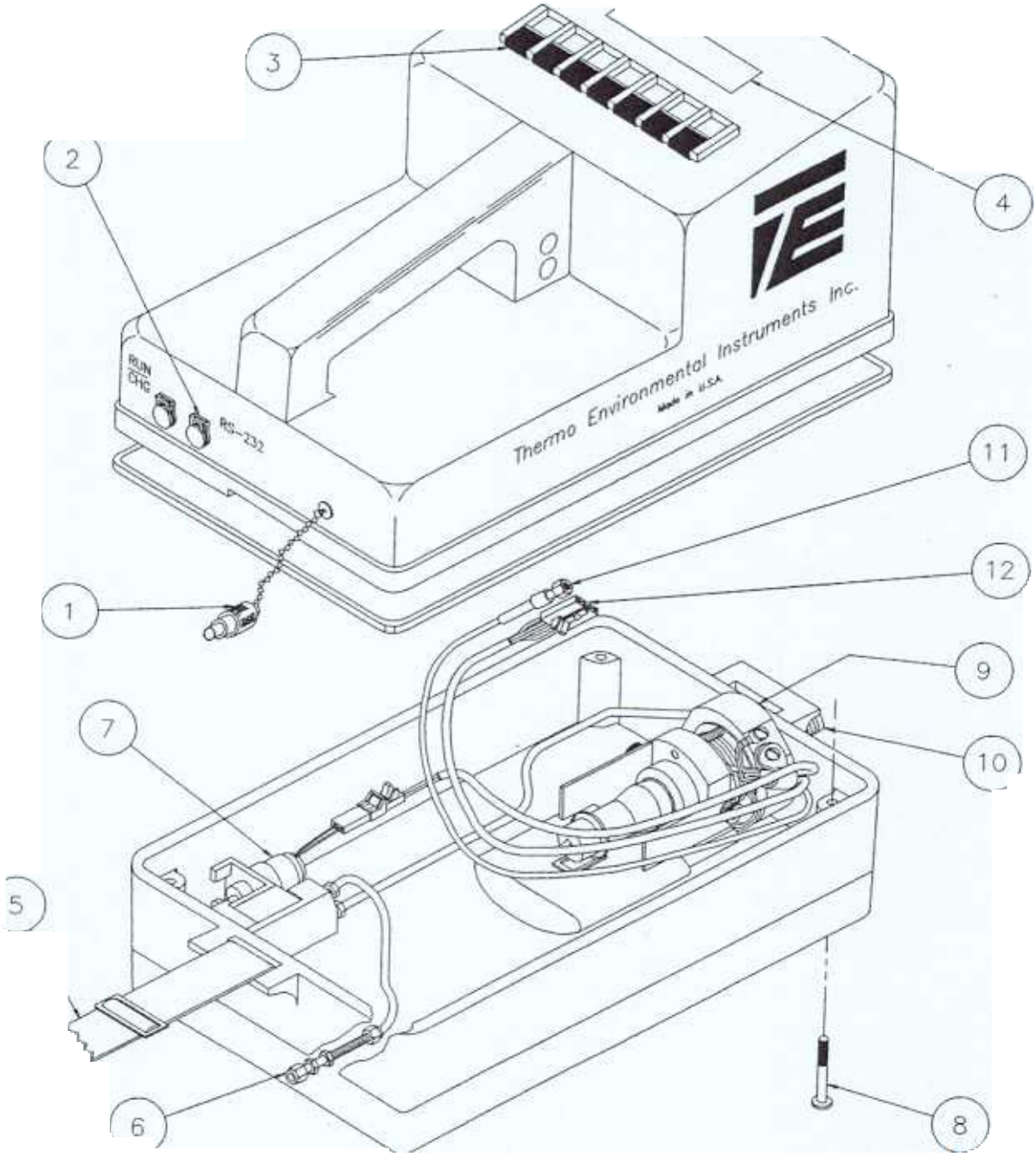
This manual is broken down into eight chapters. The first chapter (this one) provides a general overview of the 580B. Chapter two discusses, in great detail, the extensive facilities of the 580B. The focus of this chapter is on how to use the seven switches to access the various facilities. Chapter three explains, in detail, how to perform routine maintenance on the 580B. Chapter four is a technical discussion of calibration and methods for generating standards. Chapter five is a technical discussion of a few applications which illustrate some of the uses of the 580B. Chapter six is a technical discussion of methods for collecting a sample using the 580B. Chapter seven is a discussion of the communication facilities provided by the 580B. Chapter eight contains two flow charts which illustrate the 580B software flow. This chapter is a helpful tool for the new user. Appendix A is a detailed explanation of the 580B communication protocol. This chapter is provided in order to allow a programmer to develop specialized communication software for the 580B. There are several other addendums which contain miscellaneous information about the 580B.

1.2 INSTRUMENT OVERVIEW

This section describes various points of interest on the 580B. Each number refers to a number in Figure 1.1.

1. **POWER PLUG** - The power plug is used to run the instrument from its internal batteries. There is a chain attached to the power plug so that it will not be lost.

2. **RS-232 CONNECTOR** - This connector is used for communi-



Figur

rumen As: emb

cation with a serial printer or computer. A communication cable provided with the instrument fits into the receptacle.

3. **KEY PAD** - There are seven switches which operate the 580B. The switch marked ON/OFF is used to turn the pump and lamp on and off. The switch marked LIGHT will turn on backlighting for the two line display. The other five switches perform various functions. For a detailed description of the function of each switch see chapter two or the flow charts in chapter eight.

4. **DISPLAY** - The 580B has a two line by sixteen character display.

5. **SHOULDER STRAP** - There is an adjustable shoulder strap for carrying the 580B.

6. **SAMPLE EXIT PORT** - The 580B sample is drawn into the detector by a positive displacement pump and then sent back out through the exit port.

NOTE : The photoionization detector is a non destructive detector so the sample may be collected at the exit for further analysis (see Chapter Six).

7. **PUMP** - The 580B pump draws the sample into the detector.

8. **MOUNTING SCREWS** - There are four captive screws which hold the 580B top and bottom together. The screws are specially designed so that they do not fall out when they are loosened out of the case top.

9. **DETECTOR** - The photoionization detector is shown with the lamp and high voltage power supply.

10. **SAMPLE INLET** - Sample is drawn into the detector through the sample inlet at the front of the 580B.

11. **SIGNAL CABLE** - The PID signal is brought up to the microprocessor, for analysis, via the coaxial signal cable.

12. **BASE HARNESS** - The base harness plugs into a connector on the case top.

1.3 580B FEATURES

This section provides a brief overview of the various features of the 580B. After reading this section the user should have a good idea of what the instrument can do. Chapters two and three will explain, in detail, how each feature is selected.

TURNING ON PUMP AND LAMP - The pump and lamp are turned on by pressing the ON/OFF switch (the instrument power must already be on).

CALIBRATION - Calibration of the 580B is extremely impor-

tant. Chapter two explains how to calibrate the 580B in great detail. Chapter four discusses at length some of the basic theory and methods behind calibration. It is strongly suggested that this chapter be read in order to gain a deeper understanding of usage of the 580B. Chapter three also discusses calibration.

CONCENTRATIONS - Once the lamp and pump have been turned on the 580B begins to display the concentration of the incoming sample on the bottom line of the display. Normally the top line of the display will be a bar graph (logarithmic on a scale of zero to 2000). The operator may however select the MAX HOLD mode of operation. When in MAX HOLD, the top line of the display will show the highest concentration recorded.

LOGGING - The 580B provides extensive facilities for logging information. The operator may save a particular reading along with a six digit location code and a date and time stamp. If the 580B is in the MAX HOLD mode when logging is initiated then the max hold value will be logged.

AUTO LOGGING - Logging may be performed automatically by using the 580B's auto logging feature. Auto logging is not allowed while in the MAX HOLD mode. When auto logging is selected a LOGGING INTERVAL is selected (anywhere from one second to 99 minutes and 59 seconds). At the end of each logging interval the present concentration will be logged (the location code is automatically incremented each time).

AVERAGE - The 580B normally updates the concentration once per second. The operator has the option of setting the averaging time anywhere from one second up to four minutes.

NOTE : The bottom line of the display will be blank until the first averaging interval is completed. The top line will however be updated each second.

RESPONSE FACTOR - A response factor may be used in order to relate a particular gas to the calibration gas. When computing the displayed concentration the microprocessor multiplies the measured concentration by the response factor and displays the result. If the response factor is one, then the concentration is not changed. Chapters four and five explain some uses of the response factor.

LAMP SELECTION - The 580B allows for calibration data to be saved for one 10.0 eV lamp and one 11.8 eV lamp. This allows lamps to be switched in the field without requiring recalibration. A lamp serial number may also be entered.

ALARM - An alarm level may be selected. The 580B will sound an audible alarm (the top line will also indicate an alarm) whenever the concentration goes above the selected alarm level.

ACCESS - The 580B provides four access levels so that various features may be "locked out." User identification number

and instrument number are also provided.

CLOCK - The 580B has an internal clock which will run even when the instrument power is cut off.

COMMUNICATION - The 580B has a serial communication port for outputing data to a serial printer. Many of the 580B features may be accessed from a remote computer through the serial communication port (there is communication software available which will run on an IBM PC or clone).

DISPLAY LOGGED DATA - The logged data may be displayed on the 580B's two line display.

SECTION II

PRINCIPAL OF OPERATION

PHOTOIONIZATION DETECTOR OPERATION & THEORY

1 GENERAL

The sample is drawn into the ion chamber by a pump down stream of the detector. Here the sample is bombarded by ultra-violet light (UV) exciting the molecule. If the energy, (hU) of the UV light is greater than the ionization potential (IP) of the sample molecule (R) an electron will be removed, ionizing the molecule. A positively charged molecule and a free electron are produced, as :

$$R + hu \rightarrow R^+ + e^-$$

Several typical reactions follow:

C_6H_6 (benzene) + hu -> $C_6H_6^+ + e^-$ IP = 9.2 eV

$H_2C{=}CHCl$ (VCM) + hu -> $H_2C{=}CHCl^+ + e^-$ IP = 9.9 eV

C_3H_8 (propane) + hu -> $C_3H_8^+ + e^-$ IP = 10.9 eV

For this reason the ionization potential of the subject molecule plays an important role in selecting the lamp energy. Ionization potentials are expressed in electron volts (eV). A list of ionization potentials can be found in Appendix E of this manual or a more complete list in the CRC "Handbook of Chemistry and Physics".

2.1.2 LAMP ENERGIES

There are three lamps available from TEI, 10.0 eV, 10.6 eV, and 11.8 eV. The different energies are obtained by filling the lamp envelopes with different gases and selecting a window which will pass the wavelength produced when the gas is excited. The combination of gas and windows which produce these energies are listed below:

ENERGY	GAS	WINDOW	WAVELENGTH (nm)
10.0 eV	Krypton	MgF	123.6
10.6 eV	Krypton	MgF	117.4
11.8 eV	Argon	LiF	105.1

Though ionization potential will help the user select a lamp, it will not give any information as to the performance of

the detector in measuring a specific compound. The response of the system varies considerably from compound to compound even though they may have the same ionization potential. Some generalizations may help the user obtain a feeling for the difference in response between compounds.

2.1.3 COMPARATIVE RESPONSE

The following is an idealized response chart. No attempt is made to quantitate the relationship, it's a guideline.

Decreasing PID Response:
- Aromatic Compounds
- Unsaturated Compounds
- Saturated Compounds
- Ketones
- Alcohols
- Compounds with Sub Groups

It becomes obvious that sensitivity is influenced by the electronegativity of the molecule though this is not a predictable measure of performance. The only true test of performance is to measure the specific compound of interest and compare it to a good performing standard such as isobutylene.

2.1.4 RESPONSE FACTORS

This relative comparison with isobutylene mentioned above is a very effective way of measuring a variety of compounds without the need to recalibrate for each compound.

The development of a RESPONSE FACTOR allows the operator to correct the instrument's response given a one to one correspondence for all compounds measured, using isobutylene as the reference standard.

Note: Because there is variation in lamp production and hence performance, it is suggested that all calibration and subsequent development of response factors be done on the same lamp/instrument combination.

The preparation of standards and the development of response factors is discussed in subsequent sections of this manual. Once the response factor is generated, it is entered into the 580. The instrument automatically reports the concentration of the compound measured in relative units. It is important to recognize that all compounds measured at that time will be reported relative to the response factor entered in the instrument. For example, if we have calibrated the instrument on isobutylene and have entered a response factor for benzene, we will read concentrations with a one to one correspondence to benzene. If during these measurements toluene or any other compound is encountered, the instrument will report the concentration as if it was measuring benzene. For this reason care should be taken when using this facility.

The above discussion should give the reader a good overview of PID performance. To further understand the intricacies of the

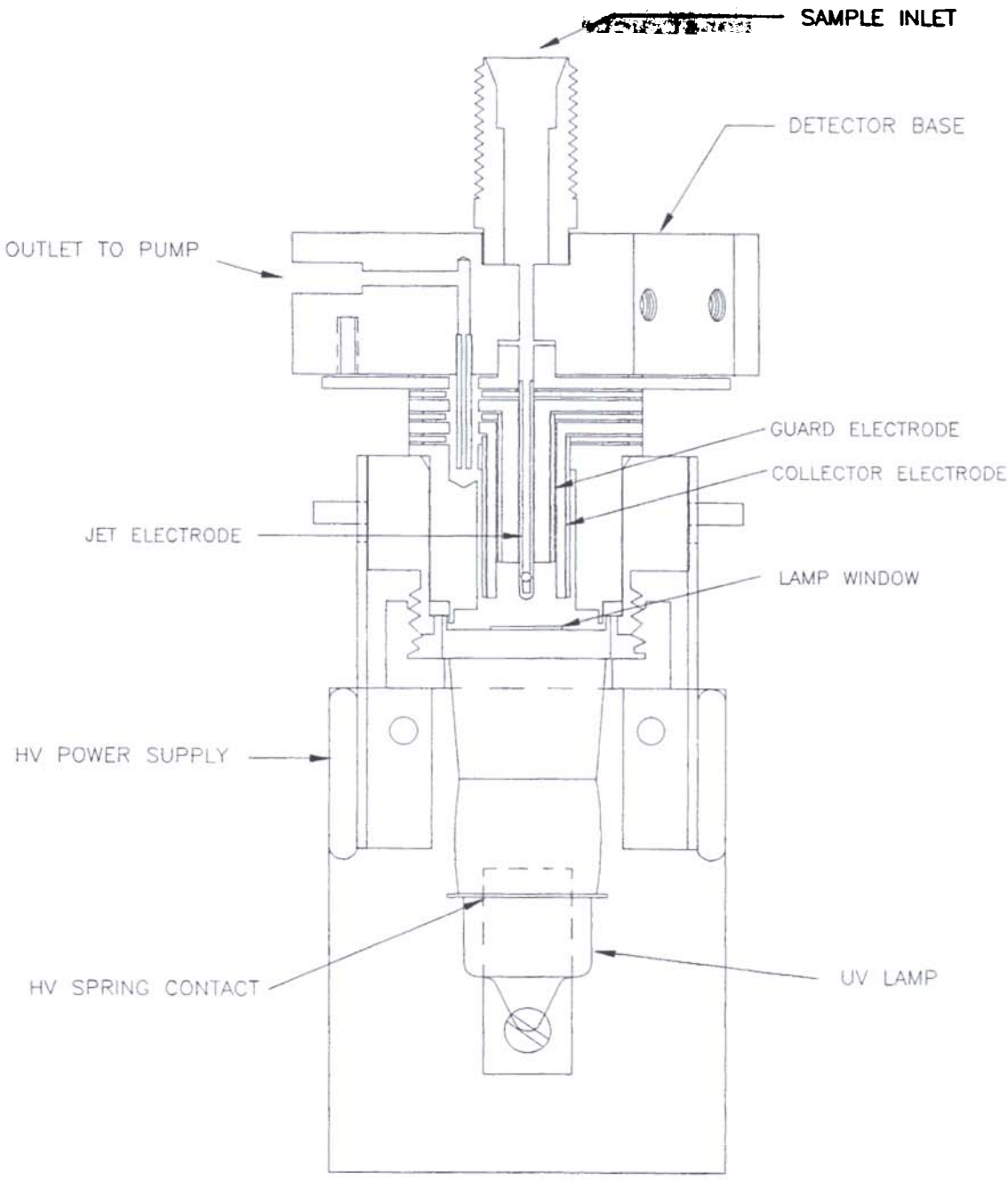


Figure 2.1
Photo Ionization Detector

instrument it is suggested that the user prepare a number of standards of different compounds and measure them relative to isobutylene. Included in this comparison should be several mixtures of compounds such as gasoline, paint thinner, or cleaning solvent, etc. Through this type of study the inequity of the PID response will be better understood making the Model 580 a more effective tool. The use of the instrument is discussed in greater detail in subsequent sections.

2.1.5 PHOTOIONIZATION DETECTOR

The detector is constructed of Teflon and stainless steel to eliminate chemical interaction with the surfaces that are encountered by the sample. To further reduce possible interaction with the surfaces, the flow rate thru the detector is high, 400 - 500 cc/min developing a very dynamic transport of the sample.

Referring to Figure 2.1, the sample is drawn into the ionization chamber through the jet electrode where the UV radiation from the lamp ionizes the sample. A bias voltage of several hundred volts is applied to the jet to aid in the collection of ions. As a result of the ionization process and the impingement of the UV energy from the lamp on the sample, positively charged ions and free electrons are produced. The jet is negative relative to the collector where the electrons are collected.

Between the jet and the collector, separated on both sides by Teflon, is the guard electrode. Its function is to eliminate surface currents which could flow between the two active electrodes. When the Teflon surfaces become dirty during use, there can be the development of a conduction path on the Teflon, which increases in high humidity situations. The guard electrode eliminates this path. The collector electrode is connected to the electrometer which measures the ion current produced during the ionization process. The sample is moved through the detector by an external pump which is on the exit of the detector.

2.2 PROGRAM OPERATION

2.2.1 INTRODUCTION

The 580B has seven switches located just below the display. They are labeled:

ON/OFF MODE/STORE RESET LIGHT +/INC -/CRSR SPKR

The ON/OFF switch toggles the lamp and pump power between on and off. The MODE/STORE, RESET, +/INC, -/CRSR and SPKR switches all have various meanings (including none at all) depending upon the mode. The SPKR switch normally is used to toggle the instrument speaker between on and off. Pressing the MODE/STORE switch will cause the 580B to return to the Run mode, except when the 580B is already in this mode. In which case it will cause the 580B to enter the Log mode.

The LIGHT switch is used to illuminate the display.

The 580B has several modes. Some of the modes may have sub modes. The modes and sub modes are tabulated below.

Run mode
    Concentration meter normal
    Max hold
Log mode
Parameter mode
    Calibration mode
Access mode
Clock mode
Communication mode

The following sections will describe each mode and how to get to them and through them. It is strongly suggested that this section be carefully read and that the 580B be used along with the manual in order to re-enforce the manual.

2.2.2 POWER FOR LAMP AND PUMP

When the 580B is first turned on (see section 1.3) the display will indicate that the lamp is not lit. Pressing the ON/OFF switch will tell the microprocessor to turn on the lamp and the pump. The microprocessor will send power to the lamp and pump and then "look" to see if the lamp is actually lit. If it did not light then the microprocessor will try again.

If after fourteen seconds the lamp still will not light, then the microprocessor will indicate a lamp out condition.

In the event that the microprocessor is unable to light the lamp, check the seating of the lamp (see section 3.1). If the problem persists, call service.

Once the lamp is lit, the display will show the PPM (parts per million) on the bottom line. The top line will either be a bar graph or the maximum reading (see section 2.2.3).

To turn the lamp and pump off simply press the ON/OFF switch.

2.2.3 RUN MODES

The 580B has two run modes, Max Hold and Concentration meter. The run mode is selected in the Parameters section (see Section 2.4). In the concentration meter mode the top line of the display will be a bar graph. The bar graph is a logarithmic bar graph over the range of 0 to 2000 PPM. The bar graph is intended as a rough visual indication of the current PPM. The bottom line will indicate the exact PPM.

In the Max Hold mode the top line of the display will indicate the maximum reading. The bottom line of the display will indicate the current PPM. Whenever a new maximum is seen, the top line will be updated. The Max Hold reading may be reset by pressing the RESET switch while in the run mode.

2.2.4 LOG MODE

The ability to "log" data is one of the 580B's greatest

features. Readings may be stored for later analysis. Each reading will have a date and time as well as a location code associated with it. Up to over 700 readings may be stored. Logged data may even be sent to a printer or computer via an RS-232 serial communication port (see section 2.7).

The Log mode is entered from the Run mode by pressing the MODE/STORE switch. When this switch is pressed from the Run mode the display will show:

LOG THIS VALUE?

on the top line and either PPM or MAX PPM on the bottom line depending upon which run mode the 580B is currently in. By pressing the +/INC switch the display will then show:

LOC. CODE 000001

on the top line (the actual location code may not be 000001). The location code may now be entered. By pressing the +/INC switch the number above the cursor may be incremented. By pressing the -/CRSR switch the cursor may be moved to the next digit. The 580B automatically increments the location code each time a data point is logged.

Once the desired location code has been entered, pressing the MODE/STORE switch will "log" the data point. This means that the reading displayed on the bottom line, along with the location code, the current date and the current time will be stored into the 580B's memory. The 580B will then return to the Run mode.

If for any reason logging is not desired, pressing the RESET switch rather than the MODE/STORE switch will cause the value not to be stored. The 580B will then go back to displaying:

LOG THIS VALUE?

Pressing the mode switch will now return the 580B to the Run mode.

It is possible, when attempting to log a data point, that rather than the display showing "LOC. CODE 000001" it will show "BAR CODE: ." Don't be alarmed. This has happened becouse the location mode selection is not properly set. Section 2.4.3 describes how to set this parameter. Pressing the mode/store switch will cancel the logging operation and return to the run mode. The location mode selection should be changed as described in section 2.4.3.

2.2.4A AUTO LOGGING MODE

The 580B may be instructed to automatically log data according to a predefined time interval. AUTO LOGGING is selected from within the Parameters section (see section 2.4). At the end of the logging interval (settable from 1 second up to 99 minutes and 59 seconds) the current average ppm value will be logged and the logging interval will be restarted.

NOTE: Auto logging is not allowed with the Max Hold mode.

2.2.5 SPEAKER

While the 580B is in the Run mode the speaker may be turned on. The speaker will generate a "clicking" which will increase in speed as the concentration increases. The purpose of the speaker is to give the operator an audible indication of the PPM. The speaker may be turned on or off by pressing the SPKR switch. The speaker rate may also be changed by changing the switches located inside of the instrument. Only one of the four speaker rate switches should be on (in the down position) at any time.

2.2.6 LOW BATTERY

The 580B will display a warning when the battery is low. The warning will be a flashing B in the left hand corner of the bottom line of the display when the 580B is in the Run mode. The 580B should be recharged when the low battery warning is activated.

2.2.7 OVERRANGE

The 580B will display an overrange warning if the concentration goes above 2000 PPM. The top line of the display will show:

OVERRANGE

Once an overrange condition occurs the instrument will "lock out". This means that the overrange warning will continue to be displayed until the instrument is brought to a "clean" area. A clean area is defined to be an area where the concentration of organic vapors is below 20 PPM. The 580B will continue to indicate PPM on the bottom line during an overrange condition.

2.2.8 ALARM

The 580B has an alarm which will sound if the PPM rises above the alarm setting. The alarm setting is entered in the Parameters mode (see section 2.4.3). If the speaker is not activated then the alarm will of course not be heard. Once the PPM drops below the alarm setting the alarm will turn off. The top line of the display will also indicate when there is an alarm condition.

MAIN MENU

By pressing the MODE/STORE switch from the Run mode and then pressing the -/CRSR switch when asked if logging is desired,

580B will display the main menu:

R/COMM -/PARAM
+/ACCESS S/CLOCK

The other four operating modes (Communication, Parameters, Access and Clock) may be entered from the Main menu. The operating mode may always be returned to by pressing the MODE/STORE switch.

2.4 PARAMETERS MODE

All of the 580B operating parameters are entered in the Parameters mode. The 580B is also calibrated from within the Parameters mode.

The Parameters mode may be entered by pressing the -/CRSR switch from the main menu.

There are nine different sections in the Parameters mode.

1. Run mode selection
2. Auto logging selection
3. Location mode selection
4. Average time selection
5. Alarm setting
6. Lamp selection
7. Response factor setting
8. Calibration
9. Free space indication

Pressing the +/INC switch will advance the 580B to the next section. Pressing the -/CRSR will advance the 580B to the previous section. Each section and any of its sub-sections will be described in the following pages. It is important to note that when the 580B is in a sub-section of any of the above sections that the +/INC and -/CRSR switches will have a different meaning. This may seem confusing at first but will become clear after stepping through each section.

2.4.1 RUN MODE SELECTION

There are two Run modes. Concentration meter normal and Max Hold (see Section 2.2.3). The top line of the display will show:

CONC. METER

the bottom line will show:

"RESET" TO CHG

the bottom line will alternate every two seconds with:

MAX HOLD

if the 580B is in the Max Hold mode. Pressing the RESET switch will cause the 580B to show:

MAX HOLD
+ = USE/ - = NO

if the +/INC switch is pressed then the Max Hold mode will be selected. If the -/CRSR switch is pressed then the Concentration meter normal mode will be selected. In either case the 580B will then return to the previous screen.

2.4.2 AUTO LOGGING SELECTION

The 580B can be configured to automatically log data points. The top line of the display will show:

AUTO LOGGING

The bottom line will alternate between "RESET TO CHG." and "ON" or "OFF". Pressing the RESET switch will cause the 580B to show:

AUTO LOGGING
+/ON -/OFF

Pressing the -/CRSR switch will turn auto logging off and return operation to the previous screen. Pressing the +/INC switch will enable auto logging and allow setting of the logging interval. The display will show:

INTERVAL 00:01
"RESET"WHEN DONE

The +/INC switch will increment the number above the cursor and the -/CRSR switch will move the cursor. The logging interval format is MM:SS (where M is minute and S is second). Pressing the RESET switch will return operation to the first auto logging screen.

2.4.3 LOCATION MODE SELECTION

The 580B may be configured to accept a six digit location code which is entered via the keypad. There is an alternate method for entering location codes however UL approval has not yet been obtained for this option. For updated information contact Thermo Environmental Instruments inc.

The display shows the currently selected location mode. For example the display will show:

Loc. code mode
"reset" to chg.

When the 580B is configured to enable operator editing of the location code, pressing the RESET switch causes the 580 to show:

Bar code mode
"reset" to chg.

The 580B is now configured for the alternate location mode (which is not presently available for use in hazardous locations). Pressing the reset switch will cause the 580B to be configured for location code mode.

2.4.4 AVERAGE TIME SELECTION

The 580B can be configured to display the average PPM from once a second up to once every four minutes. The display will show:

AVERAGE = 0:01
"RESET" TO CHG

Pressing the RESET switch will cause the 580B to show:

AVERAGE = 0:01
"RESET"WHEN DONE

The +/INC switch will increment the number above the cursor and the -/CRSR switch will move the cursor. The average time format is M:SS (where M is minutes and S is seconds).

NOTE: The maximum averaging interval is four minutes.

2.4.5 ALARM SETTING

The 580B will display the current alarm setting on the top line of the display. The setting may be changed by simultaneously pressing the RESET switch with either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor.

2.4.6 LAMP SELECTION

The 580B will display:

LAMP

on the top line. The bottom line will alternate every two seconds between:

"RESET" TO CHG

and the currently selected lamp setting and its associated serial number.
i.e.

11.8eV 000000

By pressing the RESET switch, the 580B will display:

+/10eV -/11eV

on the bottom line. Pressing the +/INC switch will select the 10.0 eV lamp. Pressing the -/CRSR switch will select the 11.8eV lamp. In either case the 580B will then allow editing of the lamp serial number. The display will show:

SERIAL # 000000
"RESET"WHEN DONE

The +/INC switch will increment the number above the cursor and the -/CRSR switch will move the cursor. Pressing the RESET switch will return operation to the original lamp screen.
When using a 10.0 eV lamp or a 10.6 eV setting should be selected. When using an 11.8 eV lamp the 11 eV setting should be selected.

2.4.7 RESPONSE FACTOR SETTING

The current Response Factor setting will be displayed on the top line of the display. The Response Factor may be changed by simultaneously pressing the RESET switch with either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor.

The response factor is used to equate the response of one organic vapor with that of the calibration gas. The current reading is always multiplied by the response factor in order to obtain the displayed concentration. A response factor of one will not change the displayed concentration.

2.4.8 CALIBRATION

The 580B will display:

"RESET" TO
CALIBRATE

The calibration mode may be entered by pressing the RESET switch.
The 580B will display:

RESTORE BACKUP
+ = YES

The previous calibration information may be restored by pressing the +/INC switch. The 580B will then return to the previous screen. If the backup is not desired, by pressing the -/INC switch the calibration routine will continue. The display will show:

ZERO GAS
RESET WHEN READY

Once zero gas has been introduced the RESET switch should be pressed. The 580B will then zero the instrument. The 580B will display:

MODEL 580B
ZEROING

Once the 580B has been zeroed the 580B will display:

SPAN PPM = 0000

The Span gas concentration may now be entered by simultaneously pressing the RESET switch and either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor. Once the span gas concentration has been entered the +/INC switch should be pressed.
The 580B will then display:

SPAN GAS
RESET WHEN READY

Once the span gas has been introduced the RESET switch should be pressed. The 580B will then calibrate the instrument. The 580B will display:

MODEL 580B
CALIBRATING

Once the 580B has been calibrated the 580B will go back to the beginning and display:

"RESET" TO
CALIBRATE

If during the zeroing or calibrating of the 580B a steady reading was not seen then the 580B will display:

CAL ERROR
RESET WHEN READY

Pressing the RESET switch will return the 580B to zeroing or calibrating (depending of course on which it came from).

See Section 4.1 for tips on calibrating the 580B.

2.4.9 FREE SPACE INDICATION

This section will give a rough indication of how much room is left for logging data points. The screen will display a bar graph on the top line and the amount of free space on the bottom line. The number indicates the total number of bytes which are available. Each data point takes fifteen bytes. Other bytes may also be needed in order to store other important information. This is why only a rough indication of room may be given.

2.5 ACCESS MODE

The Access mode is entered by pressing the +/INC switch from the main menu. The 580B has four access levels, zero through three. Level zero will only allow the operator to log data points and of course to change access levels (only if the access code is known). Level one will also allow the user to change the user identification number. Level two will allow the user complete access to the Parameters mode, and allow viewing of the date and time. Access level three allows complete access.

The access mode has three sections:

1. Access level
2. User identification number
3. Instrument number

Pressing the +/INC switch will advance the 580B to the next section. Pressing the -/CRSR switch will advance the 580B to the previous section.

TABLE OF ACCESS LEVELS

ACCESS LEVEL	OPERATIONS ALLOWED
0	Change access level Log data
1	All above operations View time and date View communication format Display logged data Change user I.D.
2	All above operations Change operating Parameters Reset logged data
3	All operations available

2.5.1 ACCESS LEVEL

The screen will display:

```
ACCESS LEVEL   3
"RESET" TO CHG
```

By pressing the RESET switch the 580B will display:

```
     KEY    00003
"RESET" WHEN DONE
```

Please note that in both screens the 3 indicates the current access level and may not necessarily be a three.

In order to change the access level the +/INC switch may be pressed to increment the digit above the cursor and the -/CRSR switch may be pressed to move the cursor. The desired access level should be entered in the right most digit. Note that only access levels between zero and three are legal. The remaining four digits are the access code. The access code will be 0000 when the instrument is shipped. The access code should then be entered. Once this is done press the RESET switch. The 580B will then return to the previous screen.

If the access code entered was not the proper access code, or if the access level was not a legal access level then the access level will not be changed.

The last and most important point regarding the access level is how to change the access code. The access code is the four rightmost digits of the instrument number. The instrument number is only viewable (and therefore only changeable) while in access level three.

2.5.2 USER IDENTIFICATION NUMBER

The screen will display:

```
I.D.# 014563977
"RESET" TO CHG
```

By pressing the RESET switch the 580B will display:

```
I.D.# 014563977
"RESET" WHEN DONE
```

The user identification number may be changed by pressing the +/INC switch to increment the digit above the cursor and the -/CRSR switch to move the cursor. The user identification number is a nine digit number (just right for fitting a social security number). Once the user identification number has been entered press the RESET switch and the 580B will return to the previous screen.

2.5.3 INSTRUMENT NUMBER

The screen will display:

```
INSTR #  000000
"RESET" TO CHG
```

By pressing the RESET switch the 580B will display:

```
INSTR #  000000
"RESET" WHEN DONE
```

The instrument number may be changed by pressing the +/INC switch to increment the digit above the cursor and the -/CRSR switch to move the cursor. Once the instrument number has been entered the RESET switch should be pressed. The 580B will then display the previous screen.

When the instrument number is changed it is very important that the last four digits be remembered. These digits are the access code and therefore will need to be known in order to change the access level.

2.6 CLOCK MODE

The Clock mode is entered from the Main menu by pressing the SPKR switch. The screen will display the date and time on the top line. The bottom line will display:

"RESET" TO CHG

By pressing the RESET switch the 580B will display:

"RESET" WHEN DONE

The date and time may be changed by pressing the +/INC switch to increment the number (or in the case of the month the months abbreviation) above the cursor. The -/CRSR switch will move the cursor. Once the proper month has been entered the RESET switch should be pressed. The 580B will return to the previous screen.

The date and time will be maintained even when the instrument is turned off! It is however advisable that the date and time periodically be checked to ensure that it is correct.

2.7 COMMUNICATION MODE

The Communication mode is entered from the main menu by pressing the RESET switch. The Communications mode has four sections.

1. Communicate with printer or computer
2. Display logged data
3. Reset logged data
4. Set communication parameters

Pressing the -/CRSR switch will advance the 580B to the next section.

NOTE: A detailed discussion of communication protocol is given in Appendix A. Further discussion of communication may be found in Section Seven.

2.7.1 COMMUNICATE WITH PRINTER OR COMPUTER

The 580B is capable of communicating with a computer or outputting logged data to a printer. The 580B will display:

COMMUNICATE?

"+" = YES

if the computer format is selected or it will display:

OUTPUT TO PRINTER
"+" = YES

if the printer format is selected. In either case pressing the +/INC switch will cause the 580B to try to establish communication. Pressing the -/CRSR switch instead will cause the 580B to advance to the next section.

2.7.2 DISPLAY LOGGED DATA

If at least one data point has been logged the 580B will display:

DISP. LOG DATA?
"+" = YES

By pressing the +/INC switch the 580B will display the first data point. The date and time which the data point was logged will be displayed on the top line. The bottom line will alternate between the location code and the PPM. Pressing the +/INC switch will advance to the next logged data point. This will continue until there are no more data points at which time the 580B will display:

NO DATA STORED

The MODE/STORE switch may be pressed to return to the Run mode.

2.7.3 RESET LOGGED DATA

The logged data can be erased so that more data points may be logged. The screen will display:

RESET LOG DATA?
"+" = YES

Pressing the +/INC switch will erase all of the logged data points. The 580B will then advance to the next section.

2.7.4 COMMUNICATIONS PARAMETERS

The 580B can be configured to communicate with a printer or a computer. The baud rate may also be set for 9600, 4800, 2400, 1200, 900, 600, 300, or 150 baud. The 580B will display the current communication format (computer or printer) on the top line and the current baud rate on the bottom line. Pressing the RESET switch will cause the 580B to display:

COMPUTER FORMAT
+ = USE - = NO

Pressing the +/INC switch will select the computer format and the 580B will advance to the baud rate screen (see below). Pressing the -/CRSR switch will cause the 580B to display:

PRINTER FORMAT
+ = USE - = NO

Pressing the +/INC switch will select the printer format and the 580B will advance to the baud rate screen (see below). Pressing the -/CRSR switch will cause the 580B to display the previous screen.

The baud rate screen will display the currently selected baud rate on the top line. The bottom line will display:

+ = USE - = NO

Pressing the +/INC switch will cause the displayed baud rate to be selected and the 580B to show the selected format on the top line and the baud rate on the bottom line. Pressing the -/CRSR switch instead will cause the next lowest baud rate to be displayed.

2.8 BATTERY / CHARGER

The model 580B uses a 1.2 amp hour lead acid (gel cell) battery. There is protection circuitry potted directly on top of the battery. The battery is rechargeable with the charger provided with the instrument. The charger is regulated so that there is no danger of "over charging" the battery. It is suggested that the 580B be charged over the weekend (as well as each evening) during periods of heavy usage in order to ensure maximum battery charge.

SECTION III

ROUTINE MAINTENANCE

The routine maintenance of the 580B involves the calibration of the instrument, the cleaning of the lamp window, and the maintaining of charge on the battery. The following pages give instructions for routine maintenance. Figure 3.1 illustrates the detector assembly.

3.1 LAMP INSERTION AND REMOVAL

3.1.1 REMOVAL

NOTE: The 580B must be off while removing the lamp.

In order to remove the lamp the four screws which hold the case top and bottom together must first be loosened. The case bottom should be placed flat on the table and the top placed on its side next to the bottom.

The high voltage power supply is removed next by loosening the thumb screws on each side and then pulling the power supply towards the rear of the instrument (see figure 3.1). The lamp may now be removed by loosening the lamp nut.

3.1.2 INSERTION

Insertion of the lamp is accomplished by performing the above tasks in the reverse order. The lamp should be placed flat against the o-ring and the lamp nut fastened down in order to create a proper seal. The high voltage power supply should then be inserted and the thumb screws fastened down. There are three pins protruding from the high voltage power supply which should fit snugly into connectors located beneath the detector. The lamp spring (mounted in the center of the high voltage power supply) should make contact with the lamp ring.

3.1.3 LAMP CLEANING

On occasion the lamp should be removed for cleaning. Cleaning of the lamp is accomplished by cleaning the lamp surface of the UV lamp. The procedures for cleaning the different lamps are as follows:

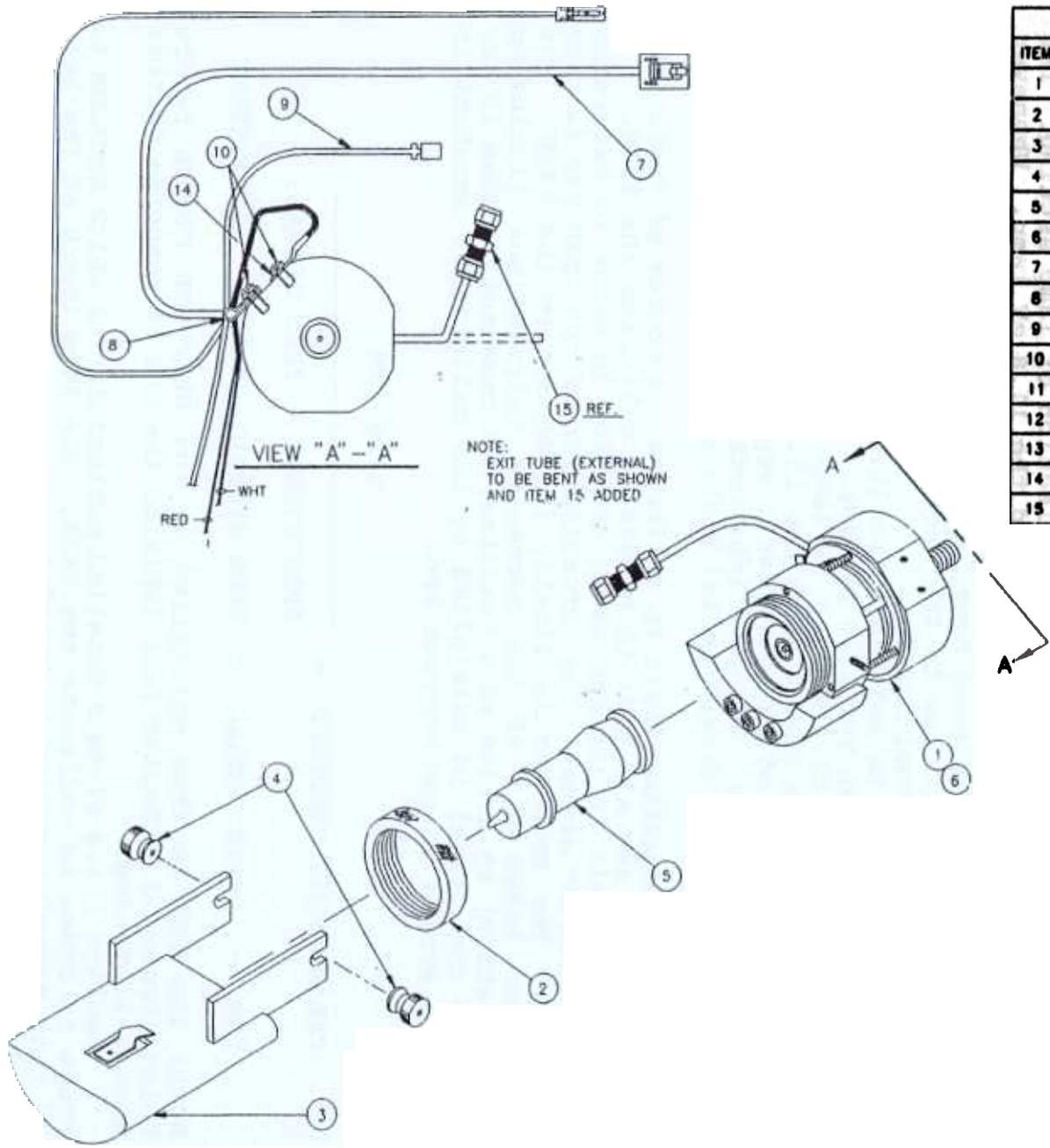
3.1.3.1 LAMP CLEANING METHOD FOR 10.6 eV OR LESS

This is accomplished by using the Aluminum Oxide scouring powder provided with the 580. First place a small amount of Aluminum Oxide scouring powder on the lens of the UV lamp. Next gently scour the lens surface with a soft tissue or cloth. Scour the lens in a rotary type motion.

After scouring the lens surface, gently blow the remaining powder from the lens. Follow this with an alcohol or acetone rinse, and then wipe dry with a soft tissue. The lamp is now able to be inserted into the detector.

3.1.3.2 LAMP CLEANING METHOD FOR 11.7 eV OR MORE

This is accomplished by gently polishing the surface of the window with anhydrous alcohol on a cotton swab, followed by an anhydrous methanol or ethanol rinse, and then wiping dry with a soft tissue. Do not allow the alcohol to remain on the surface as it will leave a film. Stubborn films may require multiple cleanings for complete removal. The lamp is now able to be inserted into the detector.



MATERIAL LIST.			
ITEM	PART NO.	DESCRIPTION	QTY
1	580B-6003	DETECTOR SUB ASSY.	1
2	580S-2010	NUT – LAMP (13567)	1
3	580S-6019	PWR. SUPPLY ASSY. (13560)	1
4	12082	NUT –, KNURLED	2
5	11929	U.V. LAMP 10.0	1
6	580S-6035	DETECTOR WIRING ASSY. (13575)	1
7	580B-6001	BASE HARNESS ASSY.	1
8	4166	STRAIN RELIEF	1
9	580S-6028	SIGNAL CABLE (13568)	1
10	5814	#4-40 X 1/4" BINDER HD. SCREW	2
11			
12			
13	5510	TEFLON TUBING 1/8" 6.5" LG	1
14	5588	#4 INT. TOOTH STAR WASHER	1
15	4417	UNION–2U–316	1

580B-6004

Figure 3.1
Detector Assembly

3.2 CALIBRATION

NOTE: Chapter four should be read before calibrating the 580B in order to gain a better understanding of the concepts behind calibration of the 580B.

The following is a brief discussion of calibration as it relates to different lamps. One of the parameters in the Parameters mode (see Section 2.4) allows selection of lamp setting. The two types of lamps are the 10.0 eV and the 11.8 eV lamp. Whenever a new lamp is used the 580B must be calibrated. This is true even if the new lamp is the same type, e.g., the new and old lamp are both 10.0 eV. This is due to the fact that each lamp will have a slightly different sensitivity.

It is important to note that the 11.8 eV lamp will in general be less sensitive than the 10.0 eV lamp. This is true despite the higher energy level of the 11.8 eV lamp. The 11.8 eV lamp will however "see" certain gases which the 10.0 eV lamp will not. See Table E.1 for a list of common organic vapors and their associated ionization potentials. Any questions regarding the use of the 580B should be directed to Environmental Instruments Company Application Laboratory.

The 580B is quite simple to calibrate. A source of "zero air" and "span gas" are all that is needed to calibrate the 580B.

The zero air is introduced to the 580B in order to determine the "background" signal. The concentration of the span gas is then selected. The span gas is finally introduced to the 580B. The instrument makes all of the necessary calculations (including linearization) to arrive at a "calibration constant." When in the Run mode the signal is multiplied by the calibration constant in order to arrive at the current PPM.

$$\text{CALIBRATION CONSTANT} = \frac{\text{SPAN PPM}}{\text{SPAN SIGNAL} - \text{ZERO SIGNAL}}$$

$$\text{PPM} = (\text{SPAN SIGNAL} \quad \text{ZERO SIGNAL}) \ \text{CALIBRATION CONSTANT}$$

NOTE: The PPM is then multiplied by the RESPONSE FACTOR before being displayed. Chapter four explains the use of response factors when calibrating.

Section 2.4.8 gives a detailed explanation of which buttons to press in order to calibrate the 580B. The flow chart at the back of this manual may also be helpful.

CHARGE

When there is a flashing "B" in the lower left corner of the display (while in the run mode) the battery is low. The battery is recharged by plugging the charger into the RUN/CHARGE plug at the rear of the 580B. The instrument runs while it is charging.

SECTION IV

CALIBRATION

GENERAL

The Model 580B Organic Vapor Meter is indeed a quantitative instrument and can certainly be used as such. It makes use of the Photoionization Detection System using a lamp with an ionization energy of 10.0 eV which is standard in the Model 580B. Almost all organic materials will be ionized at this energy level. There are some organic materials, such as a few of the freons, methane, ethane and propane that are not ionized and thus will not be detected. The ionization potentials for the various organic materials will simply tell whether the material will be detected by the Photoionization Detector. It does not give any clue as to the sensitivity of the detector for that particular material. Certainly, different organic vapors will have different sensitivities. It is important to understand that the Model 580B does indeed sense most organic vapors and that its response to these different organic vapors will be different.

In this section of the manual, the aspects of calibrating the Model 580B for various vapors will be discussed. In the following section discussing applications, various ways of using the features of the Model 580B will be explained along with the various methods for calibration of the 580B. There will also be applications of the Model 580B in specific instances where the organic vapors or the mixtures of organic vapors are completely unknown. The 580B can be an extremely useful tool, even in areas such as those.

FACTORY CALIBRATION TEST OF THE MODEL 580B

The Model 580B has been tested for calibration and linearity tested at the factory. The particular gas chosen for this calibration is isobutylene. The Model 580B has good response for isobutylene. Isobutylene standards prepared in air are relatively stable with time, undergoing no serious adsorption or reaction problems. The test information is included in the instrument packet. In addition to the above test a benzene standard is also run. It is important to note that the instrument was not calibrated. It was tested for calibration. Therefore, it should be calibrated by the operator before use.

4.3 METHODS OF GENERATING CONCENTRATIONS OF VARIOUS MATERIALS IN AIR

This section is not intended to be all inclusive as far as the preparation of gas and vapor standards in air are concerned. Only those methods that have been found most practical for the calibration of the 580B are discussed here. There are basically two types of standards, cylinder and bag.

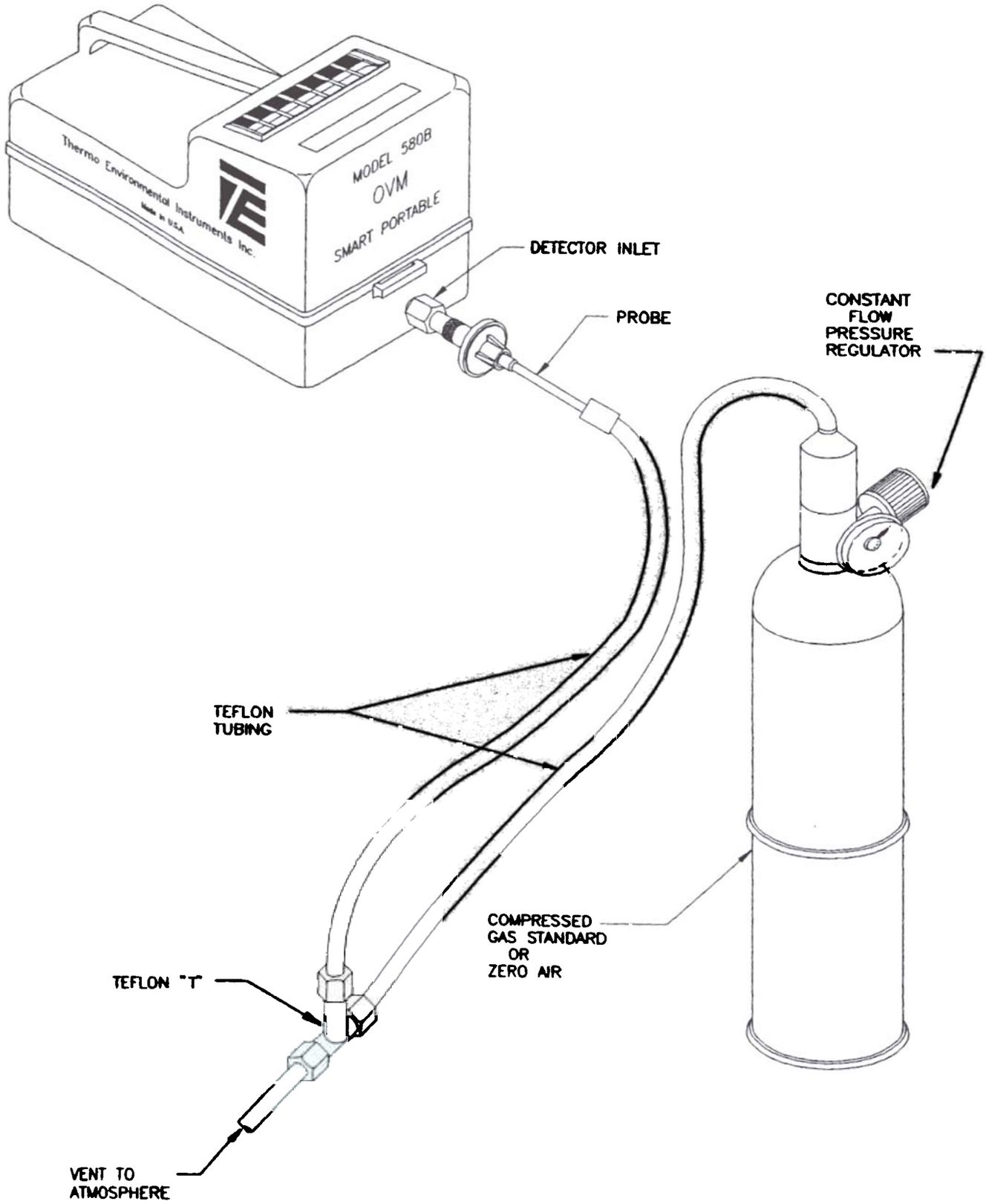


Figure 4.1
Cylinder Calibration

4.3.1 CYLINDER STANDARDS

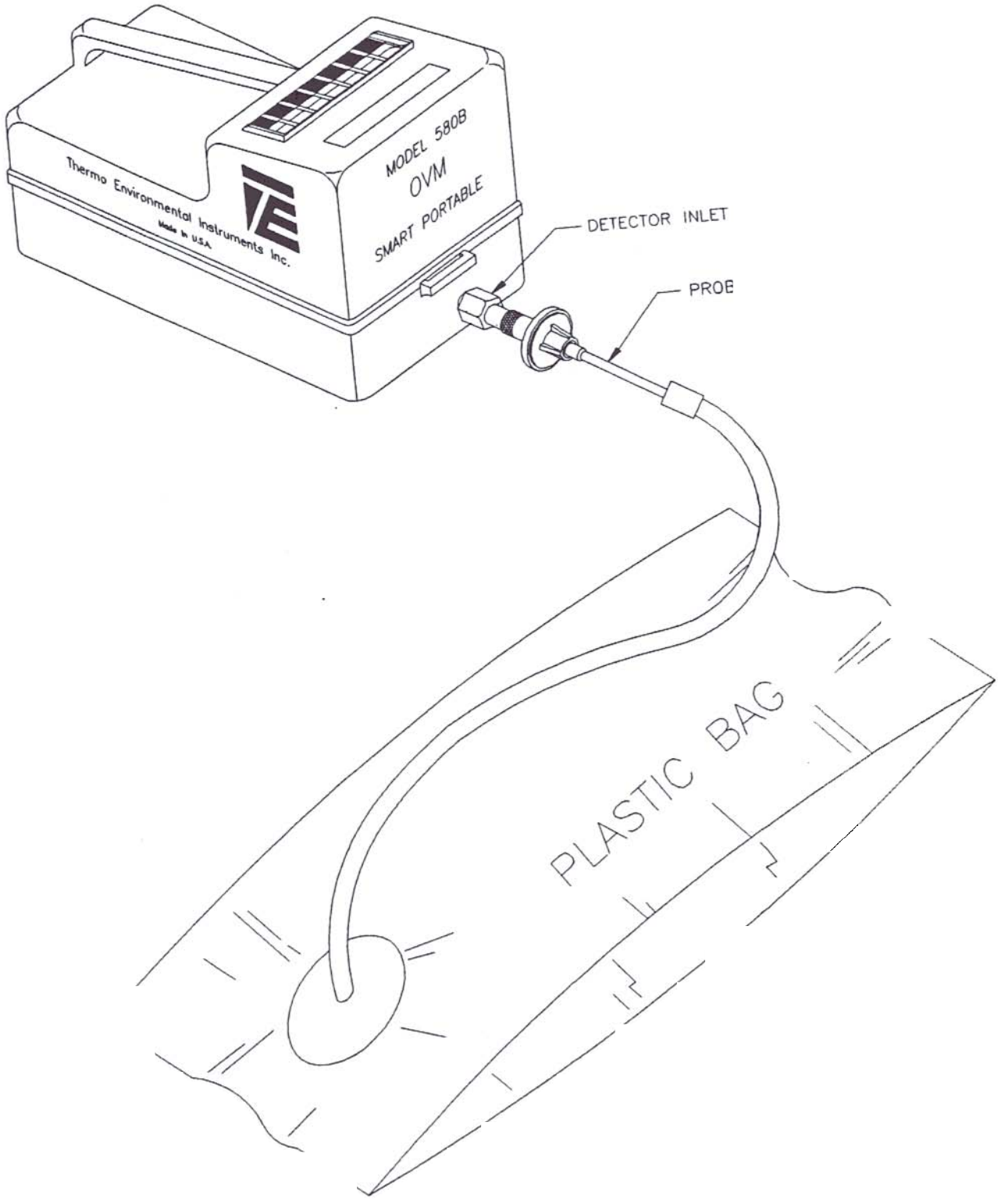
Certainly commercially available standard cylinders of gaseous materials in air offer the most convenient method of calibration. However, these are static standards. Standards prepared in this fashion in air for vapors of various organic liquids often show concentration reduction with time due to adsorption problems. In general, gases when mixed with air will maintain their concentrations with time since adsorption is generally not a problem.

However, some gases are sufficiently reactive that chemical reaction of the gas will cause a reduction of it in air. These precautions must be observed when using commercially prepared standards for calibration of the Model 580B. It is for this reason that isobutylene in air was chosen as a reference standard for factory calibration. TEI offers a cylinder standard which includes both zero and isobutylene standards. A constant flow pressure regulator sets the flow needed for calibration of the 580B. Figure 4.1 illustrates the physical calibration procedure. The inlet to the 580B is connected to the "T" as shown. It is important that this connection is tangent to the gas flow. The "T" is connected to the regulator on the standard cylinder. It is important that a length of tubing is attached to the "T" location. This prevents diffusion of ambient air into sample line. The regulator and tubing assembly will have to be moved between both the zero air and standard cylinder.

4.3.2 BAG STANDARDS (ISOBUTYLENE)

Bag standards can be prepared in a laboratory and in general are reasonable ways of calibrating the Model 580B. However, it is important that these standards be used shortly after their preparation to reduce the significance of any adsorption problems. Static standards prepared for calibration of the Model 580B are best prepared in collapsible plastic bags. This is opposed to a fixed volume container. The sampling rate of the 580B, which is 500 ml/min, requires an appreciable amount of sample. Even one minute's sampling out of a fixed container will remove 500 ml/min from it. This should not significantly reduce the pressure inside the container. Thus, the collapsible bag provides the best means as opposed to a fixed volume. A 5 gallon polyethylene bag is a convenient size to use for the preparation of static standard.

A tube is inserted into the opened end of the bag and the bag opening then sealed around the tube. The tube should have a cutoff valve or some means of closing the volume of the bag. The volume of air introduced into the bag must be measured. This is most conveniently measured by a wet test meter. However, a source of air flowing through a flow meter can be used if the flow can be held constant, then time is a measure of the volume of the air placed into the bag. All air is expelled from the bag by completely collapsing it prior to connection to the source of air.


MODEL 580B
OVM
SMART PORTABLE
Thermo Environmental Instruments Inc.
Made in U.S.A.
DETECTOR INLET
PROBE
PLASTIC BAG

Fi
ag

It can then be connected to a wet test meter or flow meter via a short length of rubber tubing hooked to the plastic tube of the bag. The air flow is started into the bag at a rate of approximately 5L/min. A total of 10 liters is a convenient volume for a 5 gallon bag. This would mean approximately 2 minutes for filling the bag. Figure 4.2 illustrates the physical configuration needed to develop bag standards.

For gaseous samples, the trace organic will be added via a glass hypodermic syringe. The 1 cc Tuberculin syringe is a convenient size. For an isobutylene standard, the 1 cc syringe is flushed with pure isobutylene and then filled to the 1cc mark. While the air is flowing into the plastic bag, the short piece of rubber tubing is pierced by the needle from the 1 cc syringe and the plunger slowly depressed such that the 1 cc of isobutylene is added to the air flowing into the plastic bag. When 10 liters of air have been added to the plastic bag, the flow is immediately stopped and the valve on the tube or the closing clamp is applied to contain the air and isobutylene within the plastic bag. It is best at this stage of the procedure not to rely solely on the diffusion of isobutylene to form a uniform mixture inside the plastic bag. Slight kneading of the plastic bag will hasten the mixing of the isobutylene in air. The plastic tube from the bag is then connected to the probe on the Model 580B via a short length of rubber tubing and the valve on the plastic tube immediately opened. The Model 580B withdraws a sample from the bag at the sampling rate of 500 ml/min. Thus, 10 liters of sample in the bag will provide approximately 20 minutes. Certainly the calibration of the 580B can be accomplished in a shorter period of time. The concentration of isobutylene in ppm by volume will be equal to the sample size, which was 1 cc, divided by the volume of the bag in liters, which would be 10 liters, times 1000. In this particular instance, the concentration would be:

$$\text{Conc (ppm by Vol)} = \frac{\text{1cc Isobutylene x 1000}}{\text{10 L Air}} = 100 \text{ ppm}$$

4.3.3 BAG STANDARDS (ORGANICS)

On occasion there is the need to prepare standards other than the normal calibration standard. As mentioned previously, isobutylene was chosen as a standard because of its stability. If other standards are to be used, it is best to develop a relation of the other standard to a standard of known stability like isobutylene. If this procedure is followed, a response factor can be developed by comparing the other organic standard to isobutylene this technique will be discussed in a later section. The following is a suggested technique for preparing other standards.

For organic materials, which are normally liquids at room temperature, the procedure is essentially the same except that an extremely small liquid sample is injected into the flowing air stream rather than the gas sample. This technique works well

only for relatively volatile organic materials. The flowing air stream must vaporize all of the material or the calculation will be off. If the material is not rapidly volatile in that flowing air stream, the liquid should be injected through the surface of the plastic bag. Immediately after withdrawing the needle, the hole in the plastic bag should be covered with a piece of plastic tape.

Again, significant kneading of the bag will hasten the evaporation of the sample and mixing of the vapor into the air to provide homogeneous samples. The introduction of this sample into the 580B is the same as before. The calculation of the concentration of the vapor in air is a two-step procedure whereby the small volume of liquid injected into the air stream and into the plastic bag is converted to a volume of vapor. This volume of vapor is then used in the same manner as the volume of gas in the case of isobutylene. The following equations apply:

$$\text{Volume Vapor (uL)} = \frac{\text{Liquid Volume (ul)} \times \text{Liquid Density} \times 24.45}{\text{Molecular Weight}}$$

The above equation gives the vapor volume at atmospheric pressure (760 torr) and 25° C (77F).

Then:

$$\text{Concentration (ppm by Volume)} = \frac{\text{Vapor Volume (ul} \times 1000}{\text{Air Volume (liters)}}$$

The following is a sample calculation for benzene:

Liquid Volume = 2 ul

Benzene Density = 0.879 g/cc

Molecular Weight Benzene = 78.1

Air Volume = 10 Liters

$$\text{Vapor Volume} = \frac{2 \times 0.879 \times 24.45 = 0.55 \text{ ul}}{78.1} \quad \text{Benzene Vapor}$$

$$\text{Conc} = \frac{0.55 \times 1000}{10} = 55 \text{ ppm (vol}$$

The syringe used for the measurement of liquids in this particular instance is a small volume-type such as those manufactured by the Hamilton Company. A convenient size syringe is the 10 microliter volume.

4.4 580B CALIBRATION

The following procedure is applicable for both Cylinder and Bag Standards. The sequence requires both Zero gas and Span gas to be used. Span gas can be either contained as a cylinder or bag, in either case the exact concentration used must be known. This concentration will be entered to the 580 when the program provides its entry. With respect to Zero gas, there are several choices. Obviously a certified zero air standard in a cylinder presents no problem. Another choice would be to build a zero air standard in a bag. This can be simply accomplished with the set-up in Figures 4.1 and 4.2 using a charcoal scrubber to remove all the hydrocarbons present in the air. Charcoal does not absorb Methane; this does not cause a problem because the PID does not respond to it. Another approach which could be used in an emergency is to use room air unscrubbed.

This is acceptable if you know that there are no hydrocarbons present or they are exceptionally low in concentration. However, it is not recommended as a standard practice. The physical set up for cylinder calibration is illustrated in Figure 4.1; bag calibration in Figure 4.2.

4.4.1 CALIBRATION ROUTINE

(A) Set-up calibration assembly with zero air cylinder or bag as described in Figures 4.1 and 4.2.
(B) Model 580B set-up and zero calibration.

1. Power-up instrument using power plug.
2. Depress ON/OFF Key to ignite lamp and initiate sample pump.
3. Depress MODE/STORE Key.
4. Depress-/CRSR Key in response to LOG THIS VALUE? Prompt.
5. Depress-/CRSR Key to select Parameters Mode from the Main Menu.
6. Depress +/INC Key to advance thru the Run Mode selection parameter prompt.
7. Depress +/INC Key to advance thru the Auto Logging Mode selection parameter prompt.
8. Depress +/INC Key to advance thru the Average Time selection parameter prompt.
9. Depress +/INC Key to advance thru the Alarm Setting parameter prompt.
10. Depress +/INC Key to advance thru Lamp Selection parameter prompt.
11. Depress +/INC Key to advance thru Response Factor Setting parameter prompt.
12. Depress RESET Key to initiate calibration sequence.
13. Depress-/CRSR Key to decline restoration of the backup calibration.
14. Connect outlet of calibration tubing assembly to the Model 580B Detector Inlet as illustrated in Figure 4.2.
15. Introduce Zero Air to Model 580B by opening flow regulator.

16. Depress RESET Key to "Zero" Model 580B.
17. Close Flow Regulator.

C) Span Calibration - assuming that the Span gas has a concentration of 250 ppm isobutylene the following procedure is followed:

18. Simultaneously Depress RESET and -/CRSR Keys to activate the movable cursor.
19. Repeat Step 18 until the cursor is at the ones place.
20. Simultaneously Depress RESET and +/INC Keys to increment the ones place value.
21. Repeat Step 20 until the ones place value reads 0.
22. Repeat Step 18 to move cursor to the tens place.
23. Repeat Step 20 until the tens place value reads 5.
24. Repeat Step 18 to move the cursor to the hundreds place.
25. Repeat Step 20 until the hundreds place value reads 2.
26. Repeat Step 18 to move the cursor to the thousands place.
27. Repeat Step 20 until the thousands place value reads 0.
28. The LCD should now read:

```
SPAN PPM = 0250
"+" TO CONTINUE
```

29. Depress +/INC to accept the span conc. value.
30. Connect isobutylene cylinder (250 ppm) to calibration tubing assembly.
31. Connect outlet of calibration tubing assembly to the Model 580B Detector Inlet.
32. Introduce isobutylene standard to Model 580B by opening flow regulator.
33. Reset key to "CALIBRATE" Model 580B.
34. Close Flow Regulator.
35. Depress +/INC. Key in response to "RESET" TO CALIBRATE message.
36. Depress MODE/STORE to return to the Run Mode.

The instrument has been calibrated and is ready to make measurements.

4.5 DETERMINATION OF RESPONSE FACTORS

As mentioned above, the Model 580 can be calibrated with isobutylene but be set to read correctly, the concentration of another substance. This is done by using the Response Factor that is set in the parameter routine. The default for the response factor is 1.0. The Response Factor is the number that is multiplied by the measured concentration to obtain the correct concentration of the measured component. If the chemical to be measured is less sensitive on a PID than the standard, (usually isobutylene) then the Response Factor is greater than 1.0. If it is more sensitive than the standard then the Response Factor is less than 1.0.

The reason for a Response Factor is practicality. If it is

know that the sample to be measured contains only benzene and therefore the user would like to read benzene concentration directly, there are two approaches. The user could make a bag standard daily of benzene vapor in air and calibrate the 580 directly. Or the Response Factor could be used. In the latter case a bag with benzene is made only once for comparison to a cylinder of a stable standard (such as isobutylene). Then daily, the Model 580 is calibrated with the cylinder standard, a simple operation compared to the work of preparing a bag standard.

As an example, if the bag containing 55 ppm benzene in air as prepared above were measured in a 580 calibrated against isobutylene, the concentration might have been read as 91 ppm. thus the 580 is more sensitive for benzene than for isobutylene.

The Response Factor can now be calculated as:

$$\text{Response Factor (RF)} = \frac{\text{Factor STD Concentration}}{\text{580 Reading of Factor STD}}$$

$$RF = 55/91 = 0.604$$

When 0.60 is entered into the 580 as the Response Factor, the 580 will read 55 ppm for the bag.

Now the 580 need only be calibrated using an isobutylene standard and a Response Factor of 0.60 to correctly respond to benzene.

SECTION V

APPLICATIONS

5.1 GENERAL

This section discusses six applications which were done on the old model 580. These applications are discussed as they relate to the model 580B. The following applications of the Model 580B are given to show some different uses and means of calibration of the Model 580B in various practical applications. It is certainly not intended to be an exhaustive list of the uses of the Model 580B. In each situation, the stress is placed on the means of calibration and the interpretation of the readout of the Model 580B. Since the Photoionization Detector responds to virtually all organic materials and since its response varies for the different organic materials, questions can certainly arise as to just how the numbers presented on the digital display relate to anything meaningful. These applications will hopefully illustrate several ways in which these numbers can be quantitative and also illustrate uses of the 580B where accurate quantitation may be impossible.

5.2 VINYL CHLORIDE MONOMER IN REACTION VESSELS.

This particular application involved measuring the vinyl chloride content in vinyl chloride polymerization vessels following the polymerization reaction and the removal of the polymer slurry. Any residual vinyl chloride left in the reaction vessel has to be flushed and scrubbed prior to the opening of the vessel. The vinyl chloride content must be below a certain prescribed level prior to this opening. The reaction vessel is flushed with nitrogen to remove the vinyl chloride from the vessel and purge it through the filter media which remove the vinyl chloride from the nitrogen stream for recovery. During this particular operation, it is known that vinyl chloride monomer comprises significantly more than 90% of the entire organic material. In this instance, if the Model 580B is calibrated for vinyl chloride measurement, indeed the readout will be virtually the true vinyl chloride concentration inside the reactor vessel. The nitrogen exit stream prior to the vinyl chloride recovery was the point used for the analysis.

Since the plant was a considerable distance from the laboratory and since the study would require a significant period of time encompassing several weeks, it was decided to calibrate the Model 580B with the isobutylene reference standard and determine a response factor setting for a vinyl chloride standard in the laboratory.

With the response factor set at 1.0, the instrument was calibrated with isobutylene. The Model 580B was then presented with a known concentration of vinyl chloride monomer in nitrogen. The response factor for the vinyl chloride was then set in order for the Model 580B to read the correct concentration of vinyl chloride in the nitrogen. Static

standards of vinyl chloride are very definitely not stable with time due to the reaction of the vinyl chloride with itself. Thus, standards need to be prepared fresh each time vinyl chloride is to be used to calibrate an instrument. Since bag preparation, which was the technique used for this laboratory calibration of the 580B, would have been inprac tical at the plant; the use of a stable reference standard of isobutylene was chosen. Thus, at the plant site, the Model 580B could be calibrated using the isobutylene standard from a cylinder. This of course, greatly simplified the plant use of the Model 580B. This relationship to a reference standard reduces the time and equipment required at the plant such that the survey of all of the reactor vessels was completed in a short period of time with the items established for the nitrogen flush of the reactor vessels prior to opening the reactor vessels.

It is important to note that when the response factor setting was determined in the laboratory, nitrogen was used as the matrix for the bag preparation of the vinyl chloride standard. If air were used a different setting (higher) would be obtained. Since the sample was in a nitrogen matrix so should be the standard. Note also it is not necessary to have the isobutylene standard in nitrogen. In addition to correcting for differences in response between isobutylene and vinyl chloride, the response factor setting can also adjust for the different readings in nitrogen and air.

5.3 MONITORING ISOLATED PLANT AREAS FOR TOLUENE AND METHYL ISOBUTYL KETONE.

Two areas of an extensive plant operation were required to be monitored for the levels of methyl isobutyl ketone and toluene. Both of these areas were relatively isolated. In one area, methyl isobutyl ketone was the only solvent to which the atmosphere was exposed other than the potential leaks that might occur in process equipment in that same area. There were no other known solvents in use in that area and the ventilating system in effect isolated this area from other areas in the plant. In the second area, toluene had just very recently been substituted as a solvent in place of benzene due to the lower TLV for benzene. Average workplace levels were therefore needed for the toluene concentration in this work area. Again, toluene was the only solvent in this area and there was no other process equipment in the immediate area for even possible leak problems.

Notice that in both of these areas in the plant, it is certainly known from the processes occurring in that area and its relative isolation from the other areas in the plant, exactly which organic vapors will be by far the predominant vapors in the workplace air. In many instances, by simply knowing the processes involved and the chemicals in use in those processes, the qualitative aspects of the environment can indeed be established without the use of instrumentation. This is one of the most overlooked aspects in establishing what organic vapors are present in the environment. It simply involves determining what are the possible organic vapors that can be present. In general, this narrows it to several and in many cases, a single organic

vapor.

In these cases, the Model 580B can be calibrated specifically for these materials and will provide quantitative data on the levels of these materials in the workplace environment. In this particular instance, even though the laboratory to be used for the calibration of the Model 580B was at the plant site, it was desired to use a single 580B to monitor both work areas sequentially and several times throughout the course of a single day. This was to be done over a period of time to establish the variations of both methyl isobutyl ketone and the toluene in these work areas. In this particular instance, changing the response factor setting can avoid considerable calibration changes, as one moves from determining concentrations of methyl isobutyl ketone to the area where one is measuring the concentrations of the toluene vapor.

For calibration, the Model 580B response factor was set at 1.0 and the instrument spanned properly using a known reference standard of isobutylene. The Model 580B was then presented with a flowing air stream containing toluene vapor as generated in the Thermo Electron Model 360 using a toluene diffusion tube. The response factor was then adjusted so that the readout of the Model 580B corresponded to the toluene concentration in this standard.

The Model 580B was then presented with a flowing air stream containing methyl isobutyl ketone. This also was generated via a diffusion tube in the Model 360 Standards Generator. Once again, the response factor was adjusted so that the digital display gave the correct reading for the concentration for the methyl isobutyl ketone presented to the instrument.

With the instrument then calibrated with the reference isobutylene standard and knowing the proper settings of the response factors for methyl isobutyl ketone and toluene, the Model 580B was then ready for its plant survey. The area containing the toluene was monitored for a period of time with the toluene levels as noted by the 580B being recorded.

The response factor was set for this toluene reading. The instrument was then moved directly to the methyl isobutyl ketone area and the response factor adjusted to read methyl isobutyl ketone. The 580B was then able to read directly the methyl isobutyl ketone concentration in the second area. There was the possibility of leaks in process equipment in this particular area. The area in general was surveyed. If significant changes in the reading of the 580B were observed, the 580B was used as a leak sourcing instrument as described in a later section. In this fashion, it could be determined if some of the varying concentrations in this area were indeed coming from a leak in the process equipment. During the survey of this particular area, no leaks from process equipment were observed, therefore, the readings obtained on the 580B could indeed be considered the methyl isobutyl ketone concentration in this particular area.

Throughout the survey of these two workplaces, the 580B could move back and forth rapidly due to its portability and could be, in effect, recalibrated for each of the two different vapors by the mere setting of the response factor.

5.4 PETROLEUM ETHER VAPORS IN WORKSPACE AIR.

A given workplace was using petroleum ether as a paint solvent and for cleaning purposes. It was desired to quantitate the amount of petroleum ether in the air being recirculated in this particular area. Petroleum ether is a distillation fraction from crude oil. Its boiling point is slightly lower than the boiling point of gasoline. This means that petroleum ether is not a single chemical entity, but a multitude of hydrocarbons in a certain boiling range fraction. Reasonable quantitative data can be obtained here without knowing the exact chemical composition of each hydrocarbon that composes petroleum ether . For this purpose, the Model 580B can be used to measure these vapors. The 580B is initially calibrated with the response factor set at 1.0 using a reference standard of isobutylene.

The 580B is calibrated on isobutylene. Then a bag sample is prepared, as detailed above, for the quantitation of the instrument to measure the petroleum ether. In this particular instance, the petroleum ether is injected into the bag in the same fashion that liquid samples are injected. The calculation, however, has to change slightly because the ppm on a volume basis cannot be calculated without knowing the exact chemical composition of the petroleum ether. However, in a situation such as this, one can still quantitate it on a weight basis of the solvent in air. The equations below show this calculation.

Weight Vapor (mg)=Liquid Volume (uL) x Density g/ml

$$\text{Conc (mg/m3)} = \frac{\text{Weight Vapor (mg x 1000}}{\text{Air Volume liters)}}$$

For Petroleum Ether In This Example:

Liquid Volume = 3 uL

Petroleum Ether Density = 0.66

Air Volume = 10 liters

Vapor Weight = 3 uL x 0.66 g/mL = 1.98 mg

$$\text{Conc} = \frac{1.98 \times 1000}{10} = 198 \text{ mg/m3}$$

This sample in the bag is then presented to the Model 580B and the response factor adjusted so that the digital readout on the front panel provides the proper reading in mg/m3. The setting of the response factor that is needed for this reading is noted. The Model 580B can now be used to monitor reasonably quantitatively the petroleum ether in the workplace environment. Any further calibration of the instrument can be done using the reference standard of isobutylene. This is a reasonably accurate

way of giving quantitative information on the amount of solvent in air even though the results are not reported in ppm on a volume basis.

This technique can be used in general when the solvents are a mixture of materials which in general will probably be petroleum distillation fractions. It would certainly also be used in the case of gasoline vapors in air. Notice from the equations used versus the equations for determining the ppm concentration in bag samples for pure liquids, the only real thing missing is the molecular weight of the material. It may be possible to assume an average molecular weight of the solvent mixture and actually report a ppm by volume basis.

5.5 LEAK SOURCING

In this particular instance, the Model 580B is to be used for determining the presence, or absence of leaks in a chemical process plant. The Model 580B is uniquely adapted to this particular operation due to its light weight. In this particular instance, it is not necessary to accurately attempt to quantitate the readings from the Model 580B. It will be used simply to determine presence of leaks and to locate these leaks.

The Model 580B is simply calibrated against a reference standard of isobutylene as normal. No further calibration is used. It is not necessary to know the particular chemicals flowing in the different pipes or what they are in the various reaction chambers. It is only necessary to know that these materials will have some response on the Photoionization Detector. That is, that their ionization potentials are below the energy of the lamp. The standard probe of the Model 580B, with the 580B fully operational, is then simply moved along the various pipes and reactor vessels in the chemical process.

All seals are traced clear around the seal with the end of the probe. As one approaches a leak, the concentration of the organic materials in the air being sampled by the Model 580B will increase significantly. The point of maximum reading will indicate the point of the leaks. As one moves further away from the leak, the concentration of the organics in air will certainly decrease. In this very rapid fashion, the presence of leaks can be detected and their source fairly accurately pinpointed so that the leak can be repaired.

In many instances, it is not necessarily the workplace hazards of these leaks that is important, but the economics of the chemical process itself. In this instance, as in many instances, the exact composition of the organic materials being measured is really unimportant to the successful use of the 580B in a specific application. Also the exact numbers that are displayed on the digital readout of the 580B are unimportant. It is only relative magnitudes that are important in this instance.

5.6 AFTERBURNER EFFICIENCY

In a particular coating process, the material, after it has been coated, is passed into a dryer where the solvents of the coating are removed. These solvents are then vented into a

stack. To reduce the hydrocarbon emission from this plant, an afterburner had been installed to combust the organic solvents from the coating prior to release to the atmosphere. It is important to determine the efficiency of this afterburner and to follow the efficiency of the afterburner to avoid dumping excess solvent into the atmosphere and, thus, become subject to pollution fines.

The Model 580B is ideally suited to this type of operation. Again, it will be unnecessary to know the exact chemical composition of the coating solvent. The Model 580B is simply standardized against the reference standard isobutylene in the usual fashion.

The Model 580B is then connected to sample the stack gas in the dryer prior to the afterburner, noting the steady state number displayed on the digital panel meter. The 580B is then connected to the exhaust gases from the stack following the afterburner.

Again, the steady state number, as displayed on the Model 580B, is noted.

The reading prior to the burner minus the reading after the burner divided by the reading prior to the burner times 100 gives efficiency of the afterburner in the stack. This number is quite accurate, even though the Model 580B was not calibrated specifically for the solvents or solvent mixture used in this particular coating operation. The individual readings before and after the afterburner may not have the exact quantitative relationship to the actual amount of material, but their ratio will be accurate since basically the same chemical or mixture of chemicals is being measured before and after the afterburner.

5.7 SAMPLE COLLECTION OF UNKNOWN ENVIRONMENTS

The Model 580B can also be used in areas where organics are known to be present, but perhaps the exact composition of the environment is not known. This may be due to several solvents being in the same general workplace or various separate processes occurring in that same workplace, all of which could and possibly are admitting organic vapors. In plant areas such as these, the Model 580B can still be extremely useful.

The 580B is calibrated against a reference standard of isobutylene, as mentioned above. The 580B is then used as a survey tool throughout the entire plant area. The readings are logged, especially changes in these readings. The exact numbers displayed will not, in general, be a quantitative measure of the ppm of the organic vapor since it is impossible to know what organic chemical or mixture of chemicals should be used for the calibration. When high readings are obtained on the Model 580B, an evacuated sample bag can be connected to the rear of the 580B at the sample exhaust port. This bag could be virtually identical to the type of bag used for standards preparation. The Model 580B is sampling the atmosphere at the rate of 500 ml/min. The detection system of Photoionization is a nondestructive system such that the sample that is exiting the Model 580B is indeed the same material that is giving the readings on the 580B. When the 580B is seeing high readings, this is the time the bag is con-

nected to the rear for sample collection. The bag, if the same type is used for sample preparation, can hold approximately 10 liters of air sample; which would permit a sampling time of 20 minutes.

This bag sample can then be closed on removal from the 580B and transported to a laboratory for subsequent analysis to identify the individual chemical compounds present in the sample causing the high readings and to ascertain if the workplace environment is harmful at those high readings.

The use of the Model 580B coupled with the bag collection ensures that the sample that is returned to the laboratory for analysis is a sample containing the desired organic vapors. This is assured because the bag collection is used only when the Model 580B is detecting high levels of organic vapor in the environment. This is an instance of the use of the Model 580B when the type of organic vapors are not known and it is desired to know them. The 580B has a very useful function even in these areas. It should be noted that a charcoal tube could be connected to the rear of the 580B as well as an evacuated plastic bag. The charcoal tube will pass the bulk of the sample, which is air, and adsorb the organic vapors. This charcoal tube can be returned to the lab for subsequent analysis for both a qualitative identification of the materials present as well as a quantitative measure of their levels.

SECTION VI

COLLECTION TECHNIQUES

6.1 GENERAL

As mentioned in the Application Section, it is possible to use the 580B in completely unknown areas as far as the organic vapors present are concerned and still obtain meaningful data. One of the techniques described here is the use of the 580B as a means of collecting the representative samples for further identification in the laboratory regarding the specific organics that may be present in addition to their concentrations.

Two techniques were mentioned in the section under the heading "Sample Collection of Unknown Environments". One of these techniques involves the use of a bag for collection and the other involves the use of charcoal tubes as a means of trapping organic vapors. In this section, each of these techniques will be explored in further depth as to the proper way of using the 580B to collect the samples for subsequent analysis. These collection techniques are quite useful when one is using the Model 580B simply as a survey instrument. When readings on the 580B become quite high in certain areas, it is impossible to determine the exact source of the high readings to perhaps pinpoint the specific organic chemical giving rise to the reading. One may very well want to identify what the chemical or chemical mixture is that is providing the high reading. This will have to be done with instrumentation significantly more sophisticated than the Model 580B; namely, an instrument that can provide specificity as well as qualitative identification. A Gas Chromatograph is such an instrument.

If it is desired to collect some of the air to send to a laboratory for further analysis, one needs to be sure that the proper samples are taken at the proper time. This means simply that one needs to be assured that the sample sent to the laboratory is indeed a sample that has a high concentration of organic vapor present in the sample. The 580B is used to indicate the presence of the high level organic vapors. The sample then is gathered at the exit port of the 580B when the 580B is reading high values. This assures that the sample sent to the laboratory does indeed have the high level vapors present in it. This generally simplifies the sampling technique of the environment and reduces the number of samples and, therefore, the expense needed to accurately identify the organics present and to quantitate them in a laboratory.

Two design features of the Model 580B make this type of operation possible. The first is that the detection system used in the Model 580B is the Photoionization Detector which is basically a nondestructive detector. Thus, the instrument is able to sense the organic vapor using the detector and virtually the same concentration of the same materials exits the detector as entered it. This does make it possible for the collection of the exact sample contributing to the high readings.

The second feature of the 580B that allows this sample collection is that a positive displacement pumping system is used to draw the sample into the Model 580B. It is a very simple procedure then connect to the exit of this positive displacement pump and trap the sample exiting the 580B after it has passed through the detector.

6.2 BAG SAMPLE COLLECTION.

One of the most convenient ways to sample the environmental air is to simply trap the entire air sample in a collection bag. As discussed before, the bags used for the calibration of the Model 580B, as discussed under the Calibration Section, can certainly be used for collection of the air samples. There are several precautions that must be mentioned immediately relative to the use of bag sample collection. When a bag has been filled with air that has organic vapor in the air sample, the organic vapor molecules will absorb onto the inside surface of the bag. This adsorption will begin immediately on introduction of the air into the bag. It will continue to progress with time until the vapor molecules that adsorb onto the wall of the bag are in equilibrium with the vapor molecules in the air. This equilibrium depends very strongly on the bag material and the chemical entity of the vapor itself. The ambient temperature also has some effect.

As mentioned under the Calibration Procedure, when one is preparing a known vapor concentration in a bag, the bag should be analyzed very rapidly after its preparation to ensure proper calibration of the instrument. The technique here is to use the standard prepared in this fashion as soon as possible such that the adsorption that has occurred is an absolute minimum amount. This adsorption becomes a bit more serious problem in using bags for sample collection. The first problem is simply when one is reusing the bag, one has to be sure that the sample contained in the bag previously has been completely desorbed from the wall. This, in general, can be checked by using clean air to fill a bag allowing the bag to set for a short period of time, about 1 hour, and then analyzing the air in the bag. If on using the 580 to analyze this air, it shows measurable organics, then the air in the bag should be dumped and new air introduced and allowed to set for the same period of time. There will be a reduction of organic vapor on the second go-around.

If it is still too high, this procedure is repeated until the bag shows virtually no organic vapor. The bag can be evacuated and reused.

The other problem associated with adsorption and sample collection is that the sample that is collected in the bag must be analyzed as soon as possible after collection if one is going to determine quantitatively the amount of organic vapor in that bag sample. The longer the sample stays in contact with the bag, the greater the adsorption will be of the organic vapors on the surface of the bag and, therefore, the lower the concentration of the organic vapors in the air sample.

If one is interested here in only doing a qualitative analysis of the organic vapors, that is identify what vapors are

present in the air sample, the bag certainly is a convenient way of taking the sample. If one in addition to getting the qualitative analysis desires to quantitate one or more of the specific organic vapors in the sample, the bag sample should be analyzed within an hour of taking this sample. If the bag sample cannot be analyzed this soon, it is recommended that one use the charcoal tube technique explained in the next section.

There are two considerations to be given relative to the size of the bag and, therefore, the size of the sample taken. The first consideration is the amount of sample needed by the laboratory for its analysis. If the analysis is to be done by gas chromatography directly on the air sample, in general only 1 to 5 mL of sample would be required for the analysis. Therefore, this does not become a major consideration here. If, however, other analytical techniques were to be used that would require significantly higher volumes of sample, this should be taken into account.

The other consideration is the sampling time. The Model 580B samples at the rate at which the bag attached to the exit port of the 580B will be filled. If the bag can conveniently hold 10 liters of air, this means that the sampling time can be up to 20 minutes. In general, collection techniques using the Model 580B are not intended to supply a four or eight hour integrated sample. They are used simply to help identify the materials contributing to a high concentration and possibly the analysis of individual toxic organic vapors in that particular air sample. Thus, a 20 minute limitation on sampling time should not be too severe.

Certainly larger bags could be used on the exit of the 580B, allowing up to several hours of sampling time should this be desired. The difficulty then becomes that the bags are quite large and physically become difficult to manipulate. It was recommended back in the Calibration Section that perhaps a 10 liter bag would certainly be the convenient bag for the calibration of the 580B. It would appear to be also a convenient bag for collection of the samples. For this purpose, a bag that has no adsorbed vapors on the interior surface is evacuated and closed to the atmosphere. Several of these bags could be carried in a very small container. When the Model 580B is reading high values, and it is impossible to determine the source of the high values, then a bag can be connected to the exit port of the 580B and immediately opened to accept the sample exiting the 580B. The bag is kept connected to this exit as long as the 580B is giving high readings or until the bag has reached its volume capacity. At this point, the bag is removed from the exit port of the 580B, immediately closed, and returned to the laboratory for analysis.

6.3 COLLECTION USING CHARCOAL TUBES

A technique very common in industrial hygiene-type analysis is to use a small charcoal tube as a collection device. An air sample is pulled through the charcoal tube at a known flow rate for a known period of time. This flow rate and time determine the total volume of air or total sample size. The organic vapors

in the air are adsorbed on the charcoal in the tube. These vapors are then desorbed from the charcoal by adding a known volume of desorbing solvent, usually carbon disulfide. The organics end up in the carbon disulfide. The carbon disulfide is then injected into a gas chromatograph using Flame Ionization Detection. The individual organic vapors can then be identified and quantitated.

The usual charcoal tubes that are used for this type of work contain two sections. One section has approximately 100 milligrams of charcoal and a backup section has 50 milligrams. The backup section is analyzed separately from the main section to determine if there is organic vapor breakthrough in the main section. These particular size tubes have a recommended maximum flow in the neighborhood of 250 to 300 mL/min. The exit of the Model 580 is at 500 mL/min. The most advantageous way of using a smaller charcoal tube would be to split the exit stream and pass it through two parallel charcoal tubes. This would give approximately 250 mL through each tube. For analysis purposes, the charcoal of each tube is removed and combined using double the amount of solvent that would be required for a single tube.

The amount of total air that can be passed through charcoal tubes certainly depends on the concentration of organic vapor in the air. It also depends to some extent on the particular organic vapor. In general, a total sample through the smaller charcoal tube of 10 liters is a reasonably safe number to use. Since the flow is split exiting the 580B using the smaller charcoal tubes, only 250 ml/min is going through the tube. It would take 40 minutes to accumulate 10 liters passing through each of the tubes. There are charcoal tubes available in the marketplace containing 300 milligrams of charcoal in the front section and 150 milligrams of charcoal in the rear section. These tubes have correspondingly larger diameter and can accommodate higher volumetric throughputs. One of these tubes could be hooked to the exit of the 580B without doing the split. Conceivably since it contains 3 times the amount of charcoal, a safe operating total volumetric throughput would be approximately 30 liters. This would be a full hour's operating time on the Model 580B. Again, it must be stressed that the 580B when used in the particular form, is not being used as a personnel sampler to end up with the time weighted average concentration over an eight hour period. The intent here is to identify the high level organics observed on the 580B and to quantitate them following identification to determine the safe working area.

SECTION VII

COMMUNICATION

The 580B provides a serial (as opposed to parallel) communication port. There is also a communication cable provided for easy link up to a serial printer or RS-232 port of a computer. Logged data may be "dumped" (sent through the communication port) to a serial printer. Many of the 580B parameters may be set by a remote computer by using the serial port and the 580B communication software (the software is an option, part number 580A-9014).

Note: The serial port is not to be used in a hazardous location

PRINTER

The 580B can be instructed to send all of its logged data through the serial port to a printer (or a dumb terminal). The 580B printer mode should be selected (see Section 2.7.4).The serial communication cable should then be plugged into the RS-232 port at the rear of the instrument and the other end of the cable plugged into the serial port of a printer. The 580B should finally be instructed to output to the printer (see section 2.7.1).

COMPUTER

The 580B provides capabilities for remote operation. Appendix A includes a detailed technical explanation of the 580B printer and computer interface protocol. The information in this appendix is sufficient for custom software to be developed for interfacing to the 580B. Thermo Environmental however has developed communication software which implements all of the available communication capabilities in a simple "menu driven" format. Remote communication may also be accomplished by using generic communication software package such as CrossTalk. Appendix A will be helpful if this route is taken.

NOTE: Generally, the RS-232 port on an IBM PC (or compatible) is a male connector. Since the communication cable provided with the 580B is also male, a "gender changer" (a DB-25 connector which converts from male to female) is needed.

7.3 COMMUNICATION SOFTWARE (OPTIONAL)

There is communication software available which will run on an IBM PC or compatible. The software provides the capability of obtaining or changing the 580B parameters (alarm setting, response factor, or operating mode to name a few). Logged data may be stored to disk or printed to a parallel printer. Concentrations may be read and displayed on the computer screen. There are a few operations which may not be accomplished remotely (for obvious reasons). The lamp may not be changed remotely. The lamp and pump may not be turned on from the computer either.

NOTE: The communication software will not work unless the 580B is attached via the communication cable.

7.3.1 HOW TO GUIDE FOR COMMUNICATION SOFTWARE

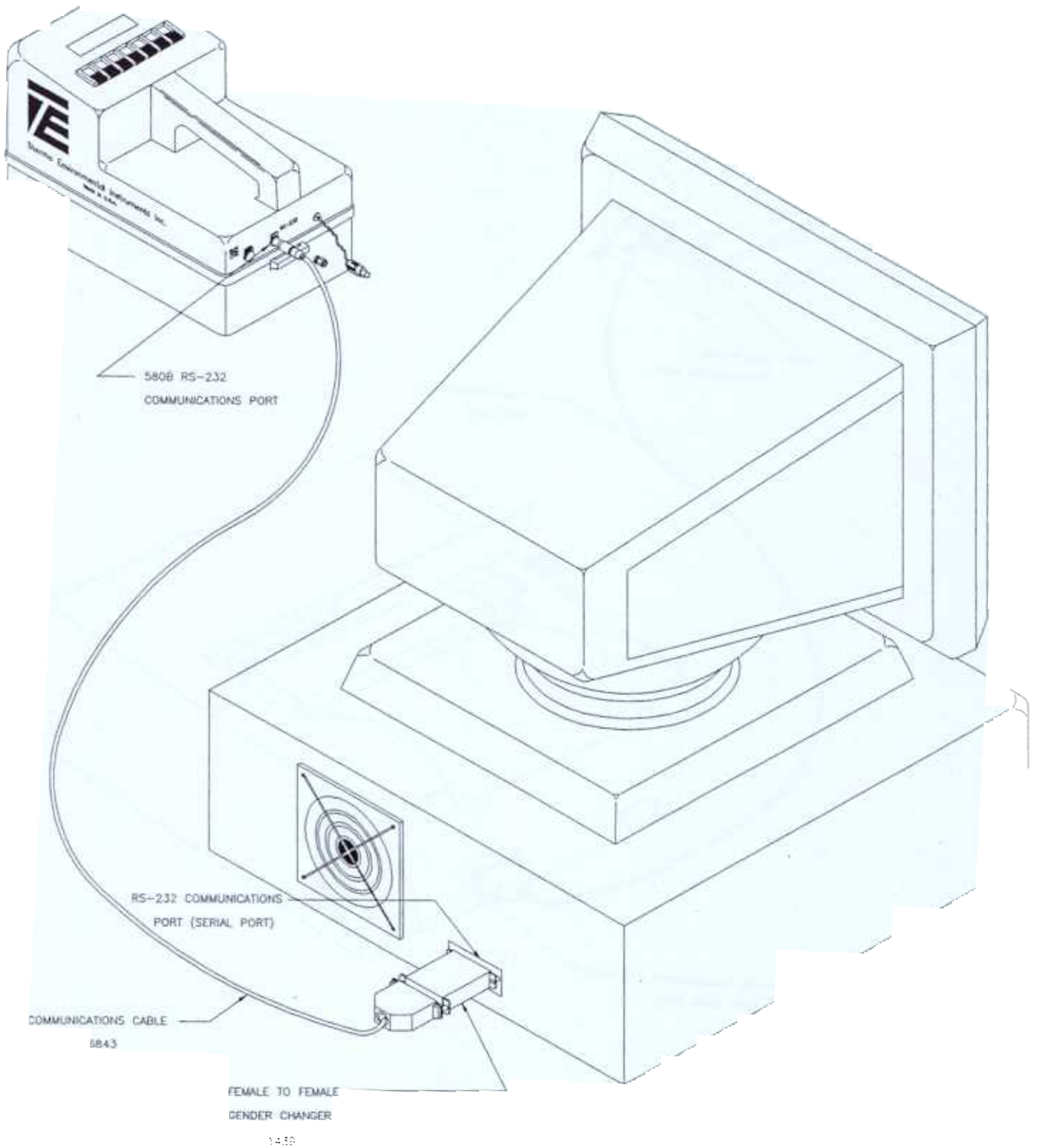
#1. The 580B must be turned on and connected to the computer's RS-232 port. The 580B must be in the computer mode (this is the default setting).

#2. The floppy disk should be inserted into the computer. Type 580B (this software was originally developed for the 580B) and then hit return. The introduction screen will appear.

#3. The software defaults to 2400 baud (as does the 580B). If some other baud rate is desired it must match the setting on the 580B.

#4. After selection of the baud rate press return. The main menu will appear.

NOTE: If the computer's screen goes blank and the main menu does not appear, then there is a problem with the communication link. Check to be sure that the communication cable is plugged into the RS-232 port and that the 580B is on.



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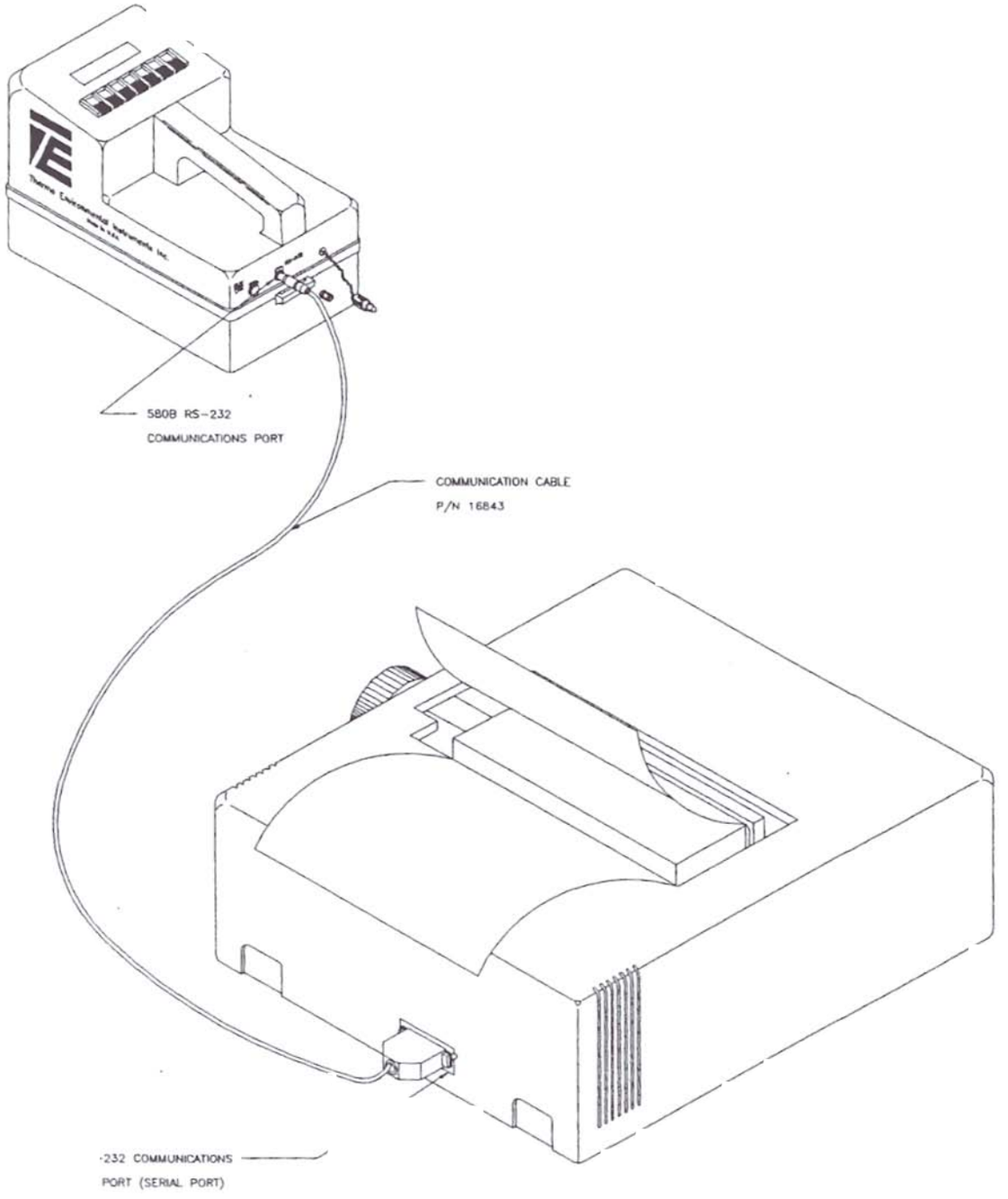


Figu
Compu Communi ion

SECTION VIII

FLOW CHART

There are two flow charts which illustrate the structure of the 580B software. The first is a "Quick Start-up" flow chart. Much of the detail is not included in this flow chart in order to diagram the basic structure of the software. The second flow chart includes extensive detail of each screen and the function of the seven buttons. These flow charts provide an easy method for determining how to get at each of the many facilities provided by the 580B.

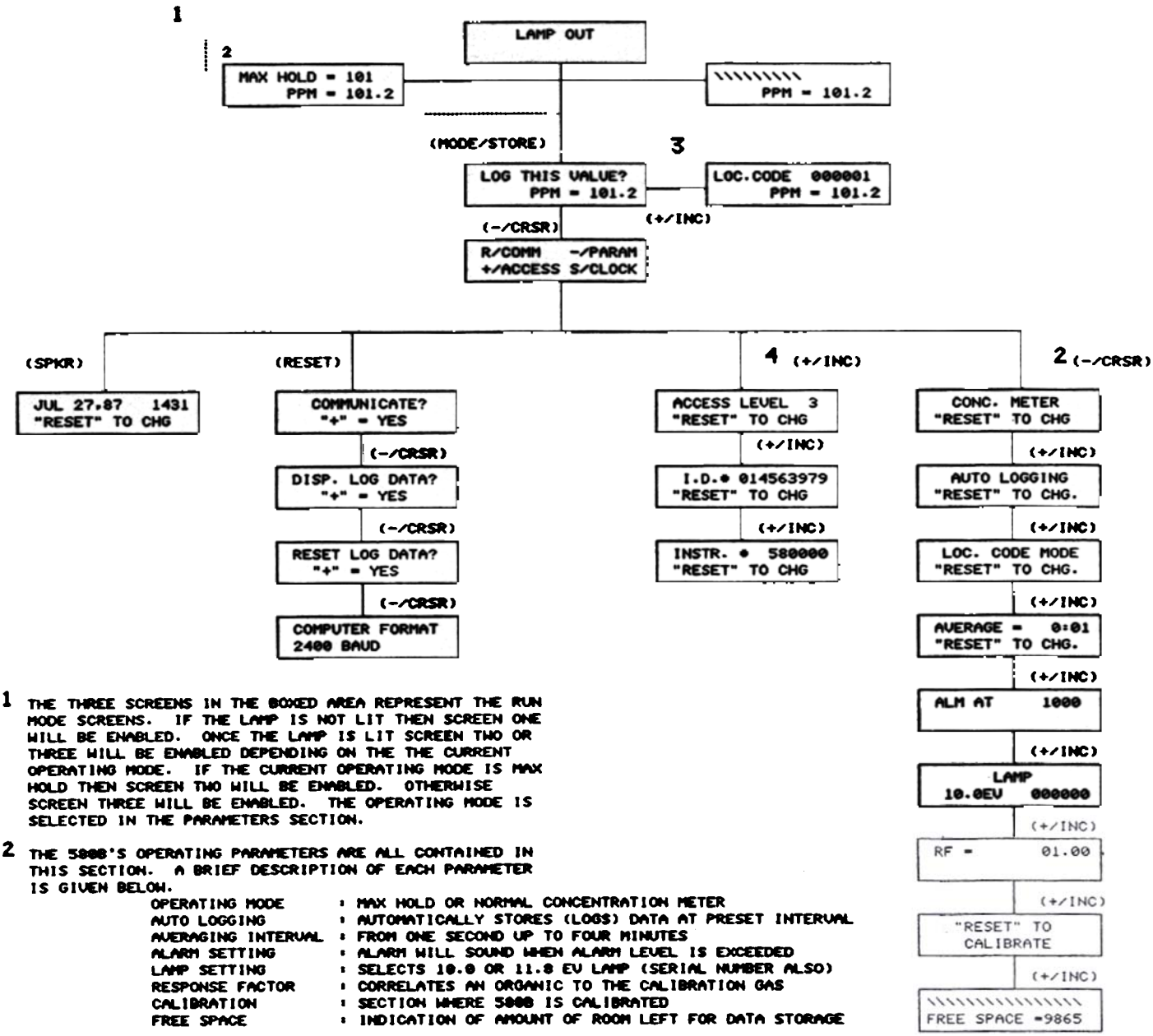
8.1 QUICK START-UP

The Quick Start-up flow chart shows each of the top level screens. The screens are ordered according to the hierarchy of the 580B software. The particular button (which advanced the 580B to the next screen) is shown in parenthesis above each screen. This flow chart does not illustrate any of the associated screens or operations (see the detailed flow chart for more in depth information).

The Quick Start-up flow chart should be fully understood before moving on to the more detailed flow chart. The best way to learn each of the flow charts is to have the 580B with you and to follow along verifying each step.

8.2 DETAILED FLOW CHART

The detailed flow chart illustrates many of the lower level screens as well as the function of buttons. Screens are shown in rectangles with the text written inside. The buttons are shown in ellipses (actually a rather flattened ellipse) with the button identifier written inside. There are a few conventions which need to be explained. The button identifiers have been abbreviated. For example the +/INC button is simply denoted as +. When two buttons need to be pressed simultaneously each identifier is shown with a slash between them. For example RESET/+ indicates that the RESET and +/INC buttons should be pressed together. Arrows indicate the direction of flow from one screen to the next.



1 THE THREE SCREENS IN THE BOXED AREA REPRESENT THE RUN MODE SCREENS. IF THE LAMP IS NOT LIT THEN SCREEN ONE WILL BE ENABLED. ONCE THE LAMP IS LIT SCREEN TWO OR THREE WILL BE ENABLED DEPENDING ON THE THE CURRENT OPERATING MODE. IF THE CURRENT OPERATING MODE IS MAX HOLD THEN SCREEN TWO WILL BE ENABLED. OTHERWISE SCREEN THREE WILL BE ENABLED. THE OPERATING MODE IS SELECTED IN THE PARAMETERS SECTION.

2 THE 5000'S OPERATING PARAMETERS ARE ALL CONTAINED IN THIS SECTION. A BRIEF DESCRIPTION OF EACH PARAMETER IS GIVEN BELOW.

OPERATING MODE	: MAX HOLD OR NORMAL CONCENTRATION METER
AUTO LOGGING	: AUTOMATICALLY STORES (LOGS) DATA AT PRESET INTERVAL
AVERAGING INTERVAL	: FROM ONE SECOND UP TO FOUR MINUTES
ALARM SETTING	: ALARM WILL SOUND WHEN ALARM LEVEL IS EXCEEDED
LAMP SETTING	: SELECTS 10.0 OR 11.8 EV LAMP (SERIAL NUMBER ALSO)
RESPONSE FACTOR	: CORRELATES AN ORGANIC TO THE CALIBRATION GAS
CALIBRATION	: SECTION WHERE 5000 IS CALIBRATED
FREE SPACE	: INDICATION OF AMOUNT OF ROOM LEFT FOR DATA STORAGE

3 THESE ARE THE SCREENS USED FOR LOGGING (STORING) DATA POINTS. AFTER ENTERING THE DESIRED LOCATION CODE, SIMPLY PRESSING THE MODE/STORE SWITCH WILL LOG THE VALUE (AS WELL AS A DATE AND TIME STAMP) AND RETURN OPERATION TO THE RUN MODE.

4 THE ACCESS (SECURITY) LEVEL IS SET IN THIS SECTION. ACCESS LEVEL THREE GIVES THE OPERATOR ACCESS TO ALL OF THE 5000'S FACILITIES. LOWER ACCESS LEVEL ZERO ARE PROGRESSIVELY MORE RESTRICTIVE. ACCESS LEVEL ZERO ONLY ALLOW READING AND LOGGING OF DATA.

Figure 8.1
Quick Flow Chart

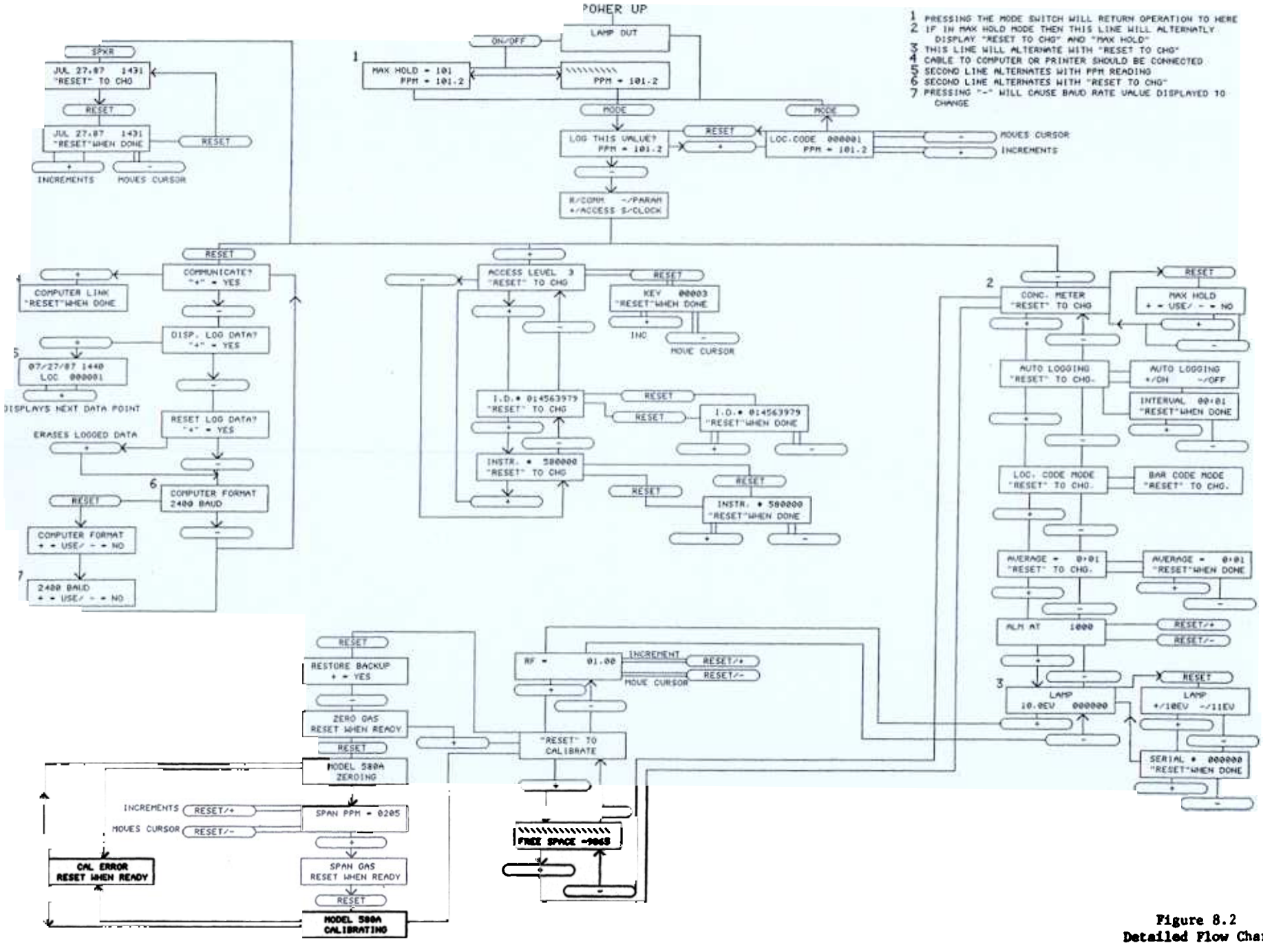


Figure 8.2
Detailed Flow Chart

APPENDIX A

COMMUNICATION

A.1 INTRODUCTION

The 580B is capable of communicating over an RS-232 link with any peripheral device which adheres to the communication protocol outlined in this document. The 580B will communicate with a peripheral device in one of two modes. While in the PRINTER mode the 580B responds to commands from its keypad and can be commanded to send characters out its RS-232 port to a peripheral device. In the COMPUTER mode the 580B responds to commands from a peripheral computer and can be commanded to send and receive data and to perform other tasks. Regardless of the mode used, the 580B sends and expects to receive data in the following format:

No Parity
1 Start Bit
8 Data Bits
1 Stop Bit
Baud Rate = 150, 300, 600, 1200, 2400, 4800 or 9600

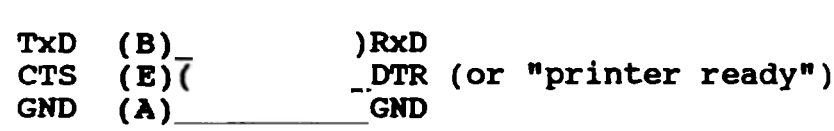
The Baud Rate may be selected from the 580B keypad (see Section 2.7)

A.2 PRINTER MODE INTERFACE

In the PRINTER mode the 580B can be instructed to send its log data out the RS-232 port to a peripheral device such as a printer.

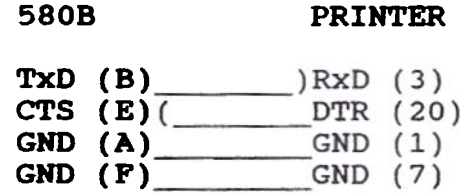
The 580B can be manually instructed from its keypad to output its log data. In this case the 580B sends all the log data points it has acquired thus far. The log data is saved in 580B memory and will NOT be automatically erased upon output. While in the PRINTER mode, the 580B log data file must be erased (reset) from the keypad. An example of a log data output is shown on page A-17. Notice that the 580B also sends header information which includes the following parameters: instrument number, user ID, and mode of operation. The date at the top of the header corresponds to the time when the first log data point was taken with the parameters set as shown in the header. If any of the parameters are changed and then new log data points are acquired, the 580B will send an updated header before it sends the new data points. It is also important to note that every log data point is time stamped to show when it was stored.

HARDWARE INTERFACE, PRINTER MODE



The connections shown above are required before the 580B can successfully send its log data to the peripheral. An example of a cable used for PRINTER MODE communication is shown below:

SAMPLE CABLE, 580B TO HP THINKJET PRINTER



Thinkjet connector = TRW/CINCH DB-25P plug connector

Please remember that the 580B must be placed into the PRINTER mode prior to output of log data to a printer. This is done from the 580B keypad.

HARDWARE HANDSHAKING, PRINTER MODE

The 580B will send log data out the port to a peripheral device as long as its CTS line is asserted (+V). If the peripheral has temporarily fallen behind, and consequently clears its DTR line (-V), the 580B will stop transmitting data. It will resume transmitting as soon as the peripheral reasserts (+V) its DTR line.

A.3 COMPUTER MODE INTERFACE

In the COMPUTER mode the 580B will respond to commands sent by a peripheral computer. The 580B will respond to 3 types of commands; DO, GET, and SET commands.

An example of a DO command is "DO RESET LOG (ret)" in which the 580B is instructed to reset its log which, in effect, clears all previously stored log data points.

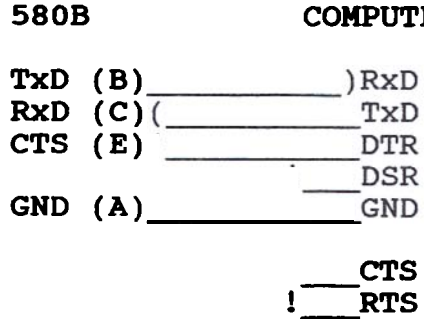
A GET command such as "GET ALARM (ret)" allows the peripheral to change a specified parameter in the 580B to a value provided by the peripheral.

A combination of DO, SET, and GET commands allow the operator at the peripheral to perform a variety of functions needed to prepare a 580B for acquiring data.

The 580B can also be operated under "computer control". For example, the 580B can be commanded to acquire readings at specified intervals and then store the readings in a log data file.

The 580B can subsequently be commanded to send its log data to a printer, a file or the screen of the peripheral computer. This can all be accomplished through commands issued by the peripheral computer. So, effectively the 580B is under "computer control".

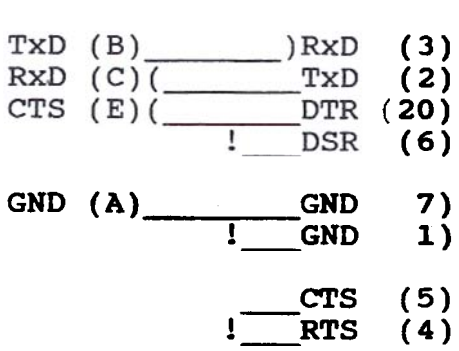
HARDWARE INTERFACE, COMPUTER MODE



The connections shown above are required before the peripheral computer can successfully communicate with the 580B. In addition, the 580B must be placed into COMPUTER mode. This is done from the 580B keypad.

An example of a cable used for COMPUTER MODE communication is shown below:

SAMPLE CABLE, 580B TO IBM PC



If the peripheral's DTR line is asserted (+V), the 580B will look for and then respond to peripheral commands. As long as DTR remains high, the 580B will NOT perform functions such as counting, updating the display, storing log data, etc. If DTR is asserted, the 580B will display the following message:

"COMPUTER LINK"
"WAITING FOR COMM"

The 580B must be allowed 100ms between the time the peripheral asserts DTR and the time it sends the 580B its first message. If the 580B is busy communicating the display will read:

"COMPUTER LINK"
"COMMUNICATING"

When DTR is low (-V), the 580B will return to its normal mode of operation where it counts, updates the display, etc. and will NOT respond to peripheral commands.

XON/XOFF HANDSHAKING, COMPUTER MODE

If the 580B is transmitting log data and detects that an XOFF has been received, it will stop transmitting. The 580B will not resume transmission until the peripheral device sends XON. The 580B will respond to XON and XOFF characters only if it is NOT communicating with the computer (i.e., if 580B CTS is NOT asserted).

It is important to note that if the 580B receives an XOFF it will stop transmitting and will simply wait for the peripheral to send XON, it will not acquire data, update the display or perform other functions. The 580B is essentially "locked up" until it receives XON. For this reason, it would be good programming practice to send out an XON to the 580B prior to 580B/peripheral communication and afterwards also. Consequently, if an XOFF character is sent to the 580B inadvertently, the 580B will not be locked up indefinitely.

PERIPHERAL/580B COMMAND MESSAGES

A command message is a string of upper-case ASCII characters terminated by an ASCII carriage return. The carriage return may immediately follow the command or a space may separate the command and the carriage return as shown in the example below. The command messages which the 580B will accept from the peripheral are listed in TABLE A.1. The 580B will accept the messages as shown in TABLE 1 or the command portion of the message can be abbreviated as follows:

"S R F 01.00 (ret)" instead of
"SET RESPONSE FACTOR 01.00 (ret)"

Please note that the abbreviation must contain the first letter of each and every word of the command. There are some additional guidelines for abbreviating the SET OPERATING MODE commands.

If the abbreviated version of a command is sent, an ASCII blank must separate each letter of the command and must separate the command from the data. Note that the command message will contain data only if a SET command is being sent.

If the spelled-out version of a command is sent, an ASCII blank must separate each word of the command and must separate the command from the data.

Every SET command message contains a data value. The data sent as part of a SET command must conform to the formats described in TABLE A.1. It is important to note that the 580B does NOT perform error-checking on data sent as part of a SET command.

It is up to the user to insure that the data value is "reasonable" and formatted as shown in TABLE A.1.

The following are examples of valid command messages:

"SET ACCESS LEVEL 3 (ret)"
or "S A L 3 (ret)"

"SET REAL TIME 02/15/86 1723 (ret)"
or "S R T 02/15/86 1723 (ret)"

580B/PERIPHERAL RESPONSE MESSAGES

A response message is a string of upper-case ASCII characters terminated by an ASCII carriage return. The response messages which the 580B will send to the peripheral are listed in TABLE A.1. The messages which the 580B sends in response to a GET command contain data formatted as shown. The notes which follow TABLE A.1 describe the GET command response messages in more detail.

SOFTWARE HANDSHAKING, COMPUTER MODE

Every command message must be preceded by the "WAKE UP/PROCEED" sequence. This sequence begins when the peripheral sends a WAKE UP ("?") character to the 580B. The 580B must respond with a PROCEED ("!") character before the computer can send a command message. In the discussion to follow, the WAKE UP/PROCEED sequence will be referred to as (WAKE UP/PROCEED).

The peripheral sends command messages to the 580B an entire line at a time. When the 580B receives the command line it will echo the line back to the peripheral. The peripheral will examine the echo to determine if the 580B received the command correctly. If the echo was correct, the peripheral will signal the 580B with the PROCEED character. The 580B will then perform the task specified by the command message. In the discussion ahead, the command message and echo sequence will be referred to as (COMMAND/ECHO/PROCEED).

The software handshaking sequences for each of the 3 types of commands are given below:

DO COMMANDS

The handshake sequence for a DO command is as follows:

1. (WAKE UP/PROCEED)
2. (COMMANDS/ECHO/PROCEED)
3. 580B performs task
4. 580B sends PROCEED or ERR

If the 580B was able to successfully complete the task it will send a PROCEED character in step 4, otherwise it will send the error message ("ERR").

GET COMMANDS

The handshake sequence below applies to all of the GET commands with the exception of GET LOG DATA and GET CONTINUED LOG:

1. (WAKE UP/PROCEED)
2. (COMMAND/ECHO/PROCEED)
3. 580B sends data message
4. Peripheral echoes data message
5. 580B sends PROCEED or ERR

The message sent by the 580B in response to a GET command are shown in TABLE 1. When the peripheral receives the message containing the data it echoes the entire message back to the 580B. If the echo is correct the 580B will send the PROCEED character so that the peripheral knows it received the data correctly. If the echo is not correct, the 580B will send "ERR".

The GET LOG DATA and GET CONTINUED LOG commands differ from the other GET commands in that the 580B sends an indefinite number of data values. The handshake sequence for these commands is as follows:

1. (WAKE UP/PROCEED)
2. (COMMAND/ECHO/PROCEED)
3. 580B sends a log data point message
4. If message = "EOT" (end of transmission) then DONE, otherwise go on to step 5.
5. Peripheral echoes entire message
6. 580B sends PROCEED or ERR
7. Peripheral sends PROCEED
8. Go to step 3

In step 4, the 580B sends "EOT" if it has sent all the log data points available. If some time later the peripheral sends "GET CONTINUED LOG (ret)" the 580B will send any additional data points it may have acquired since the GET LOG DATA command. In step 6, the 580B will send PROCEED if the peripheral echoed the message correctly in step 5. The 580B will also increment its data buffer pointer. If however, the peripheral did NOT correctly echo the message in step 5, the 580B will send "ERR" in step 6 and will NOT increment its data buffer pointer. This means that the next time through step 3, the 580B will send the same data point again. In either case, the peripheral must send a PROCEED in step 7.

COMMANDS

handshaking sequence for a SET command is as follows:

1. (WAKE UP/PROCEED)
2. (COMMAND/ECHO/PROCEED)
3. 580B sets parameter to value
4. 580B sends PROCEED or ERR

In step 3 the 580B sets the parameter specified by the command to the value provided by the peripheral in the command message. The data value sent in the SET command message must be formatted as shown in TABLE 1. If the 580B is able to successfully set the parameter it will send the PROCEED character, otherwise it will send "ERR".

NOTE: The flowcharts shown in Figures A.1 - A.4 are included to further explain the software handshaking sequences required for successful communication between the peripheral and the 580B.

TABLE A.1
MESSAGE FORMATS

PERIPHERAL COMMAND	580B RESPONSE
DO COMMANDS	
DO END COMMUNICATIONS (ret	! (ret)
DO RESET LOG (ret)	! (ret)
GET COMMANDS	
GET ACCESS LEVEL (ret)	ACCESS LEVEL I (ret)
GET ALARM SETTING (ret)	ALARM SETTING IIII (ret)
GET CONTINUED LOG (ret)	(see notes which follow)
GET INSTRUMENT NUMBER (ret)	INSTRUMENT # IIIIII (ret)
GET LOCATION CODE (ret)	LOCATION CODE IIIIII (ret)
GET LOG DATA (ret)	(see notes which follow)
GET OPERATING MODE (ret)	OPERATING MODE: ASCII (50) (ret)
GET MAX READING (ret)	LAST MAX VALUE IIII ASCII (8) (ret)
GET RATEMETER READING (ret)	LAST CONC VALUE IIII ASCII (8) (ret)
GET REAL TIME (ret)	REAL TIME CLOCK II/II/II IIII (ret)
GET USER ID (ret)	USER I.D. # IIIIIIIII (ret)
GET RESPONSE FACTOR (ret)	RESPONSE FACTOR II.II (ret)
GET SPAN CONCENTRATION (ret)	SPAN CONCENTRATION IIII (ret)
GET VERSION NUMBER (ret)	580B VERSION 1.0 (ret)
GET LOGGING INTERVAL (ret)	I:II (ret)
SET COMMANDS	
SET ACCESS LEVEL I (ret)	! (ret)
SET ALARM SETTING IIII (ret)	! (ret)
SET INSTRUMENT NUMBER IIIIII (ret)	! (ret)
SET LOCATION CODE IIIIII (ret)	! (ret)
SET OPERATING MODE ASCII (50) (ret)	! (ret)
SET REAL TIME II/II/II/ IIII (ret)	! (ret)
SET USER ID IIIIIIIII (ret)	! (ret)
SET RESPONSE FACTOR II.II (ret)	! (ret)
SET SPAN CONCENTRATION IIII (ret)	! (ret)
SET LOGGING INTERVAL I:II (ret)	! (ret)

NOTES ON TABLE A.1

The peripheral will receive data back from the 580B in response to a GET command only. The 580B response to a DO or SET command is the PROCEED ("!") character. This is because the 580B does not actually return data but signals the peripheral with the PROCEED character if it was able to perform the task requested. If for some reason the 580B is unable to perform the task it will send an error message "ERR" rather than PROCEED.

The data values sent by the peripheral as part of a SET command message and the data received by the peripheral in response to a GET command must be formatted as shown in TABLE A.1.

The data format codes used in TABLE 1 are described below:

I	a single digit
III...II	an integer "string", the number of I's shown indicates the length of the string. Note: the "string" MUST be the length specified, use leading zeros if necessary.
ASCII (n)	an ASCII string with a maximum of n characters

Several of the command messages listed in TABLE 1 require additional explanation. These comments are listed according to the command name:

DO RESET LOG

This command instructs the 580B to clear its log data file All log data values acquired previously will be erased.

GET CONTINUED LOG

This command instructs the 580B to send any log data points acquired since the last GET LOG DATA command. The format of the 580B response will be the same as the response to the GET LOG DATA command.

GET LOG DATA

This command instructs the 580B to send all of its log data points. The log data file is saved in 580B memory and is NOT automatically erased upon output. The log data file may be erased (reset) with the DO RESET LOG command.

GET OPERATING MODE

The 580B responds to a GET OPERATING MODE command by sending a string "MODE: ", followed by an ASCII string which describes the current 580B mode of operation. The 580B responses to the

GET OPERATING MODE command are listed below:

OPERATING MODE: CONCENTRATION METER NORMAL
OPERATING MODE: CONCENTRATION METER MAX HOLD

GET MAX READING

The 580B responds to a GET MAX READING command by sending the max value as shown in TABLE A.1. After the 580B sends the max reading it resets the max value to 0.

GET REAL TIME

The 580B will return its real time as an ASCII string in the following format: "05/29/86 1422"

SET ACCESS LEVEL

The access level must be an integer in the range 0 to 3

SET INSTRUMENT NUMBER

The instrument number is a string of 6 integers. If the instrument number = 2 then the number must be represented as 000002 (i.e. leading zeros must fill in excess spaces). An example of a valid SET INSTRUMENT NUMBER command is "SET INSTRUMENT NUMBER 000002 (ret)".

SET LOCATION CODE

The location code is a string of 6 integers. If the location code = 234 then the correct SET LOCATION CODE command is "SET LOCATION CODE 000234 (ret)". (Leading zeros must fill excess spaces.)

SET OPERATING MODE

As shown in TABLE A.1, the command SET OPERATING MODE must be followed by an ASCII string which describes the mode. The list of valid SET OPERATING MODE commands along with valid abbreviations are listed below:

SET OPERATING MODE CONCENTRATION (ret)
S O M C (ret)

SET OPERATING MODE MAX HOLD (ret)
S O M M H (ret)

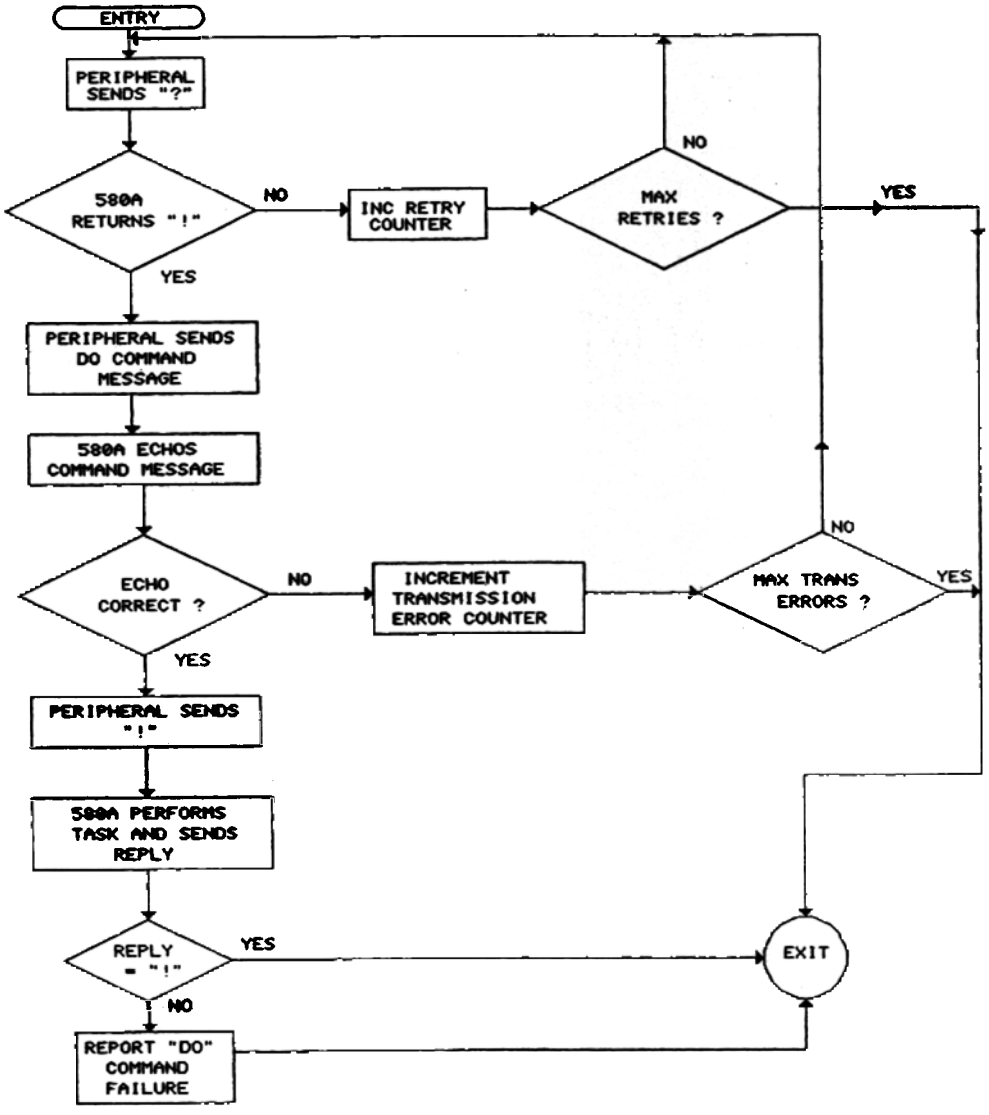


Figure A.1
Software Handshake
Do Commands

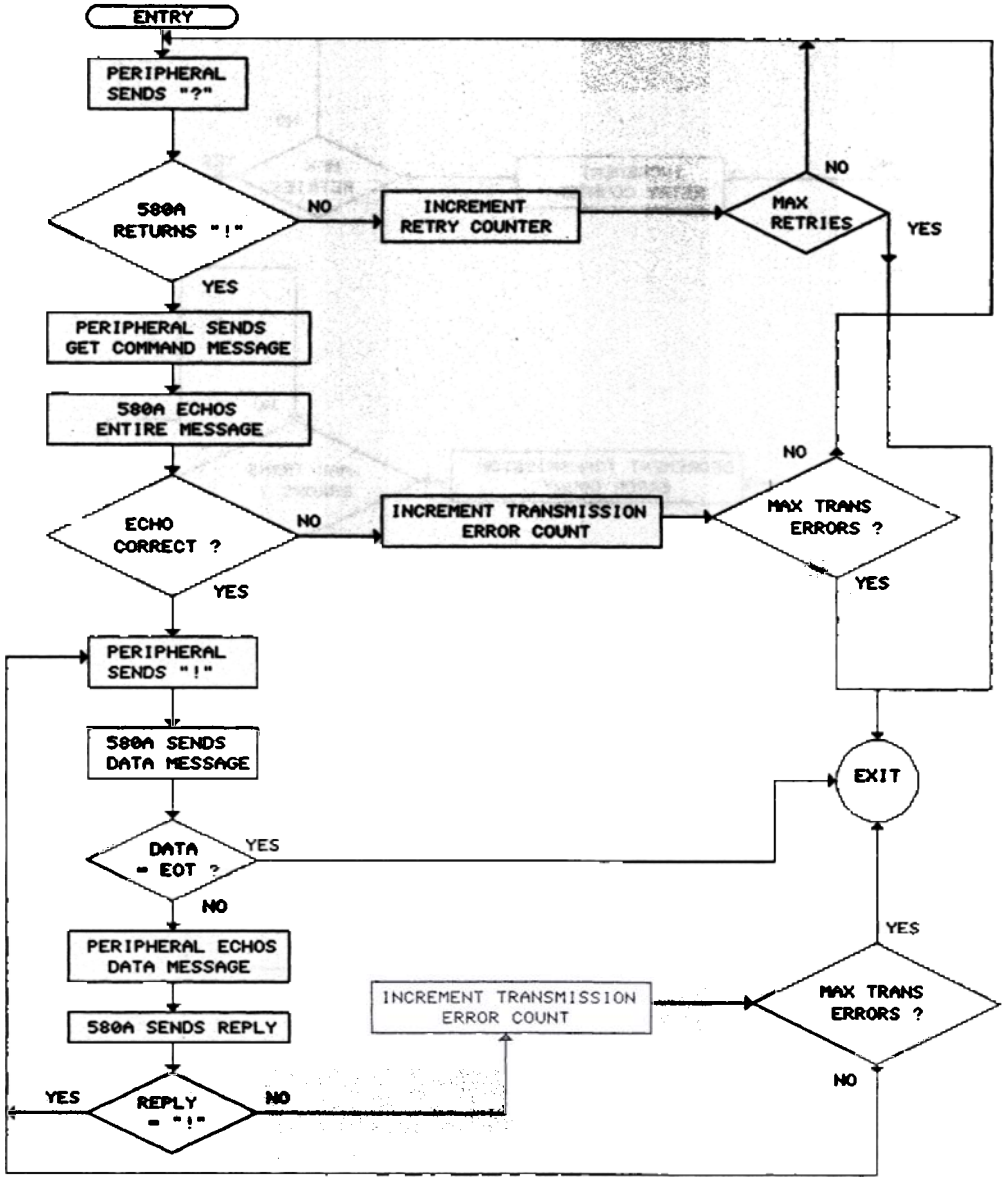


Figure A.2
Software Handshake
Get Log Data, Get Continue Log Command

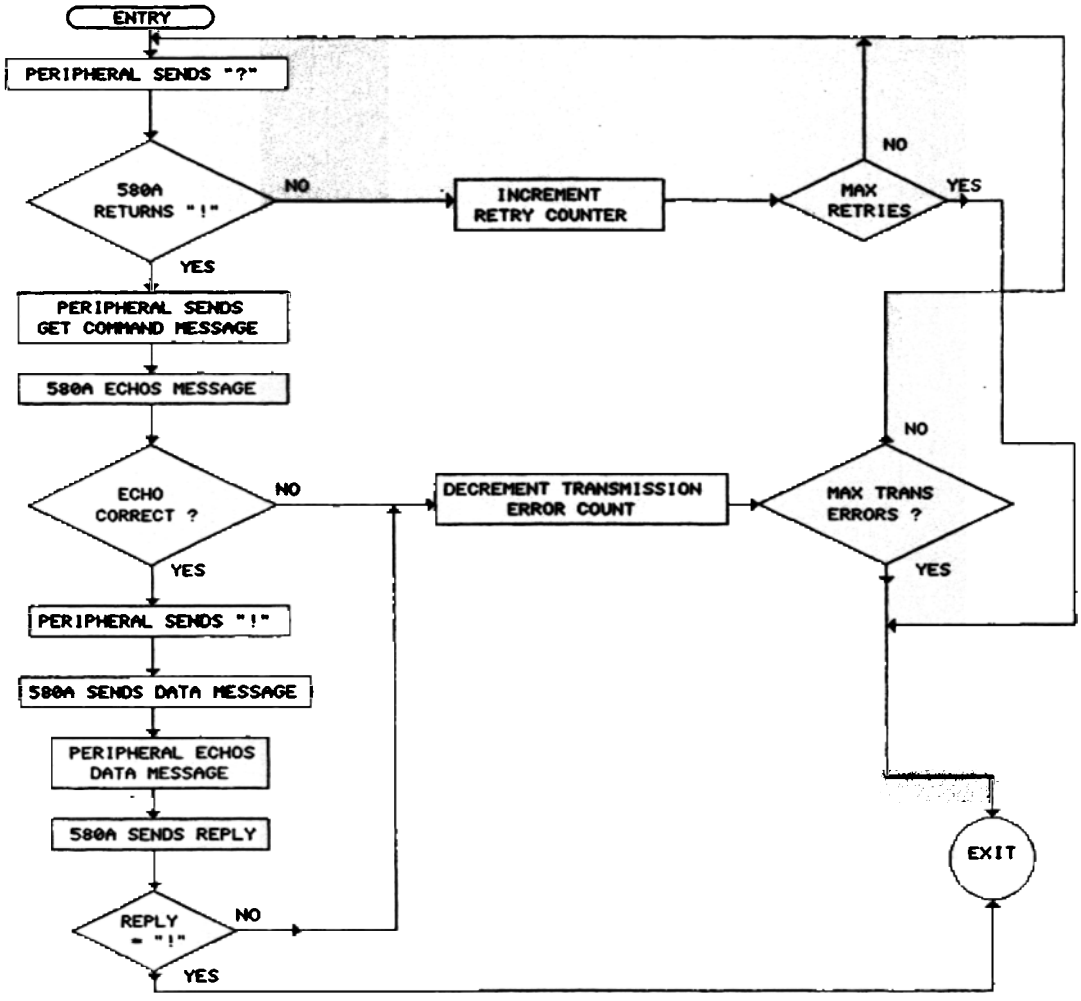


Figure A.3
Software Handshake
Get Command
(Except Get Log Data, Get Continue Log)

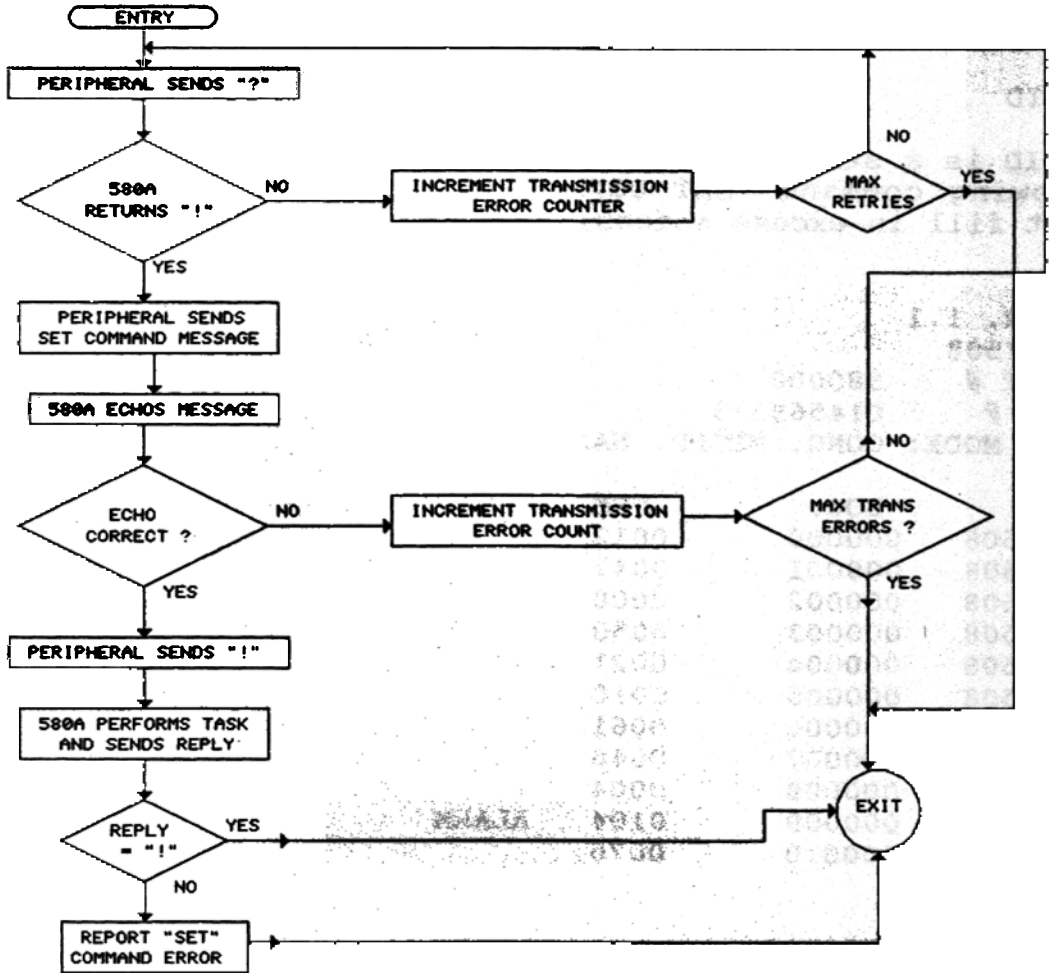


Figure A.4
Software Handshake
Set Commands

SET REAL TIME

The format for setting the real time is as follows:

```
II/II/II IIII
 .  .  .  . .
    .  .  . .........minutes (max=59)
    .  .  ...........hours (max=23)
    .  ..............year
    ..................day
    ..................month
```

For example: SET REAL TIME 02/15/86 1723 (ret)
This instructs the 580B to set its real time clock to February 15, 1986 5:23 p.m.

SET USER ID

The user ID is a string of 9 integers. To set user ID = 66, use the following command "SET USER ID 000000066 (ret)" (leading zeros must fill in excess spaces).

```
 580B   VER. 1.1
 07/11/88 1508
 INSTRUMENT #     580000
 USER I.D. #      014569373
 OPERATING MODE: CONC. METER, MAX HOLD

                   LOC.         PPM     STATUS
07/11/88 1508    000000       0012
07/11/88 1508    000001       0047
07/11/88 1508    000002       0000
07/11/88 1508    000003       0050
07/11/88 1508    000004       0021
07/11/88 1508    000005       0010
07/11/88 1509    000006       0061
07/11/88 1509    000007       0046
07/11/88 1509    000008       0004
07/11/88 1509    000009       0104    ALARM
07/11/88 1509    000010       0076

 07/11/88 1509
 INSTRUMENT #     580000
 USER I.D. #      014569373
 OPERATING MODE: CONC. METER

                   LOC.         PPM     STATUS
07/11/88 1509    000011       0000
07/11/88 1509    000012       0064
07/11/88 1509    000013       0052
07/11/88 1509    000014       0001
07/11/88 1509    000015       0007
07/11/88 1509    000016       0101    ALARM
```

Figure A.5
Data Log Output

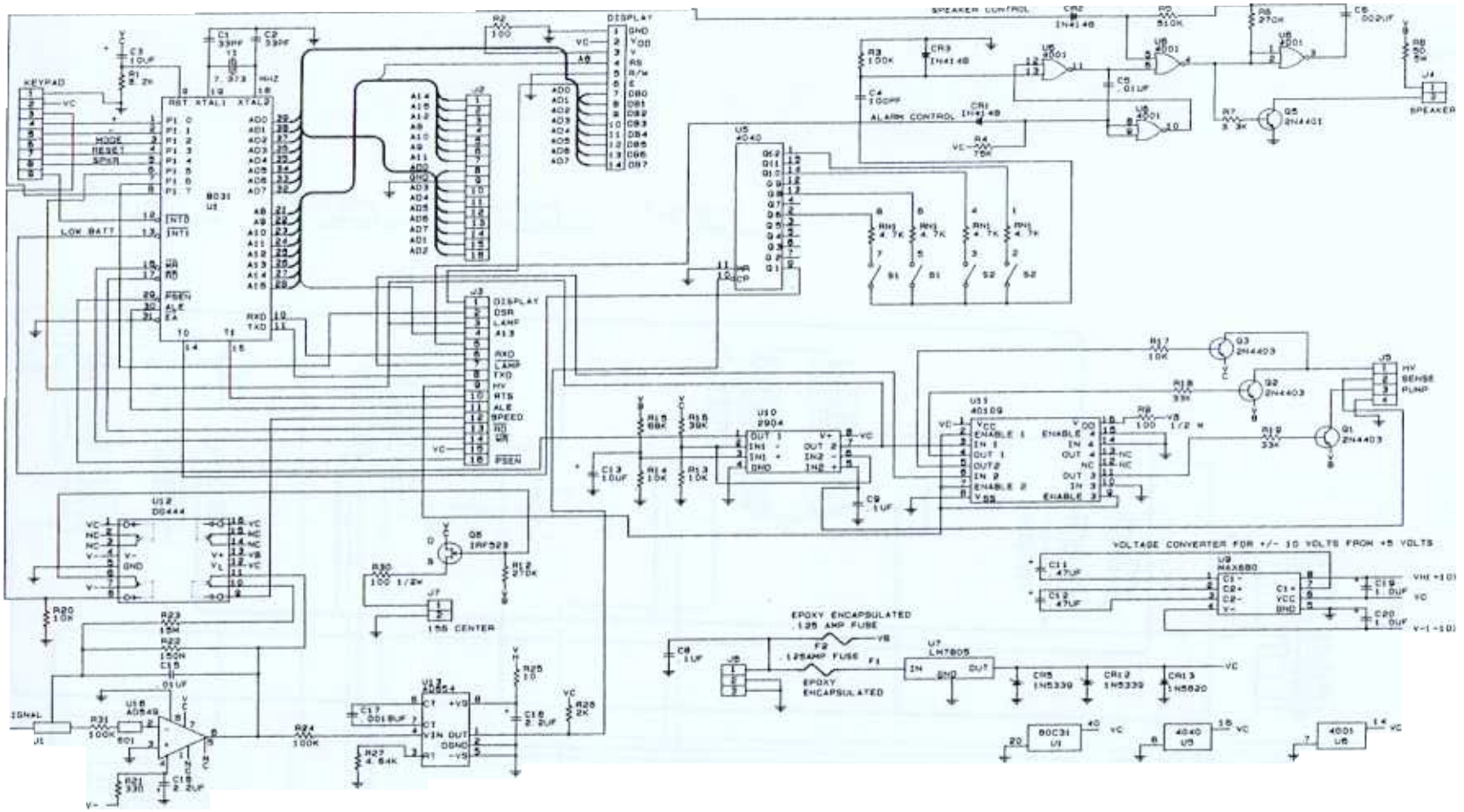


Figure B-1

Processor/Ranging Bd. Schematic

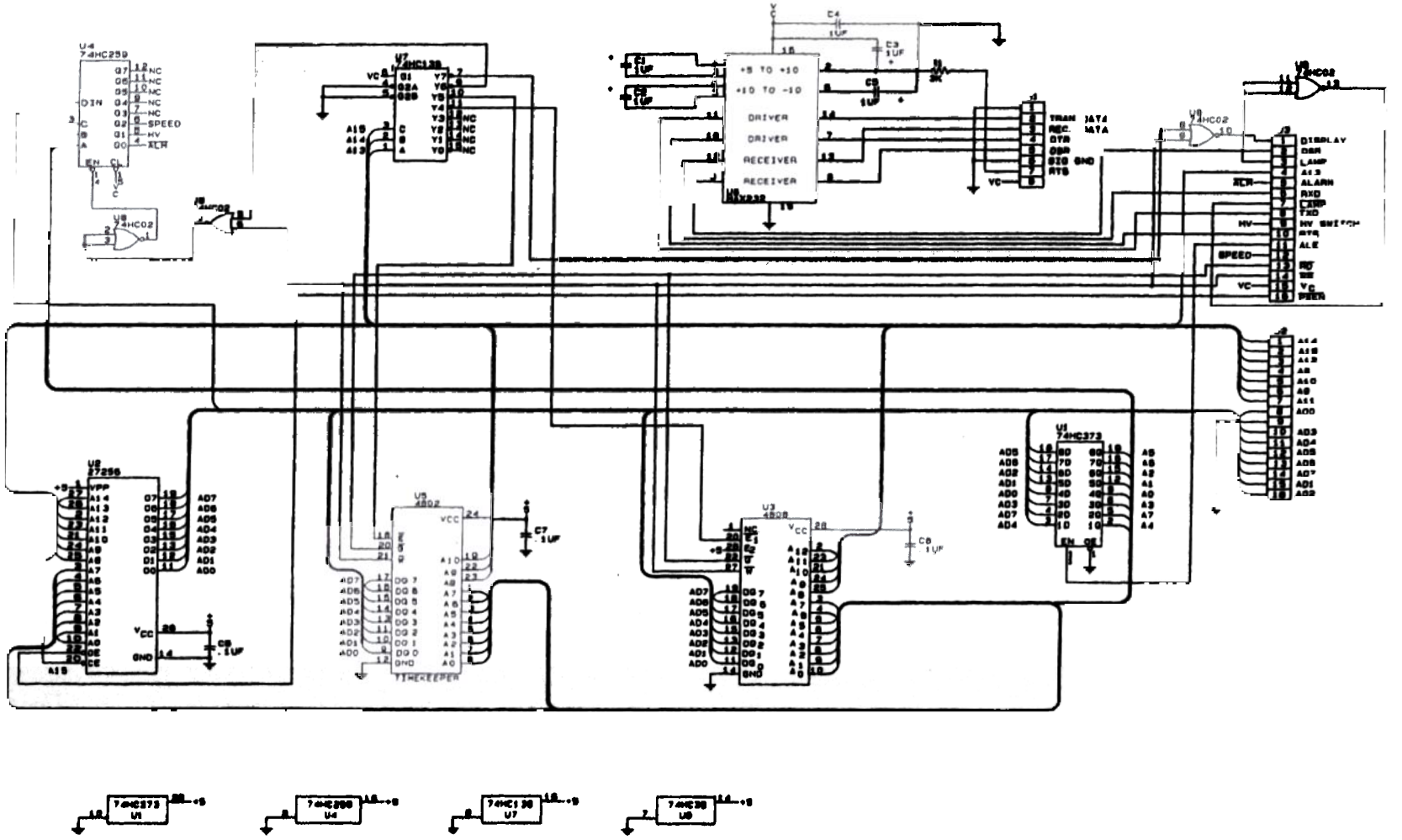


Figure B.2

Memory Board Schematic

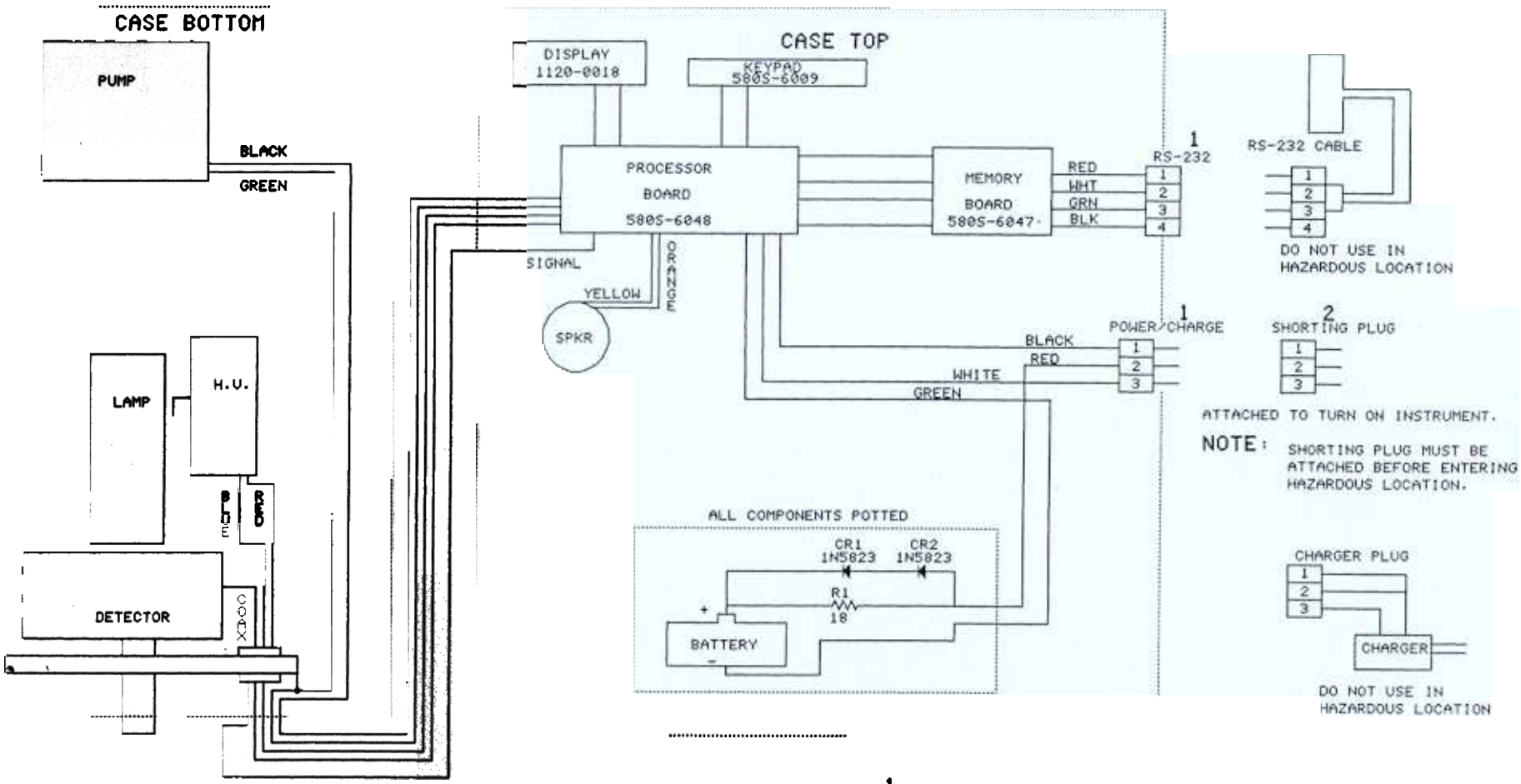


Figure C.1
Block Diagram
C-1

1 CONNECTORS MOUNTED ON REAR OF CASE TOP.

2 SHORTING PLUG IS TETHERED TO PREVENT ITS LOSS.

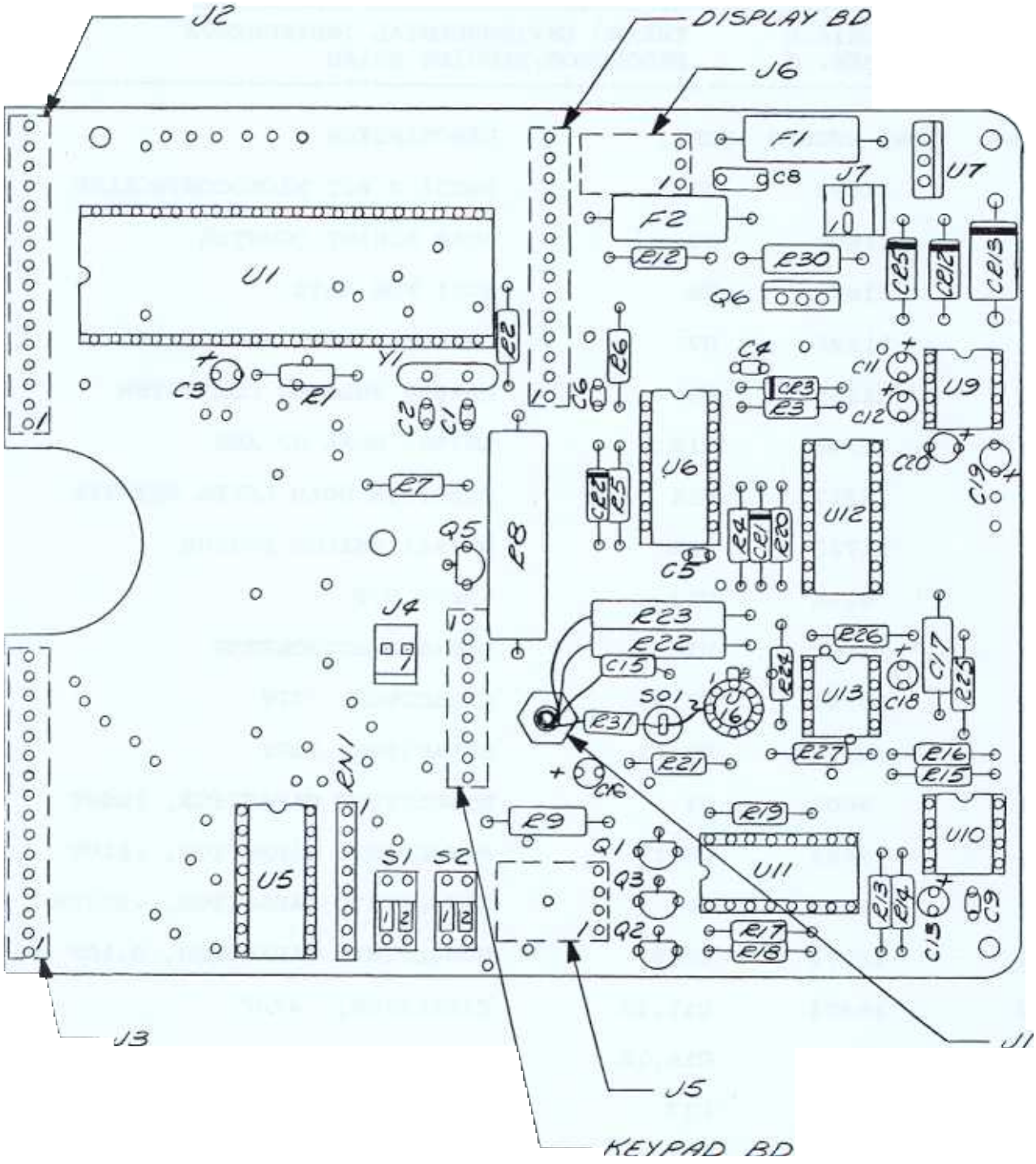


Figure D-1
Processor/Ranging Bd. Loading Drawing

BILL OF MATERIALS THERMO ENVIRONMENTAL INSTRUMENTS
580S-6048 REV. A PROCESSOR/RANGING BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	11695	U1	80C31 8 BIT MICROCONTROLLER
1	11696	U5	4040 BINARY COUNTER
1	11697	U6	4001 NOR GATE
1	11126	U7	LM7805 5 VOLT REGULATOR
1	11159	U9	MAX680 VOLTAGE CONVERTER
1	11716	U10	LM2904 DUAL OP AMP
1	11717	U11	CD40109B QUAD LEVEL SHIFTER
1	11723	U12	DG-444 ANALOG SWITCH
1	9296	U13	AD654 V/F
1	11152	U16	AD549 ELECTROMETER
2	10391	C1,2	CAPACITOR, 33PF
2	10376	C3,13	CAPACITOR, 10UF
1	5609	C4	MONOLITHIC CAPACITOR, 100PF
2	5605	C5,15	MONOLITHIC CAPACITOR, .01UF
1	10399	C6	MONOLITHIC CAPACITOR, .002UF
2	10372	C8,9	MONOLITHIC CAPACITOR, 0.1UF
2	10403	C11,12	CAPACITOR, .47UF
2	10390	C16,18	CAPACITOR, 2.2UF
1	10402	C17	POLYPROPYLENE CAP 1.8NF,5%
2	10378	C19,20	CAPACITOR, 1.0UF
1	10790	R1	RESISTOR, 8.2K
1	10847	R2	RESISTOR, 100, 1/4 W
3	10862	R3,24,31	RESISTOR, 100K
1	10928	R4	RESISTOR, 75K

BILL OF MATERIALS THERMO ENVIRONMENTAL INSTRUMENTS
580S-6048 REV. A PROCESSOR/RANGING BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	10929	R5	RESISTOR, 510K
2	10930	R6,12	RESISTOR, 270K
1	5986	R7	RESISTOR, 3,3K
1	10939	R8	RESISTOR, 50, 5W
2	10704	R9,30	RESISTOR, 100, 1/2W
4	10864	R13,14,17,20	RESISTOR, 10K
1	10938	R15	RESISTOR, 68K
1	6025	R16	RESISTOR, 39K
2	2219	R18,19	RESISTOR, 33K
1	10786	R21	RESISTOR, 330
1	10951	R22	RESISTOR, 150M, 5%
1	10952	R23	RESISTOR, 15M, 5%
1	10846	R25	RESISTOR, 10
1	10936	R26	RESISTOR, 2K
1	10865	R27	RESISTOR, 4.64K
1	11641	RN1	NETWORK, 4.7K X 6
3	11807	CR1-3	1N4148
2	11829	CR5,12	1N5339
1	11805	CR13	1N5820
1	10557	Y1	7.373 MHZ
1	10446	J1	RADIAL SMC
1	11398	J4	2 PIN AMP, .100 CENTER
1	11418	J5	4 PIN MTE

BILL OF MATERIALS THERMO ENVIRONMENTAL INSTRUMENTS
580S-6048 REV. A PROCESSOR/RANGING BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	11419	J6	3 PIN MTE
1	11405	J7	2 PIN AMP .156 CENTER
2	580S-6040	F1,2	.125 AMP FUSE ASSEMBLY
1	10422	S01	STANDOFF
2	12138	S1,2	SPST SWITCH, DUAL
3	11762	Q1-3	2N4403
1	11759	Q5	2N4401
1	11773	Q6	IRF523
1	580S-2057 REV. A		P.C. BOARD BLANK

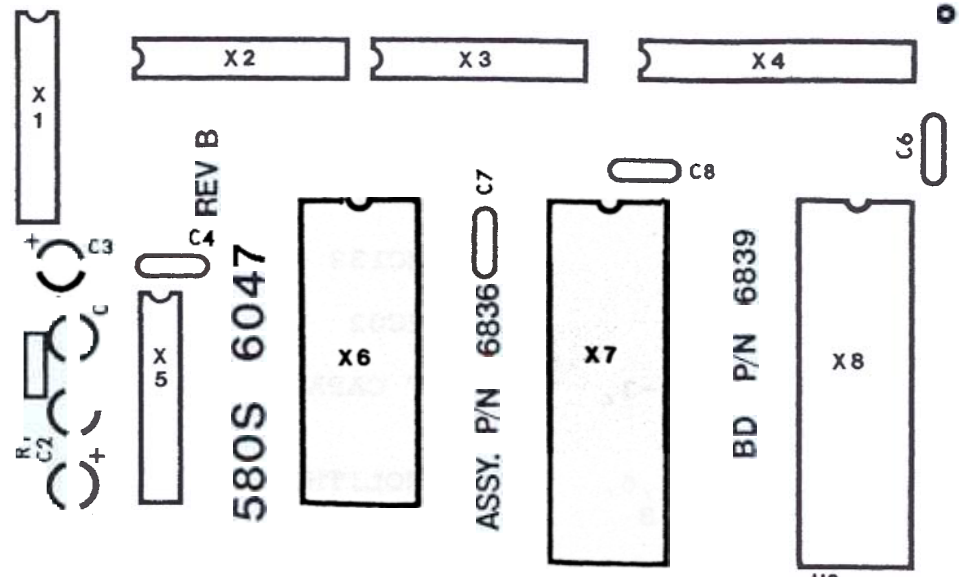
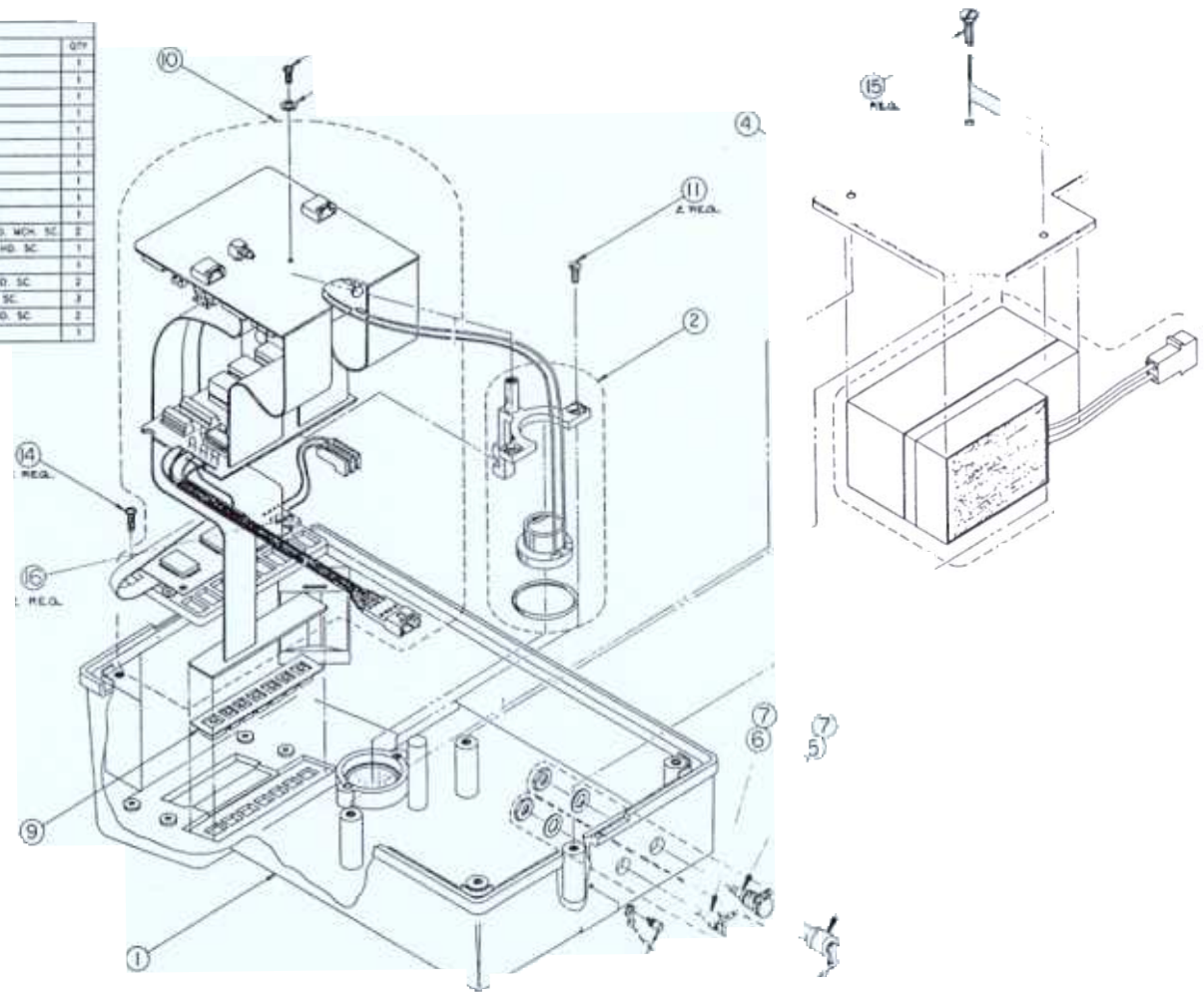


Figure D.2
Memory Board Loading Drawing

BILL OF MATERIALS THERMO ENVIRONMENTAL INSTRUMENTS
580S-6047 REV. A MEMORY BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	11689	U1	74HC373
1	11728	U2	27C256 8K EPROM
1	11687	U3	48Z08 8K X 8 BATTERY BACKED RAM
1	11688	U4	74HC259 OUTPUT PORT
1	11691	U5	48T02 2K X 8 TIMEKEEPER RAM
1	11685	U6	MAX232 RS-232 CHIP
1	11151	U7	74HC138
1	11730	U8	74HC02
4	10378	C1-3, 5	1UF CAPACITOR
4	10372	C4,6, 7,8	MONOLITHIC CAPACITOR, .1UF
1	10789	R1	RESISTOR, 3K
1	11374	X7	28 PIN SOCKET
1	580S-2056 REV. A		P.C. BOARD BLANK

MATERIAL LIST			
ITEM	PART NO.	DESCRIPTION	QTY
1	16801	CASE TOP	1
2	13675	SPEAKER ASSY.	1
3	13592	BATTERY ASSY.	1
4	13497	BATTERY HOLDER	1
5	13555	POWER HARNESS	1
6	5808-8029	RS-232 HARNESS ASSY.	1
7	11431	DUST COVER	1
8	16808	SHORTING PLUG	1
9	10460	KEYPAD	1
10	16838	P.C. BD SET UL	1
11	5868	8-32 X 1/4" LG. FLT. HD. MCH. SC.	2
12	5815	4-40 X 5/16" LG. BND. HD. SC.	1
13	7534	NYLON WASHER	1
14	5856	8-32 X 1/2" LG. PAN. HD. SC.	2
15	12088	8-32 X 1" LG. FLAT HD. SC.	3
16	5822	8-32 X 5/8" LG. BIND HD. SC.	2
17	9383	4-40 INSERT	1



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APPENDIX F
COMMON ORGANIC SOLVENTS AND GASES DATA SHEET

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{O}C)*	I.P. (EV)**	TWA (PPM)***
Acetaldehyde	44.05	0.788	21	10.21	
Acetamide	59.07	1.159	221	9.77	
Acetic Acid	60.05	1.049	116-117	10.37	10
Acetic Anhydride	102.1	1.10	138-117	9.88	5
Acetone	58.1	0.79	56	9.69	1000
Acetonitrile	41.1	0.79	82	12.22	40 cell
Acetophenone	120.15	1.033	202	9.27	
Acetyl Bromide	122.96	1.52	75-75	10.55	
Acetyl Chloride	78.50	1.104	52	11.02	
Acetylene	26.02	0.90		11.41	
Acrolein	56.06	0.8389	53	10.10	0.1
Acrylonitrile	53.06	0.8004	77	10.91	10
Allyl Alcohol	58.1	0.85	96-98	9.67	2
Allyl Chloride	76.5	0.94	44-46	9.9	1
Aniline	93.1	1.02	184	7.70	5
Anisole	108.13	0.9956	154	8.22	
Ammonia	17.03	gas		10.15	
Arsine	77.9	gas			
Benzaldelyde	106.12	1.053	178-185	9.53	
Benzene	78.1	0.88	80	9.25	1
Benzonitrile	103.12	1.010	188	9.71	
Benzotriflouride	146.11	1.1886	102	9.68	
Benzyl Chloride	126.6	1.10	177-181	9.14	1
Biphenyl	154.21	0.992	255		
Bromine	159.81	3.1023	58.8	10.55	
Bromobenzene	157.02	1.495	156	8.98	
1-Bromobutene	137.03	1.276	100-04	10.13	
2-Bromobutene	137.03	1.255	91	9.98	
1-Bromo-2-Chlorethene	143.42	1.723	106-07	10.63	
Bromochloromethane	129.39	1.991	68	10.77	
1-Bromo-2-Flourobenzene	175.01	1.593	150	8.99	
Bromoform	252.8	2.9	150-01	10.47	0.5
1-Bromo-2-methyl propane	137.03	1.260	90-92	10.09	
2-Bromo-2-methyl propane	137.03	1.189	72-74	9.89	
1-Bromopentane	151.05	1.218	130	10.10	
2-Bromopropane	123.00	1.354	71	10.18	
2-Bromopropene	123.00	1.310	59	10.08	
1-Bromopropene	120.98	1.413	58-63	9.30	
3-Bromopropene	120.98	1.398	70-71	9.70	
2-Bromothiophene	163.04	1.684	149-151	8.63	
M-Bromotoluene	171.04	1.4099	183.7	8.81	
O-Bromotoluene	171.04	1.431	58.60	8.79	
P-Bromotoluene	171.04	1.431	184	8.67	
1,3-Butadiene	54.1	gas	9.07		1000
Butane	58.12	gas	10.63		
	90.19	.842	9.14		

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{o}C)*	I.P. (EV)**	TWA (PPM)***
2-Butanone	72.1	0.81	80	9.53	200
1-Butene	56.10	0.6255		9.58	
N-Butyl Acetate	116.2	0.88	124-26	10.01	150
S-Butyl Acetate	116.2	0.88	111-12	9.91	150
N-Butyl Alcohol	74.1	0.81	117.7	10.04	100
N-Butyl Amine	73.1	0.73	73	78	5
S-Butyl Amine	73.1	0.73	63	8.70	5
T-Butyl Amine	73.1	0.73	46	8.64	5
N-Butyl Benzene	134.21	0.8604	183	8.69	
S-Butyl Benzene	134.21	0.8604	173-04	8.68	
T-Butyl Benzene	134.21	0.8669	169	8.68	
N-Butyraldehyde	72.10	0.8016	75	9.86	
N-Butyric Acid	88.10	0.959	162	10.16	
N-Butyronitrile	69.10	0.7954	115-17	11.67	
Camphor	152.2	0.99	204	8.76	2 mg/m
Carbon Dioxide	44.01	gas		13.79	5000
Carbon Monoxide	28.01	gas		14.01	50
Carbon Tetrachloride	153.8	1.59	77	11.47	10
Chlorine	70.90	gas		11.48	1 cell
Chlorobenzene	112.6	1.10	132	9.07	75
Chloroform	119.4	1.48	60.5-61.5	11.37	50 cell
1-Chloro-2-Methylpropane	92.57	.883	68-69	10.66	
2-Chloro-2-Methylpropane	101.64	.851	51-52	10.61	
1-Chloropropane	78.54	.892	46-47	10.82	
2-Chloropropane	78.54	.859	34-36	10.78	
3-Chloropropane	76.53	.939	44-46	10.04	
2-Chlorothiophene	118.59	1.286	127-29	8.68	
M-Chlorotoluene	126.58	1.076	160-162	8.83	
O-Chlorotoluene	126.58	1.0826	157-159	8.83	
P-Chlorotoluene	126.58	1.0697	162	8.70	
M-Cresol	108.1	1.034	203	8.52	5 cell
O-Cresol	108.1	1.048	191	8.50	5 cell
P-Cresol	108.1	1.034	202	8.38	5 cell
Crotonaldehyde	70.09	0.853	104	9.73	2
Cumene	120.2	0.86	152-154	8.75	50
Cyanogen	52.04	0.9537		13.80	
Cyclohexane	84.2	0.81	80.7-81	9.98	300
Cyclohexane	100.2	0.96	160-161	10.0	50
Cyclohexanone	98.1	0.95	155	9.14	50
Cyclohexene	82.1	0.81	83	8.95	300
Cyclo-Octatetraene	104.15	0.925	142-43	7.99	
Cyclopentane	70.13	0.7460	50	10.53	
Cyclopentanone	84.11	1.4366	130-131	9.26	
Cyclopentene	68.12	0.744	44	9.01	
Cyclopropane	42.08	gas		9.91	
Diborane	27.68	gas		11.00	
Diazomethane	42.0	gas		9.0	0.2

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{O}C)*	I.P. (EV)**	TWA (PPM)**
Dibromodiflouromethane	209.83	2.297	22-23	11.07	
1,2-Dibromoethane	187.87	2.180	131-32	9.45	
1,3-Dibromopropane	201.90	1.937	167	10.07	
Dibutylphthlate	278.3	1.04	340		5 mg/m
M-Dichlorobenzene	147.01	1.288	172-73	9.12	50
O-Dichlorobenzene	147.01	1.306	179-180	9.07	50
P-Dichlorobenzene	147.01	1.241	173	8.94	75
1,1-Dichlorethane	99.0	1.18	57	11.06	100
1,2-Dichlorethane	98.96	1.256	83	11.12	
1,2-Dichlorethylene	97.0	1.28	46-60	9.66	
Dichloromethane	84.93	1.325	39.8-40	11.35	
1,2-Dichloropropane	112.99	1.156	95-96	10.87	
1,3-Dichloropropane	112.99	1.190	120-22	10.85	
2,3-Dichloropropane	110.97	1.204	94	9.82	
N,N-Diethyl Acetamide	115.18	0.925	182-86	8.60	
Diethylamine	73.1	0.71	55	8.01	25
Diethyl Ether	74.12	0.7134	34.6	9.53	
N.N-Diethyl Formamide	101.15	0.908	176-77	8.89	
Diethyl Ketone	86.13	0.816	102	9.32	
Diethyl Sulfide	90.19	.837		8.43	
Diethyl Sulfite	138.19	1.883	158-60	9.68	
Dihydropyran	84.12	0.922	86	8.34	
Diisopropylamine	101.2	0.72	84	7.73	5
1,1-Dimethoxyethane	90.12	0.863	64	9.65	
N,N-Dimethyl Acetamide	87.12	0.937	164.5-66	8.81	10
Dimethyl Amine	45.1	0.68		8.24	10
N,N-Dimethyl Aniline	122.2	0.96	193-94	7.13	
2,2-Dimethyl Butane	86.18	0.649	50	10.06	
2,3-Dimethyl Butane	86.18	0.662	50	10.02	
3,3-Dimethyl Butanone	100.16	0.801	106	9.17	
N,N-Dimethyl Formamide	73.09	0.9445	153	9.12	10
Dimethlyl Sulfide	63.13	0.846	38	8.69	
P-Dioxane	88.1	1.03	100-102	9.13	
Dipropyl Amine	101.19	0.738	105-110	7.84	
Durene	134.12	0.84	80-82	8.03	
Epichlorohydrin	92.5	1.18	115-117		5
Ethane	30.07	gas		11.65	
Ethanethiol	62.13	0.8315	35	9.29	
Ethyl Acetate	88.1	0.90	76.5-77.5	10.11	400
Ethyl Alcohol	46.1	0.80	78	10.48	1000
Ethyl Amine	45.1	0.69	19.20	8.86	10
Ethyl Benzene	106.2	0.87	136	8.76	100
Ethyl Bromide	109.0	1.45	37-40	10.29	200
Ethyl Butyl Ketone	114.2	0.82	146-49	9.02	50
Ethyl Chloride	64.52	0.9214		10.98	1000
Ethyl Disulfide	122.25	0.993	153	8.27	
Ethylene Dibromide	187.9	2.17	131-132	10.52	20
Ethylene Dichloride	99.0	1.26	83	11.32	50

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{o}C)*	I.P. (EV)**	TWA (PPM)**
Ethyl Ether	74.1	0.73	34.6	9.59	400
Ethyl Formate	74.1	0.92	52-54	10.61	100
Ethyl Iodide	155.98	1.950	67-73	9.33	
Ethyl Isothiocyanate	87.15	1.003	60	9.14	
Ethyl Methyl Sulfide	76.16	0.842	66-67	8.55	
Ethyl Nitrate	75.07	0.90	112	11.22	
Ethyl Propionate	102.13	0.891	99	10.00	
Ethyl Thiocyanate	87.14	1.007		9.89	
Ethynylbenzene	102.13	0.9300	142-44	8.82	
Fluorine	37.99	gas		15.70	
Flourobenzene	96.10	1.024	85	9.20	
O-Fluorophenol	112.10	1.256	172-74	8.95	
M-Fluorotoluene	110.13	0.997	178	8.92	
O-Fluorotoluene	110.13	1.004	172-172	8.92	
P-Fluorotoluene	110.13	1.001	185	8.79	
Formaldehyde	30.03	1.083		10.87	3
Formahide	45.04	1.1334	210	10.25	
Formic Acid	46.02	1.220	110-101	11.05	
2-Furaldehyde	96.09	1.160	182	9.21	
Furan	68.07	0.9371		8.89	
Heptane	100.2	0.68	98	10.08	
2-Heptanone	114.18	0.8068	149-50	9.33	
Hexane	86.2	0.66	68-69	10.18	500
1-Hexane	84.16	0.673	64	9.46	
Hexone	100.2	0.80		9.53	
Hydrogen	2.017	gas		15.43	
Hydrogen Bromide	80.92	gas		11.62	3
Hydrogen Chloride	36.47	gas		12.74	5 cell
Hydrogen Cyanide	27.03	0.687		13.91	10
Hydrogen Flouride	20.01	gas		15.77	3
Hydrogen Iodide	127.93	gas		10.38	
Hydrogen Selenide	80.98	gas		9.88	0.05
Hydrogen Sulfide	34.08	gas		10.46	20 cell
Hydrogen Telluride	129.63	gas		9.14	
Iodine	253.81	4.93		9.28	0.1 cell
Iodobenzene	204.02	1.8384	188	8.73	
1-Iodobutene	184.02	1.617	130-31	9.21	
2-Iodobutene	184.02	1.4991	119-120	9.09	
1-Iodo-2-Methylpropane	184.02	1.599	120-21	9.18	
1-Iodopentane	198.05	1.517	154-55	9.19	
1-Iodopropane	169.99	1.743	101-02	9.26	
2-Iodopropane	169.99	1.703	88-90	9.17	
O-Iodotoluene	218.04	1.713	211	8.62	
M-Iodotoluene	218.04	1.698		8.61	
P-Odotoluene	218.04		211-5	8.50	
Isoamyl Acetate	130.2	0.88	142	9.94	
Isoamyl Alcohol	88.2	0.81	130-1	10.42	

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{O}C)*	I.P. (EV)**	TWA (PPM)**
Isobutyl Amine	73.14	0.724	64-71	8.70	
Isobutyl Formate	102.13	0.885	98.4	10.46	
Isobutylene	56.11	0.5942	-6.9	9.23	
Isobutyraldehyde	72.11	0.794	63	9.74	
Isobutyric Acid	88.11	0.950	153-54	10.02	
Isoctane	114.2	0.70	98-99	17.9	
Isopentane	114.23	0.692	30	10.32	
Isoprene	68.12	0.681	34	8.85	
Isopropyl Acetate	102.1	0.87	85	9.99	250
Isopropyl Alcohol	60.1	0.79		10.16	400
Isopropyl Amine	59.1	0.69	33-34	8.72	5
Isopropyl Benzene	120.2	0.86	152-54	8.75	50
Isopropyl Ether	102.2	1.37	68-69	9.20	500
Isovaleraldehyde	86.13	0.785	90	9.71	
2,3-Lutidine	107.15	0.945	162-63	8.85	
2,4-Lutidine	107.15	0.927	159	8.85	
2,6-Lutidine	107.15	0.9252	143-45	8.85	
Malaic Anhydride	98.1	0.93	200	11.1	
Mesitylene	120.19	0.8637	162-64	8.40	
Mesityl Oxide	98.14	0.8592	129	9.08	
Methane	16.04	gas		12.98	
Methanethiol	48.11	0.96		9.44	
N-Methyl Acetamide	73.10	0.957	204-05	8.90	
Methyl Acetate	74.08	0.9279	57.5	10.27	
Methyl Acrylate	86.1	0.96	80	9.9	10
Methyl Amine	31.06	gas	48	8.97	
Methyl Bromide	95.0	gas		10.53	
2-Methyl-1-Butane	70.16	0.650	31	9.12	
3-Methyl-1-Butane	70.14	0.627	20	9.51	
3-Methyl-2-Butane	70.14	0.643		8.67	
Methyl Butyl Ketone	100.6	0.83	127	9.34	
Methyl Butyrate	102.13	0.898	102-103	10.07	
Methyl Chloride	50.5			11.28	
Methyl Cyclohexane	98.19	0.770	101	9.85	
Methyl Disulfide	94.20	1.046	109	8.46	
Methyl Ethyl Ketone	72.10	0.805	80	9.53	
Methyl Formate	60.1	1.34	34	10.815	100
2-Methyl Furan	82.10	0.827	63-66	8.39	
Methyl Iodide	142.0	2.28	41-43	9.54	5
Methyl Isobutyl Ketone	100.2	0.80	117-18	9.30	100
Methyl Isobutyrate	102.13	0.891	90	9.98	
Methyl Isopropyl Ketone	86.12	0.805	94-95	9.32	
Methyl Isothiocyanate	73.12		37-39	9.25	
Methyl Methacrylate	100.1	0.94	100	9.9	100
1-Methyl Napthalene	142.20	1.001	240-243	7.96	
2-Methyl Napthalene	142.20	1,000	241-242	7.96	
2-Methyl Pentane	86.18	0.653	62	10.12	
3-Methyl Pentane	86.18	0.664	64	10.08	
Methyl Propionate	88.11	0.915	79	10.15	

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{O}C)*	I.P. (EV)**	TWA (PPM)**
Methyl Propyl Ketone	86.13	0.809	100.01	9.38	
2-Methyl Styrene	165.4	1.068	131	10.07	
Monomethyl Aniline	107.16	0.989			
Monomethyl Hydrazine	46.1	0.87			
Morpholine	87.1	1.01	129	8.88	20
Nephthalene	93.7	1.16	217.7	8.12	10
Nitric Oxide	162.2	1.01		9.25	
P-Nitroaniline	138.1				1.0
Nitrobenzene	123.1	1.21	210-211	9.92	1.0
4-Nitrobiphenyl	199.2				
P-Nitrochlorobenzene	157.6	1.52		9.96	1.0 mg/m
Nitrogen Dioxide	46.01	1.448		9.78	
Nitroethane	75.1	1.38	112	10.81	100
Nitromethane	61.0	1.13	100.8-101	11.08	100
1-Nitropropane	89.1	0.99	131-32	10.88	25
2-Nitropropane	89.1	0.98	120	10.71	25
N-Nitrosodimethylamine	74.1	1.00	153	9.07	
Nitrotoluene	137.1	1.16	225-238	11.63	5
Oxygen	31.9988	gas		12.08	
Ozone	48.00	gas		12.08	
Pentaborane	63.17	0.61		10.40	
Pentane	72.15	0.62638	35	10.35	
2,4-Pentanedione	70.13	0.6429	140.4	8.87	
1-Pentene	70.13	0.6503	29.9-30.1	9.50	
Phenetol	122.16	0.967	169-70	8.13	
Phenol	94.1	1.07	182	8.50	5
Phenyl Hydrazine	108.1	1.1	238-41	7.86	5
Phenyl Isocyanate	119.12	1.0887	162-63	8.77	
Phenyl Isothiocyanate	135.18	1.1288	221	8.52	
Phosgene	98.9	gas		11.77	0.4 mg/m
Phosphine	34.0	gas			0.3
Phosphorous Pentachloride	208.2	1.6		10.7	1 mg/m
Phosphorous Trichloride	137.3	1.57	76	10.5	
2-Picoline	93.12	0.950	128-29	9.02	
3-Picoline	93.12	0.9613	144	9.02	
4-Picoline	93.12	0.9571	145	9.04	
Propane	44.09	gas		11.07	
1-Propanethiol	76.16	0.841	67-68	9.20	
Propiolactone	72.06	1.146	162	9.70	
Propionic Acid	74.08	0.99336	141	10.24	
Propionaldehyde	58.08	0.8071	46-50	9.98	
Propionitrile	55.08	0.7818	97	11.84	
N-Propyl Acetate	102.1	0.84	120	10.04	200
Propyl Alcohol	60.10	0.804	97	10.20	
Propyl Amine	59.11	0.719	48	8.78	
Propyl Benzene	120.20	0.862	159	8.72	
Propylene	42.08	gas		8.73	
Propylene Oxide	58.08	0.859	34	10.22	

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{O}C)*	I.P. (EV)**	TWA (PPM)***
Methyl Propyl Ketone	86.13	0.809	100.01	9.38	
2-Methyl Styrene	165.4	1.068	131	10.07	
Monomethyl Aniline	107.16	0.989			
Monomethyl Hydrazine	46.1	0.87			
Morpholine	87.1	1.01	129	8.88	20
Napthalene	93.7	1.16	217.7	8.12	10
Propyl Ether	102.17	0.7360	88.90	9.27	
Propyl Formate	88.10	0.901		10.54	
Pyrene	202.3	gas		7.41	
Pyridine	79.1	0.98	115	9.32	5
Pyrrole	67.09	0.9691	131	8.20	
Styrene	104.14	9.9059	145-146	8.47	
Styrene Oxide	120.2	1.054	194	9.04	
Tetrachloroethylene	165.9	1.63	121	9.32	100
Tetrahydrofuran	72.10	0.8892	67	9.54	
Tetrahydropyran	86.13	0.8814	88	9.26	
Thiophene	84.1	1.53	84	8.86	
Toluene	93.13	0.866	111	8.82	
O-Toluidine	107.2	1.01	199-200	7.44	5
Trichloroethene	131.40	1.4649	87	9.45	
Triethylamine	101.19	1.069	88.18	7.50	
Trimethyl Amine	59.11	0.636	3-4	7.82	
2,2,4-Trimethyl Pentane	114.23	0.692	98-99	9.86	
Tripropyl Amine	143.27	0.753	155-58	7.23	
Valeraldehyder	86.13	0.8095	103	9.82	
Valeric Acid	102.13	0.939	185	10.12	
Vinyl Acetate	118	0.94	72-73	9.19	10
Vinyl Bromide	106.96	1.517	16	9.80	
Vinyl Chloride	62.5	gas		10.00	1
Water	18.016	1.00	100	12.59	
M-Xylene	106.16	0.8684	138-39	8.56	100
O-Xylene	106.16	0.8801	143-45	8.56	100
P-Xylene	106.16	0.8614	138	8.45	100

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

APPENDIX G

DILUTION PROBE OPERATION

(Not investigated as part of UL classified product)

G.1 GENERAL

The dilution probe is constructed of stainless steel and Teflon, with a charcoal filter mounted on the dilution inlet. The purpose of the charcoal filter is to provide hydrocarbon free air to the probe assembly so that the dilution of the incoming sample is not affected by the dilution air. The charcoal filter should be changed every 3 months to ensure proper operation, eliminating the problem of hydrocarbon breakthrough. It is easy to evaluate the performance of the charcoal, by challenging the Model 580A with hydrocarbon free air, then introducing a standard through the charcoal filter, with the inlet of the probe plugged. If there is breakthrough, a reading other than zero will be observed on the readout.

Another important part of the dilution probe is the 10 micron filter that is placed in the inlet of the probe assembly. The flow through this may reduce with time, as dirt collects on the inlet filter. This filter should be changed on a regular basis, depending upon the operator's experience and the environment in which he is working.

It is important to realize that the charcoal filter is not a totally efficient device. This does not cause a problem with the 580A, however, because the photoionization detector does not respond to ethane or methane.

TECHNICAL CONSIDERATION

Need For Dilution - The Model 580B dilution system was developed to increase the dynamic range of the Model 580A. As the instrument is manufactured, it has a workable range of 0 to 2,000 ppm. Above this upper limit, the detector is found to be non-linear. It does not absorb ethane or methane. The 580B will "lock out" for concentrations above 2,000 ppm. To meet the requirements of fugitive emission measurements as defined in EPA Method 21, there is the need to make measurements above the 2,000 ppm level. To accomplish this using a detector system that is limited by linearity, a dilution probe was developed. this probe provides a nominal 10 to 1 dilution ratio, increasing the dynamic range of the Model 580B from 2,000 to 20,000 ppm.

G.3 CALIBRATION OF THE DILUTION PROBE

The dilution probe is not factory calibrated. It has been tested and evaluated proper performance. It is the responsibility of the operator to properly calibrate the dilution probe.

The following is a simple procedure for this activity:

1. The performance of the 580B should have previously seen verified and calibrated.

2. Place the 580B in close proximity to a standard with the appropriate range. For example, if the instrument is to be used in a 5,000 ppm sampling range, then a standard of that concentration should be selected.

3. Connect the dilution probe making sure that the charcoal filter and the 10 micron filter are in place to the front of the 580B.

4. Challenge the instrument with the new standard gas and adjust the micro metering valve until a tenth of the reading is seen on the instrument readout.

5. As in the example of the 5,000 ppm standard, 500 ppm should be seen on the readout.

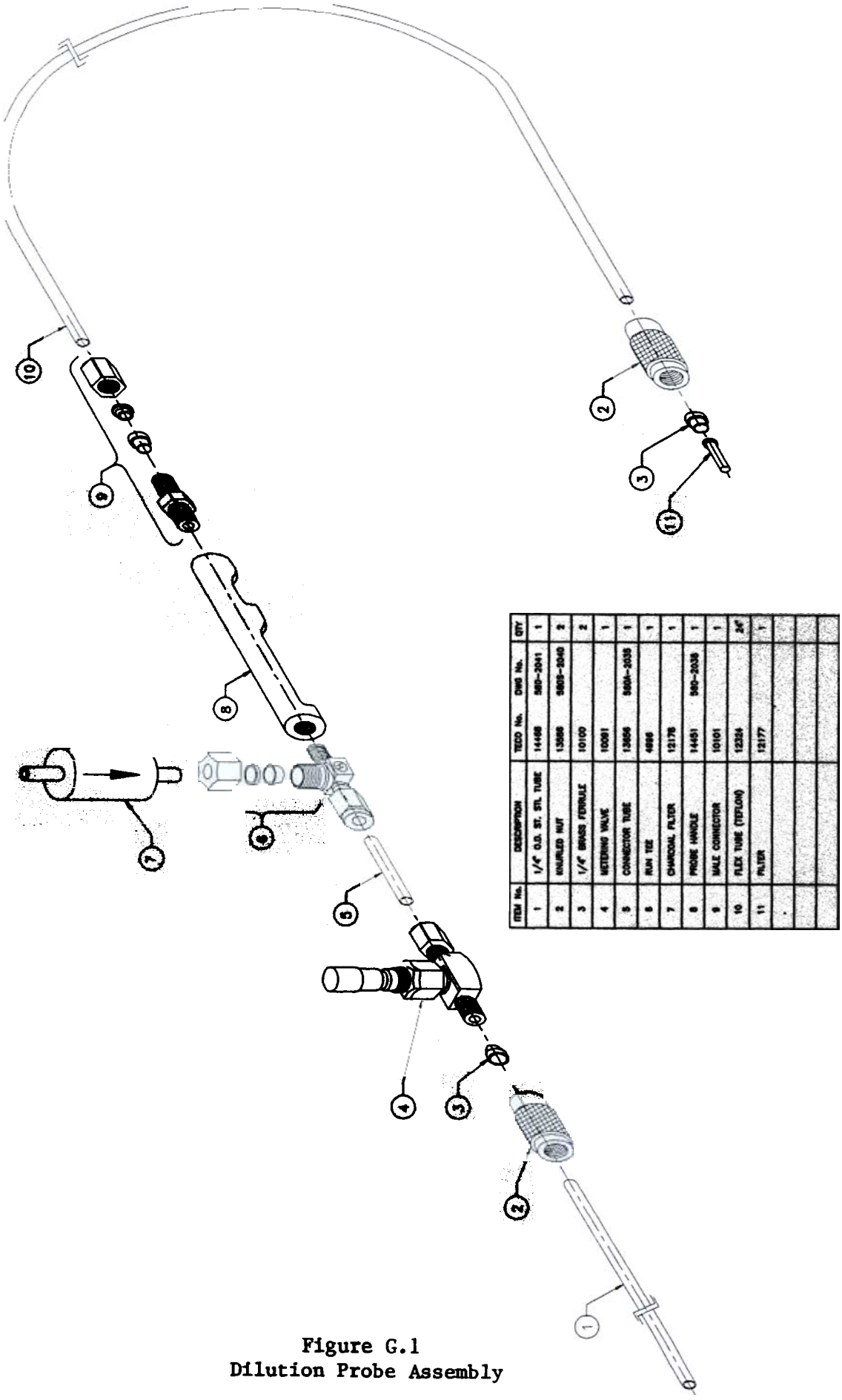
6. This is all that is required to calibrate the 580A or 580B.

   Note: It is important that both zero and span of of the 580A have been properly verified prior to initiating this procedure. It is very simple after using the dilution probe, to remove it and recheck the performance of the instrument on your low concentrator standard.

7. It should be noted that due to the environment that you are operating in, there may be a change in the back pressure of the charcoal filter and the 10 micron filter. Any changes in these over a period of time will cause a change in the split ratio of the dilution probe. Therefore, it is important to calibrate the dilution probe as regularly as you calibrate the 580A or 580B.

REPLACEMENT PARTS

1.	Inlet Probe Assembly	580A-6016
2.	Charcoal Filter	3150-0018
3.	Inlet Filter 10 Micron	3150-0017



ITEM No.	DESCRIPTION	TECO No.	DWG No.	QTY
1	1/4" O.D. ST. STL TUBE	14468	560-2041	1
2	KNURLED NUT	13588	560A-2040	2
3	1/4" BRASS FERRULE	10100		2
4	METERING VALVE	10091		1
5	CONNECTOR TUBE	13656	560A-2035	1
6	RUN TEE	4886		1
7	CHARCOAL FILTER	12178		1
8	PROBE HANDLE	14451	560-2039	1
9	MALE CONNECTOR	10101		1
10	FLEX TUBE (TEFLON)	12324		24'
11	FILTER	12177		1

Figure G.1
Dilution Probe Assembly

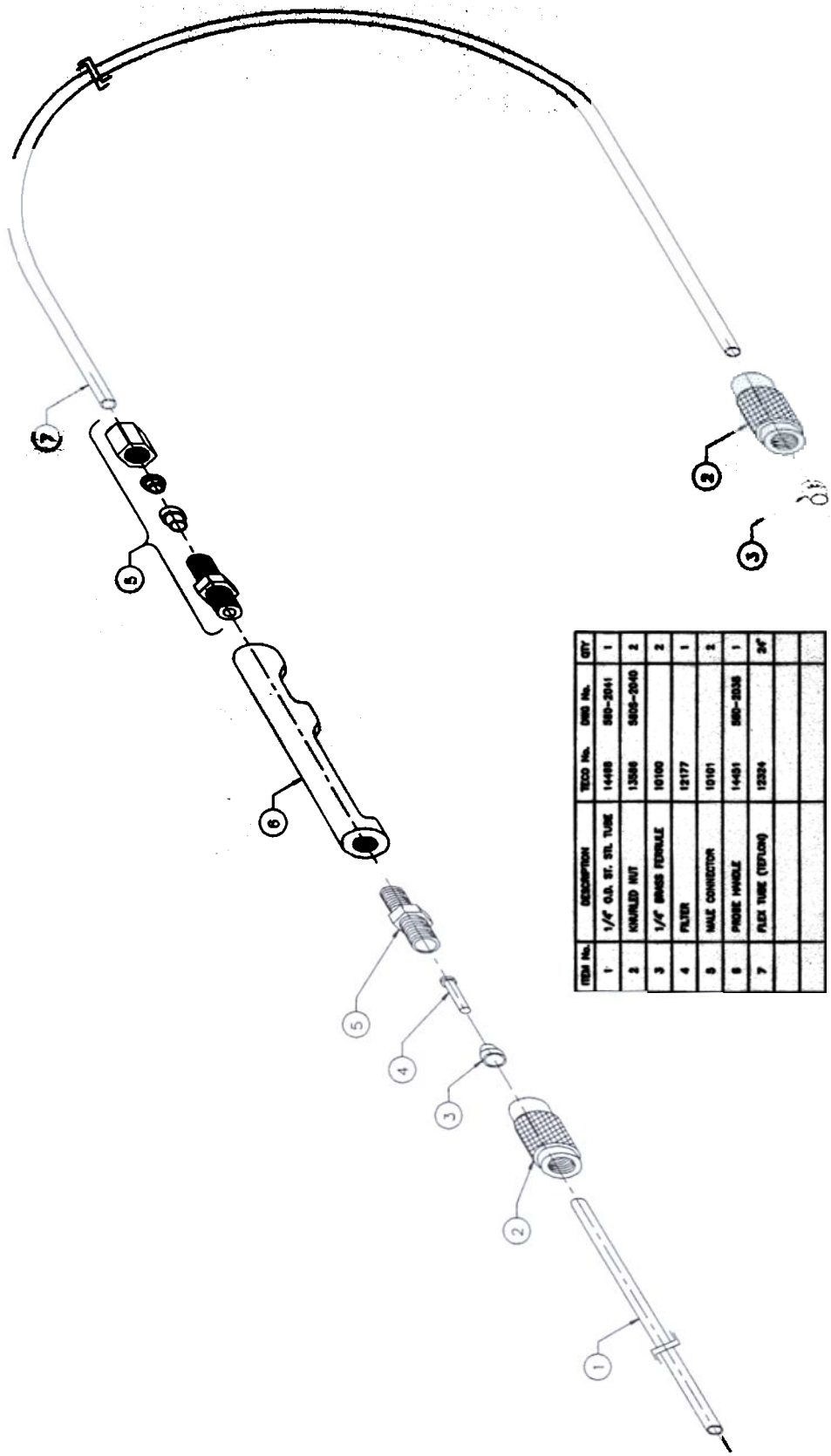


Figure H.1
Probe Extension Assembly

APPENDIX I

INSTRUCTION SHEET
OPTION 11 WATER TRAP ASSEMBLY

(Not investigated as part of the UL classified product)

INSTALLATION

The water trap assembly (16846) is to be installed on the end of the 580 sample probe. The tygon tubing included is to be placed on the syringe side of the filter then connected to the probe. Refer to the drawing below.

USE

The water trap will effectively stop water from entering the instrument. Water traps can be re-used after drying at room temperature, but the user must be cautious of possible contamination. Contaminated traps should be discarded.

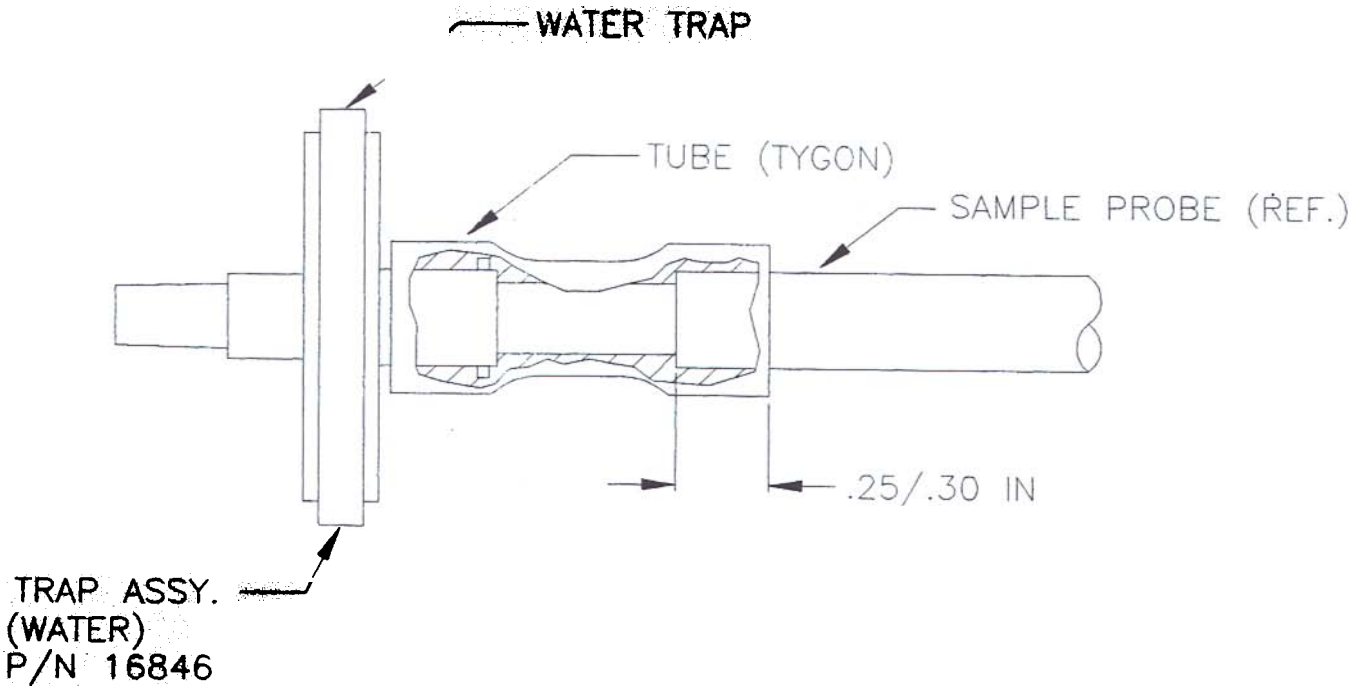


Figure I.1
Water Trap Assembly

APPENDIX J

Revised documents having an earlier revision level to the existing manual documents.

B-1	Processor Circuit	580S-9002	Sht. 1
B-2	Processor Circuit	580S-9002	Sht. 2
B-3	Memory Circuit	580S-9007	
C-1, 2, 3	Processor Board Bill of Materials	580S-6008	
	Processor Board Silkscreen	580S-6008	
	Memory Board Bill of Materials	580S-6007	
	Memory Board Silkscreen	580S-6007	

APPENDIX K

RS-232 Information Sheet

This procedure will retrieve logged data from Thermo Environmental Instruments' Model 580, and download the data into the HyperTerminal program for Windows 95 or Windows NT.

Hardware needed:

- Model 580
- Personal computer running either Win95 or WinNT with an available free serial communication port.
- RS-232 cable supplied with the Model 580

Software needed:

- HyperTerminal (supplied with Win95 and WinNT)

NOTE:
Even though this document uses HyperTerminal as the communication package, it is not necessary to use this program. HyperTerminal was chosen because it is part of the Win95 and WinNT software packages and is therefore readily available to users of these operating systems. Any communication program can be used to retrieve data from the Model 580 as long as it allows direct connections to a free communication port.

Procedure for setting up HyperTerminal for Win95 and WinNT

Click on the **HyperTerminal** folder within the **Programs/Accessories** folder in the start menu
Double Click on the icon called "HYPERTRM" or "HYPERTRM.EXE". This will execute the program and a dialog box called Connection Description should appear. In this dialog box it should say New Connection. In the edit box labeled **Name** enter *Model 580.* Then click **OK.**
Another dialog box called Phone Number should appear. The only edit box that needs to be changed is labeled **Connect Using.** From the list, selected by clicking on down arrow on the side of the edit box, pick from the *Direct to Comm* choices, the communications port you are planning to connect the Model 580 to. Then click **OK.**
A dialog box called Comm # Properties (# being the communications port you selected) should appear. These are the communications settings that should be set for communications with the Model 580.

Bits per second	9600	(Also called BAUD RATE)
Data bits	8	
Stop bits	1	
Flow Control	NONE	(Also called handshaking)

Once the above properties have been entered and set into the appropriate edit boxes, click **OK**
At this point you should have the main HyperTerminal program window on the screen. On the left side of the status bar which appears at the bottom of the screen, it should read "connected" with a timer counting up.
From the **FILE** menu select **SAVE.** This will save the properties you just entered for later use. The file will be named *Model 580* or *Model 580.ht* and can be used to start HyperTerminal by just double clicking on this file in the HyperTerminal folder.

Procedure for setting up Thermo Environmental Instruments' Model 580

To setup the Model 580 to communicate with the HyperTerminal or any other communications program see section 2.7.4 in the Manual. The Model 580 should be set to use *PRINTER FORMAT* with the baud rate set to *9600*. A basic understanding of the Model 580's menu system will be helpful.

To transfer data between HyperTerminal and the Model 580

HyperTerminal should be setup and the Model 580 should be set up and running. Before transferring the data, HyperTerminal or the communications program you are using should be set to capture the text being transmitted by the model 580.

To turn on the capture text feature in HyperTerminal

1. Select **CAPTURE TEXT** from the **TRANSFER** menu. A dialog box will appear asking for a file name to which the data will be written.
2. Enter the directory and filename in the edit box and then click **START**. The capture file is now open and ready to receive data.
3. Use the **BROWSE** button to open the appropriate file and directory into which you wish the captured data to be saved. Using the **BROWSE** feature only allows you to open an existing file and it will not let you create a new file.

Connect the communication cable between the selected communication port on the computer and the Model 580.
Once the Model 580 is setup and connected select Output Logged Data on the Model 580 and data should appear in the HyperTerminal Window. This is the logged data and even though it may scroll off screen all the data is being captured into the above capture file described above.
Once the data is transferred, close the capture text file. This is done in HyperTerminal by selecting **STOP** from the **TRANSFER/CAPTURE TEXT** menu.
This capture text file can now be printed using any text editor or imported into a spread sheet program for data analysis.

Thermo Environmental Instruments provides spare parts and servicing from the following locations:

Thermo Environmental Instruments Inc.
8 West Forge Parkway
Franklin, Massachusetts 02038
Telephone: (508) 520-0430
Facsimile: (508) 520-1460

Thermo Environmental Instruments Inc.
325 E. Arrow Hwy. #506
San Dimas, CA 91773
Telephone: (909) 394-2373
Facsimile: (909) 394-2367

Thermo Environmental Instruments has additional service personnel located throughout the country. Contact either service center for more information.

APPENDIX D
VALIDATION SOPS AND CHECKLISTS

CHEMIST REVIEW-VALIDATION CHECKLIST

Project: Olin Chemical Superfund Site **Method: _____**
Project #: _____ **Laboratory and SDG: _____**
Date: _____ **Reviewer: _____**

□ **Chemist Review** □ **Full Validation (add page 2)**

1. **Case Narrative and Data Package Completeness (COC Review)**

2. **Holding Time and Sample Preservation/Collection**

3. **QC Blanks**

4. **Laboratory Control Sample Review**

5. **Field Duplicate Precision**

6. **Lab Duplicate Precision**

7. **Matrix Spike Results (if applicable)**

8. **Surrogate Recovery (if applicable)**

9. **Internal Standard Recovery (if applicable)**

CHEMIST REVIEW-VALIDATION CHECKLIST

FULL VALIDATION CHECKS

Ten Percent of data sets will have full validation checks completed during chemist review.

☐ **Initial Calibration**

☐ **Continuing Calibration Verification**

<u>Transcription and Calculation Checks</u>

☐ **Instrument Calibration**

☐ **Blank Review – raw data/chromatogram check**

☐ **Laboratory Control Sample**

☐ **Matrix Spike**

☐ **Field Sample Results**

☐ **Surrogate Recovery**

☐

☐

INORGANICS

TAL METALS (including mercury)

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifiers: Region I Guidelines (11/08)

TIER I / II / III (circle one)

SITE: **Olin Chemical** Project #:_____ SDG #:_____

LAB # :_____

Sample IDs: Attached tracking sheet or sample listing.

This checklist is designed to be used with the USEPA Data Validation Guidelines Part IV (November 2008). During Level III validation, calculation and transcription checks are completed for instrument tuning, surrogates, target compounds, spike recoveries, calibration data, and internal standards as specified in the guideline. These checks are documented on attached validation notes.

YES NO NA	
Data completeness	
☐ ☐ ☐ All data summaries, QC forms and raw data available from hard copy or electronic data package	Contact lab if missing data. Lab to respond with 24 hours.
☐ ☐ ☐ Data summaries match EDD	
Holding Times and Preservation	
☐ ☐ ☐ Hold times met (6 months, 28 days Hg)	
☐ ☐ ☐ Preserved (waters HNO_3)	
Calibration	
☐ ☐ ☐ ICP/MS Instrument Tune.	Verify that tuning meets mass, resolution, and RSD method criteria.
☐ ☐ ☐ Appropriate number of standards used to establish calibration curve.	ICP: at least one blank and one standard Hg: at least one blank and four standards
☐ ☐ ☐ Correlation coefficient > 0.995 for Hg	Correlation coefficient criteria applicable to all analyses except ICP-AES.
☐ ☐ ☐ Calibrated daily.	
☐ ☐ ☐ ICV/CCV %R within acceptance range.	90-110% for ICP-AES/MS, 80-120% for Hg. See additional qualification actions in the Region 1 guidelines.
☐ ☐ ☐ CCVs analyzed at the proper frequency.	Every 10 samples or every 2 hrs.
☐ ☐ ☐ QL Standard within limits	70-130% for QL Standard. If out low, (J) detects less then 2X QL standard and (UJ) non-detects. See additional validation actions in the Region I guidelines.

INORGANICS

TAL METALS (including mercury)

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifiers: Region I Guidelines (11/08)

TIER I / II / III (circle one)

Blanks Method: ☐ ☐ ☐ Method blank was prepared with each batch of samples or with a maximum of 20 samples ☐ ☐ ☐ Results >MDL ☐ ☐ ☐ Absolute value negative MB results > 5x MDL ☐ ☐ ☐ For ICP/MS verify IS responses meet method criteria	Evaluate all blanks for contamination. Highest contaminant level used for action level. 5X the highest blank contamination is the action level.
Calibration Blanks: ☐ ☐ ☐ ICB/CCB results > IDL ☐ ☐ ☐ Absolute value of negative ICB/CCB results > 5x MDL ☐ ☐ ☐ CCB analyzed every 10 samples or 2 hrs. Equipment/Rinseate Blanks: ☐ ☐ ☐ Results >MDL ☐ ☐ ☐ Absolute value of negative ICB/CCB results > 5x MDL	
Interference Check Sample ☐ ☐ ☐ ICS analyzed at proper frequency ☐ ☐ ☐ Interference present in sample at > 50% concentration in ICS ☐ ☐ ☐ ICS AB %R 80%-120% ICP-MS Internal Standard Intensities ☐ ☐ ☐ Internal standard relative intensities reported by the laboratory ☐ ☐ ☐ Internal standard relative intensities are within 60 – 125 %	An ICS must be run at the beginning and end of run or every 8 hours. If interferences (Al, Ca, Fe, Mg) are not > 50% ICS concentration in sample, do not apply. Qualify data based on Region 1 guideline
Matrix Spikes ☐ ☐ ☐ All compounds are within %R of 75-125% excluding results exceeding the spike concentration by ≥4x ☐ ☐ ☐ Were post-digestion spikes reported for unacceptable pre-digestion spike recoveries ☐ ☐ ☐ Was a field blank used for spike analysis	Post-digestion spikes %R limits = 75% - 125%

INORGANICS

TAL METALS (including mercury)

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifiers: Region I Guidelines (11/08)

TIER I / II / III (circle one)

Laboratory Control Samples (LCS) ☐ ☐ ☐ Percent recoveries are within limits (waters and soil 80-120%) ☐ ☐ ☐ An LCS was analyzed for each matrix, batch of samples, or every 20 samples.	
Laboratory Duplicate ☐ ☐ ☐ Was a field blank used as the lab duplicate ☐ ☐ ☐ Is the RPD within water control limits of ±20% for sample values >5x RL(35% for soil) ☐ ☐ ☐ Is the control limit of ± RL met for sample values <5x RL (2x RL for soil) ☐ ☐ ☐ Was a duplicate analyzed for every matrix and every 20 samples or batch	
Field Duplicate ☐ ☐ ☐ For sample values >5x RL, the RPD control limit of ± 30% (50% for soil) was met ☐ ☐ ☐ For sample values <5x RL, the control limit of ±2x RL (4x RL for soil) was met	
Serial Dilution ☐ ☐ ☐ Are any percent difference criteria > 15% (for samples with a concentration >50 times the IDL) ☐ ☐ ☐ Are results of the diluted samples > the original sample results	

Validator's Signature:_____

Date:_____

Reference:

MACTEC, Project Operation Plan Volume IIB, Quality Assurance Project Plan for Remedial Investigation/Feasibility Study – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting. .April 2009.

1.0 Laboratory Deliverable Requirements

1.1 Laboratory Information: Was all of the following provided in the laboratory report? Check items received. **Yes [__]** No [__] N/A [__] Comments:

☐ *Name of Laboratory* ☐ *Address* ☐ *Project ID* ☐ *Phone #* ☐ *Sample identification – Field and Laboratory*

Client Information: ☐ *Name* ☐ *Address* ☐ *Client Contact* *(IDs must be cross-referenced)*

☐ *Data Package Narative* ☐ *Results and QC Summaries* ☐ Raw Data ☐ Chromatograms

ACTION: If no, contact lab for submission of missing or illegible information.

1.1 Laboratory Case Narrative: ***Yes [__]*** *No [__]* *N/A [__]* *Comments:*

☐ *Narrative serves as an exception report for the project and method QA/QC performance.* ☐ *Narrative includes an explanation of each discrepancy on the Certification Statement.*

***ACTION**: If no, contact lab for submission of missing or illegible information.*

1.2 Chain of Custody (COC) copy present of completed COC? **Yes [__]** No [__] N/A [__] Comments:

Does the laboratory report include a copy of the completed Chain of Custody forms containing all samples in this SDG?

ACTION: If no, contact lab for submission of missing completed *COC*.

1.3 Sample Receipt Information (*Cooler Receipt Form*): Were each of the following tasks completed and recorded upon receipt of the sample(s) into the laboratory? **Yes [__]** No [__] N/A [__] Comments:

☐ Sample temperature confirmed: must be 1° – 10° C. (If samples were sent by courier and delivered on the same day as collection, temperature requirement does not apply).

☐ Container type noted ☐ Condition observed ☐ Field and lab IDs cross referenced

ACTION: If no, contact lab for submission of missing or incomplete documentation.

1.3.1 Were the correct bottles and preservatives used? **Yes [__]** No [__] N/A [__] Comments:

Soil - 8 oz soil jar/cool to $4^{o}C$

ACTION: If no, inform senior chemist. Document justification for change in container/volume (if applicable), qualify positive and non-detect data (J) if cooler temperature exceeds 10°C. Rejection of data requires professional judgment.

1.3.2 Were all samples delivered to the laboratory without breakage?

Yes [__] No [__] N/A [__] Comments:

1.3.3 Does the *Cooler Receipt Form* or Lab Narrative indicate other problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? Yes [__] **No [__]** N/A [__] Comments:

1.4 Sample Results Section: *Was the following information supplied in the laboratory report for each sample?* **Yes [__]** No [__] N/A [__] Comments:

☐ Field ID and Lab ID ☐ *Date and time collected* ☐ Analyst Initials ☐ *Dilution Factor* ☐ % moisture or solids ☐ *Reporting limits* ☐ *Clean-up method* ☐ Analysis method ☐ *Preparation method* ☐ *Date of preparation/extraction/ and analysis,* ☐ Matrix

☐ Target analytes and concentrations ☐ Units (soils must be reported in dry weight)

ACTION: If no, contact lab for submission of missing or incomplete information.

1.5 QA/QC Information: Was the following information provided in the laboratory report for each sample batch? **Yes [__]** No [__] N/A [__] Comments:

☐ Method blank results ☐ LCS recoveries ☐ MS/MSD recoveries and RPDs ☐ Surrogate recoveries

ACTION: If no, contact lab for submission of missing or incomplete information.

2.0 Holding Times Yes [__] **No [__]** N/A [__] Comments:

Have any technical holding times, determined from date of collection to date of analysis, been exceeded?

NOTE: For water samples, the holding time is 14 days from sampling to extraction and 30 days from extraction to analysis.

ACTION: If technical holding times are exceeded, qualify all positive results (J) and non-detects (UJ). For water samples that are grossly exceeded (>2X hold time) reject (R) all non-detect results. For soil samples professional judgement will be used to determine if rejection is necessary.

3.0 Laboratory Method **Yes [__]** No [__] N/A [__] Comments:

3.1 Was the correct laboratory method used?
Soil Extraction *3546* or 3550B
N-nitrosodimethylamine mod 8270

ACTION: If no, contact project manager to inform Client of change; request variance from Client; contact laboratory to provide justification for method change compared to the requested method.

3.2 Are the practical quantitation limits the same as those specified by the method? **Yes [__]** No [__] N/A [__] Comments:

NOTE: *The method specifies a reporting limit (RL) of 5.0 µg/kg. Results between the MDL and RL are qualified estimated J..*

ACTION: If no, evaluate change with respect to sample matrix, preparation, dilution, moisture, etc. If sample MDL is indeterminate, contact lab for explanation.

3.3 Did the laboratory complete a mulit-point initial calibration with a RPD ≤ 25? Is the low point standard ≤ 0.5 µg/L? **Yes [__]** No [__] Comments:

Has a second source standard or spike been analyzed to verify working standards? **Yes [__]** No [__]

3.4 Did the laboratory analyze a continuing calibration every 12 hours or every 20 samples? Was the percent difference ≤ 30? **Yes [__]** No [__]

ACTION: If no, contact the lab for submission.

4.0 **Method Blanks**

4.1 Is the Method Blank Summary present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, call the laboratory for submission of missing data.

4.2 For the analysis of NDMA, has a method blank been analyzed for each analysis batch of field samples of 20 or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, document discrepancy in case narrative and contact lab for justification. Consult senior chemist for action needed.

4.3 Is the method blank less than the PQL? **Yes [__]** No [__] N/A [__] Comments:

4.4 Do any method blanks have positive results for NDMA parameters? Qualify data according to the following: Yes [__] **No [__]** N/A [__] Comments:

For NDMA contaminants:

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

5.0 Laboratory Control Sample

5.1 Was a laboratory control sample run with each analytical batch of 20 samples or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: Call laboratory for LCS form submittal. If data are not available, use professional judgment to determine the usability of sample results associated with that batch.

5.2 Is a LCS Summary Form present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact lab for resubmission of missing data.

5.3 Is the recovery of any analyte outside of control limits? Yes [__] **No [__]** N/A [__] Comments:

NOTE: LCS recovery limits 50-150% (MACTEC QAPP, 2009).

ACTION: If recovery is above the upper limit, qualify all positive sample results within the batch as (J). If recovery is below the lower limit but > 10%, qualify all positive and no-detect results within the batch as (J). If LCS recovery is <10%, non-detect results are rejected (R).

6.0 Matrix Spikes

6.1 Were project specified MS/MSDs analyzed? List project samples that were spiked. **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact senior chemist to see if any were specified.

6.2 Is the MS/MSD recovery form present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact lab for resubmission of missing data.

6.3 Were matrix spikes analyzed at the required frequency of 1 per 20 samples per matrix? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If any matrix spike data are missing, call lab for resubmission.

6.4 Are any NDMA spike recoveries outside of the QC limits? Yes [__] **No [__]** N/A [__] Comments:

NOTE: $\%R = \frac{(SSR-SR)}{SA} \times 100\%$ Where: SSR = Spiked sample result
SR = Sample result
SA = Spike added

NOTE: *MS/MSD recovery limits as 50-150.*

NOTES: 1) Use professional judgment for the MS/MSD flags.

2) If the MS/MSD was performed by the laboratory on a non-project sample, no qualification is required.

ACTION: MS/MSD flags are applied to samples based on professional judgement. If the recoveries of the MS and MSD exceed the upper control limit, qualify positive results as estimated (J). If the recoveries of the MS and MSD are lower than the lower control limit, qualify both positive results and non-detects (J).

6.5 Are any RPDs for MS/MSD recoveries outside of the QC limits? Yes [__] **No [__]** N/A [__] Comments:

NOTE: RPD = $\frac{S-D}{(S+D)/2}$ x 100% Where: S = MS sample result
D = MSD sample result

NOTE: *MS/MSD RPD limits for soil $\leq$ 50.*

ACTION: If the RPD exceeds the control limit, qualify positive results and non-detects (J).

7.0 Surrogate Recoveries

Is the surrogate recovery outside of limits for any sample or method blank? Yes [__] **No [__]** N/A [__] Comments:

NOTE: %R = Q/D x 100% Where: Q = sample surrogate result
D = surrogate concentration

NOTE: *QAPP (MACTEC, 2008) surrogate limits for waters 50-150%.*

ACTION: If recoveries are >10%: (1) For recoveries below the QC limit, qualify non-detects and positives (J), and (2) For recoveries above the QC limit, qualify only positives (J). If any surrogate recovery is <10% (unless the lab QC limits are below 10%, in which case, results are flagged as stated above), flag positives (J) and reject non-detects (R).

8.0 Sampling Accuracy

8.1 Were rinsate blanks collected? Prior to evaluating rinsate blanks, obtain a list of the associated samples from the project chemist. **Yes [__]** No [__] N/A [__] Comments:

8.2 Do any rinsate blanks have positive results? Yes [__] **No [__]** N/A [__] Comments:

NOTE:

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

9.0 Field Duplicates

9.1 Were field duplicate samples collected? Obtain a list of the samples and their associated field duplicates. **Yes [__]** No [__] N/A [__] Comments:

9.2 Was the RPD ≤ 50% for soils? **Yes [__]** No [__] N/A [__] Comments:

ACTION: RPD must be ≤50% for soil. Qualify data (J) for both sample results if the RPD exceeds 50%.

10.0 Calculation and Transcript Checks

- ☐ Initial Calibration
- ☐ Continuing Calibration
- ☐ Method Blank Raw Data Review
- ☐ NDMA Sample Result
- ☐ LCS
- ☐ Surogate Recovery

REFERENCES:

MACTEC, 2009. Project Operation Plan Volume IIIB Quality Assurance Plan – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting..April 2009.

U.S. Environmental Protection Agency (USEPA), 1996. "Region 1 EPA-NE Data Validation Guidelines For Evaluating Environmental Analyses"; Quality Assurance Unit Staff; Office of Environmental Measurement and Evaluation; December 1996

MADEP, 2002. E-mail response dated November 13, 2002 from Oscar C. Pancorbo, Ph.D, Director, Sen. W.X. Wall Experiment Station, Massachusetts Dept. Environmental Protection and Christopher Pyott, Massachusetts Dept. of Environmental Protection, Northeast Regional Office.

OLIN-WILMINGTON
CHEMIST REVIEW
STANDARD OPERATING PROCEDURE AND CHECKLIST
N-NITROSODIMETHYLAMINE (NDMA) AND
N-NITROSODI-N-PROPYLAMINE (NDPA) BY METHOD 521

Reviewer/Date_____
Sr. Review/Date_____
Lab Report # _____
Project _____

1.0 Laboratory Deliverable Requirements

1.1 Laboratory Information: Was all of the following provided in the laboratory report? Check items received. **Yes [__]** No [__] N/A [__] Comments:

☐ *Name of Laboratory* ☐ *Address* ☐ *Project ID* ☐ *Phone #* ☐ *Sample identification – Field and Laboratory*

Client Information: ☐ *Name* ☐ *Address* ☐ *Client Contact* *(IDs must be cross-referenced)*

☐ Data Package Narrative ☐ results and QC summaries ☐ raw data ☐ chromatograms

ACTION: If no, contact lab for submission of missing or illegible information.

1.1 Laboratory Case Narrative:

Yes [__] *No [__]* *N/A [__]* *Comments:*

☐ *Narrative serves as an exception report for the project and method QA/QC performance.*

***ACTION**: If no, contact lab for submission of missing or illegible information.*

1.2 Chain of Custody (COC) copy present of completed COC? **Yes [__]** No [__] N/A [__] Comments:

Does the laboratory report include a copy of the completed Chain of Custody forms containing all samples in this SDG?

ACTION: If no, contact lab for submission of missing completed *COC*.

1.3 Sample Receipt Information (*Cooler Receipt Form*): Were each of the following tasks completed and recorded upon receipt of the sample(s) into the laboratory? **Yes [__]** No [__] N/A [__] Comments:

☐ Sample temperature confirmed: must be 1° – 10° C. (If samples were sent by courier and delivered on the same day as collection, temperature requirement does not apply).

☐ Container type noted ☐ Condition observed ☐ Field and lab IDs cross referenced documentation. **ACTION**: If no, contact lab for submission of missing or incomplete documentation.

1.3.1 Were the correct bottles and preservatives used? **Yes [__]** No [__] N/A [__] Comments:

Water - 1 Liter amber bottle cool to 4°C. Sodium thiosulfate may be added if source is chlorinated.

ACTION: If no, inform senior chemist. Document justification for change in container/volume (if applicable), qualify positive and non-detect data (J) if cooler temperature exceeds 10°C or use professional judgment for data rejection.

1.3.2 Were all samples delivered to the laboratory without breakage? **Yes [__]** No [__] N/A [__] Comments:

1.3.3 Does the *Cooler Receipt Form* or Lab Narrative indicate other problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? Yes [__] **No [__]** N/A [__] Comments:

1.4 Sample Results Section: *Was the following information supplied in the laboratory report for each sample?* **Yes [__]** No [__] N/A [__] Comments:

☐ Field ID and Lab ID ☐ *Date and time collected* ☐ Analyst Initials ☐ *Dilution Factor* ☐ *Reporting limits*
☐ Analysis method ☐ *Preparation method* ☐ *Date of preparation/extraction/ and analysis,* ☐ Matrix ☐ Target analytes and concentrations ☐ Units

ACTION: If no, contact lab for submission of missing or incomplete information.

1.5 QA/QC Information: Was the following information provided in the laboratory report for each sample batch?

Yes [__] No [__] Comments:

☐ Method blank results ☐ LCS recoveries ☐ MS/MSD recoveries and RPDs ☐ Internal Standard Recoveries

ACTION: If no, contact lab for submission of missing or incomplete information.

2.0 <u>Holding Times</u> Yes [__] **No [__]** N/A [__] Comments:

Have any technical holding times, determined from date of collection to date of analysis, been exceeded?

NOTE: For water samples, the holding time is 7 days from sampling to extraction and 40 days from extraction to analysis.

ACTION: If technical holding times are exceeded, qualify all positive results (J) and non-detects (UJ). For water samples that are grossly exceeded (>2X hold time) reject (R) all non-detect results. For soil samples professional judgement will be used to determine if rejection is necessary.

3.0 Laboratory Method **Yes [__]** No [__] N/A [__] Comments:

3.1 Was the correct laboratory method used?

Water Extraction 3510C or *3520C*
NDMA and NDPA 521

ACTION: If no, contact project manager to inform Client of change; request variance from Client; contact laboratory to provide justification for method change compared to the requested method.

3.2 Are the practical quantitation limits the same as those specified by the ☐ QAPP **Yes [__]** No [__] N/A [__] Comments:

NOTE: *The project PQL is 2 ng/L for GW and 5 ng/L for SW.*

ACTION: If no, evaluate change with respect to sample matrix, preparation, dilution, moisture, etc. If sample PQL is indeterminate, contact lab for explanation.

3.3 Did the laboratory complete a mulit-point initial calibration with a NDMA RPD ≤ 20? Is NDPA RPD ≤ 30? Is the low point standard equal to the PQL? **Yes [__]** No [__] Comments:

Has a second source standard been analyzed to verify initial calibration? Is the percent difference < 25? **Yes [__]** No [__]

3.4 Did the laboratory analyze a continuing calibration every 12 hours or every 20 samples? Was the NDMA recovery 80-120 percent (percent difference ≤ 20)? Was the NDPA recovery 70 -130 percent (percent difference ≤ 30)? **Yes [__]** No [__]

3.5 Is the RRT in the CCAL ± 0.06 min from ICAL? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact the lab for submission.

4.0 **Method Blanks**

4.1 Is the Method Blank Summary present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, call the laboratory for submission of missing data.

4.2 For the analysis of NDMA, has a method blank been analyzed for each analysis batch of field samples of 20 or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, document discrepancy in case narrative and contact lab for justification. Consult senior chemist for action needed.

4.3 Is the method blank less than the PQL? **Yes [__]** No [__] N/A [__] Comments:

4.4 Do any method blanks have positive results for NDMA parameters? Qualify data according to the following: Yes [__] **No [__]** N/A [__] Comments:

For NDMA contaminants:

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

5.0 Laboratory Control Sample

5.1 Was a laboratory control sample extracted and run with each analytical batch of 20 samples or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: Call laboratory for LCS form submittal. If data are not available, use professional judgment to determine the usability of sample results associated with that batch.

5.2 Is a LCS Summary Form present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact lab for resubmission of missing data.

5.3 Is the recovery of any analyte outside of control limits?

Yes [__] **No [__]** N/A [__] Comments:

NOTE: QAPP LCS recovery limits 60-140.

ACTION: If recovery is above the upper limit, qualify all positive sample results within the batch as (J). If recovery is below the lower limit but > 10%, qualify all positive and no-detect results within the batch as (J). If LCS recovery is <10%, non-detect results are rejected (R).

6.0 Matrix Spikes

Matrix spikes may be collected at different frequencies based on monthly, quarterly, or task specific schedules. Confirm spike requirements for each set with the senior chemist.

6.1 Were project specified MS/MSDs analyzed? List project samples that were spiked.

Yes [__] No [__] N/A [__] Comments:

ACTION: If no, contact senior chemist to see if any were specified.

6.2 Is the MS/MSD recovery form present?

Yes [__] No [__] N/A [__] Comments:

ACTION: If no, contact lab for resubmission of missing data.

6.3 Were matrix spikes analyzed at the required frequency of 1 per 20 samples per matrix?

Yes [__] No [__] N/A [__] Comments:

ACTION: If any matrix spike data are missing, call lab for resubmission.

6.4 Are any NDMA spike recoveries outside of the QC limits? Yes [__] No [__] N/A [__] Comments:

NOTE: $\%R = \frac{(SSR-SR)}{SA} \times 100\%$

Where: SSR = Spiked sample result SA = Spike added
SR = Sample result

NOTE: *QAPP MS/MSD recovery limits are 60-140.*

NOTES: 1) Use professional judgment for the MS/MSD flags.
2) If the MS/MSD was performed by the laboratory on a non-project sample, no qualification is required.

ACTION: Professional judgment used to qualify associated samples. If the recoveries of the MS and MSD exceed the upper control limit, qualify positive results as estimated (J). If the recoveries of the MS and MSD are lower than the lower control limit, qualify both positive results and non-detects (J).

6.5 Are any RPDs for MS/MSD recoveries outside of the QC limits? Yes [__] **No [__]** N/A [__] Comments:

NOTE: $RPD = \frac{S-D}{(S+D)/2} \times 100\%$ Where: S = MS sample result
D = MSD sample result

NOTE: *QAPP MS/MSD RPD limits for water $\leq$ 20.*

ACTION: If the RPD exceeds the control limit, qualify positive results and non-detects (J).

7.0 Isotope IS Recoveries

Were internal recoveries outside of laboratory limits for any sample or method blank? Yes [__] **No [__]** N/A [__] Comments:

NOTE: *Lab IS recovery limits 25 -150.*

ACTION: If recoveries are >10% and sample extracts were not diluted, reject non-detects and qualify positive detections as estimated (J). For recoveries outside the lab QC limit, qualify non-detects and positives (J).

8.0 Sampling Accuracy

If ground water samples are collected directly from a tap, process stream, or with dedicated tubing, rinse blanks will not be collected.

8.1 Were rinsate blanks collected? Prior to evaluating rinsate blanks, obtain a list of the associated samples. **Yes [__]** No [__] N/A [__] Comments:

8.2 Do any rinsate blanks have positive results? Yes [__] **No [__]** N/A [__] Comments:

NOTE:

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

9.0 Field Duplicates

9.1 Were field duplicate samples collected? Obtain a list of the samples and their associated field duplicates. **Yes [__]** No [__] N/A [__] Comments:

9.2 Was the RPD ≤ 30% for waters? Calculate the RPD for all results and attach to this review. **Yes [__]** No [__] N/A [__] Comments:

ACTION: RPD must be ≤30% for water. Qualify data (J) for both sample results if the RPD exceeds 30%.

10.0 Calculation and Transcription Checks

- ☐ Initial Calibration
- ☐ Continuing Calibration
- ☐ Method Blank Raw Data Reveiw
- ☐ Sample NDMA and NDPA Results
- ☐ LCS
- ☐ Internal Standard Recovery

REFERENCES

MACTEC, 2009, "Project Operation Plan Volume III-B Quality Assurance Project Plan, Olin Wilmington Superfund Site, 51 Eames Street, Wilmington, MA", April 2009.

U.S. Environmental Protection Agency (USEPA), 1996. "Region 1 EPA-NE Data Validation Guidelines For Evaluating Environmental Analyses"; Quality Assurance Unit Staff; Office of Environmental Measurement and Evaluation; December 1996.

Note: The following analyses will be evaluated according to the "MADEP QA/QC Guidelines for **Sampling, Data Evaluation and Reporting Activities.**" MADEP, however, may not list QA/QC criteria for every chemical analysis. Where not defined by MADEP, criteria will default to values stipulated in the QAPP. Where the QAPP does not define criteria, QA/QC requirements will default to limits employed by the laboratory.

1.0 Laboratory Deliverable Requirements

1.1 Laboratory Information: Was all of the following provided in the laboratory report? **Yes [__]** No [__] N/A [__] Comments:
Check items received.

☐ *Name of Laboratory* ☐ *Address* ☐ *Project ID* ☐ *Phone #* ☐ *Sample identification – Field and Laboratory*

Client Information: ☐ *Name* ☐ *Address* ☐ *Client Contact* *(IDs must be cross-referenced)*

ACTION: If no, contact lab for submission of missing or illegible information.

1.2 Laboratory Report Certification Statement ***Yes [__]*** *No [__]* *N/A [__]* *Comments:*

Does the laboratory report include a completed Analytical Report Certification in the required format?

***ACTION**: If no, contact lab for submission of missing certification or certification with correct format.*

1.3 Laboratory Case Narrative: ***Yes [__]*** *No [__]* *N/A [__]* *Comments:*

☐ *Narrative serves as an exception report for the project and method QA/QC performance.*

☐ *Narrative includes an explanation of each discrepancy on the Certification Statement.*

***ACTION**: If no, contact lab for submission of missing or illegible information.*

1.4 Chain of Custody (COC) copy present with all documentation completed? **Yes [__]** No [__] N/A [__] Comments:

Does the laboratory report include copies of Chain of Custody forms containing all samples in this SDG?

NOTE: Olin receives and maintains the *original* COC.

ACTION: If no, contact lab for submission of copy of missing completed COC.

1.5 Sample Receipt Information (*Cooler Receipt Form*): Were each of the following tasks completed and recorded upon receipt of the sample(s) into the laboratory?

Yes [__] No [__] N/A [__] Comments:

☐ Sample temperature confirmed: must be 1° – 10° C. (If samples were sent by courier and delivered on the same day as collection, temperature requirement does not apply).

☐ Container type noted ☐ Condition observed ☐ pH verified (where applicable) ☐ Field and lab IDs cross referenced

ACTION: If no, contact lab for submission of missing or incomplete documentation.

1.5.1 Were the correct bottles and preservatives used?

Yes [__] No [__] N/A [__] Comments:

Ammonia,– 1 Liter polyethylene/H_2SO_4 to pH<2,cool to 4^oC

Alkalinity – 1 500mL polyethylene/cool to 4°C

Chemical Oxygen Demand – 250 mL polyethylene/H_2SO_4 to pH<2,cool to 4^oC

Chloride, pH, sulfate - 1 500mL polyethylene/cool to 4^oC

Organic Carbon – 500 mL amber glass bottle/HCl or H_2SO_4 to pH<2,cool to 4^oC

Sulfide – 50 mL polyethylene/ZnAcetate + NaOH to pH>9, cool to 4°C

Specific conductance, TDS, TSS – 100 mL polyethylene/cool to 4°C

TSS, TDS - 1 500mL polyethylene/cool to 4°C

ACTION: If no, inform senior chemist. Document justification for change in container/volume (if applicable), qualify positive and non-detect data (J) data if cooler temperature exceeds 10°C. Rejection of data requires professional judgment

1.5.2 Were all samples delivered to the laboratory without breakage?

Yes [__] No [__] N/A [__] Comments:

1.5.3 Does the *Cooler Receipt Form* or Lab Narrative indicate other problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data?

Yes [__] **No [__]** N/A [__] Comments:

1.6 Sample Results Section: *Was the follow ing information supplied in the laboratory report for each sample?* **Yes [__]** No [__] N/A [__] Comments:

☐ Field ID and Lab ID ☐ Date and time collected ☐ Analyst Initials ☐ Dilution Factor ☐ % moisture or solids ☐ Reporting limits
☐ *Clean-up method* ☐ Analysis method ☐ Preparation method ☐ Date of preparation/extraction/digestion clean-up and analysis, where applicable
☐ Matrix ☐ Target analytes and concentrations ☐ Units (soils must be reported in dry weight)

ACTION: If no, contact lab for submission of missing or incomplete information.

1.7 QA/QC Information: Was the following information provided in the laboratory report for each sample batch? **Yes [__]** No [__] N/A [__] Comments:

☐ Method blank results ☐ LCS recoveries ☐ MS/MSD recoveries and RPDs ☐ Laboratory duplicate results (where applicable)

ACTION: If no, contact lab for submission of missing or incomplete information.

2.0 <u>Holding Times</u> Yes [__] **No [__]** N/A __] Comments:

Have any technical holding times, determined from date of collection to date of analysis, been exceeded? The holding times are as follows:

28 days = ammonia, chemical oxygen demand, chloride, organic carbon, specific conductance, total organic carbon and sulfate

Alkalinity = 14 days Sulfide, TDS, TSS = 7 days pH = analyze immediately Nitrate nitrogen as N = 48 hrs

Nitrite nitrogen as N = 48 hrs

NOTE: List samples that exceed hold time with # of days exceeded on checklist

ACTION: If technical holding times are exceeded qualify results (J). For water samples that are grossly exceeded (>2X hold time) reject (R) all non-detect results. Professional judgment used to qualify soils.

3.0 Laboratory Method **Yes [__]** No [__] N/A [__] Comments:

3.1 Was the correct laboratory method used?

ACTION: If no, contact lab to provide justification for method change compared to the requested method. Contact senior chemist to inform Client of change or to request variance.

3.2 Are the practical quantitation limits the same as those specified by the ☐ SOW ☐ QAPP ☐ Lab? **Yes [__]** No [__] N/A [__] Comments:

Note: *The MADEP QA/QC Guidelines do not yet list PQLs for wet chemistry analyses, therefore all criteria will default to values stipulated in the QAPP*. Where the QAPP does not define criteria, QA/QC requirements default to limits employed by the lab**. Other criteria may also apply.*

Ammonia* ☐ = 0.1 mg/ L	Alkalinity**☐ = 1 mg/L	Bicarbonate Alkalinity** ☐ = 1 mg/L	Carbonate Alkalinity** ☐ = 1 mg/L
Nitrate Nitrogen as N* ☐ = .05 mg/L	Nitrite Nitrogen as N* ☐ = .01 mg/L	Chloride* ☐ = 1 mg/L	Hardness *☐ = 2.64 mg/L
Spec. Cond.** ☐ 3 umhos/cm	Total Organic Carbon** ☐ = 1 mg/L		Sulfate (EPA 300.0)* ☐ = 2 mg/L
COD:* Low – 20 mg/L	pH* ☐ < 2 to > 12	TDS* ☐ = 10 mg/L	TSS* ☐ = 5 mg/L

Other parameter(list) _____PQL = _____ ☐ Source of PQL = _____

Other parameter(list) _____ PQL = _____ ☐ Source of PQL = _____

ACTION: If no, evaluate change with respect to sample matrix, preparation, dilution, moisture, etc. If sample PQL is indeterminate, contact lab for explanation.

3.3 Are the appropriate parameter results present for each sample in the SDG? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, check Request for Analysis to verify if method was ordered and COC to verify that it was sent, and contact lab for resubmission of the missing data

3.4 If *dilutions were required, were dilution factors reported?* **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact the lab for submission.

4.0 <u>Method Blanks</u> **Yes [__]** No [__] N/A [__] Comments:

4.1 Are the Method Blank Summaries present?

ACTION: If no, call the laboratory for submission of missing data.

4.2 Was a method blank analyzed for each analysis batch of wet chemistry field samples of 20 or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, document discrepancy in case narrative and contact lab for justification. Consult senior chemist for action needed.

4.3 Is the method blank less than the PQL? (See Section 3.2 for PQLs).	**Yes [__]**	No [__]	N/A [__]	Comments:
4.4 Do any method blanks have positive results for wet chemistry parameters? Qualify data according to the following: If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL. If the sample concentration is > 5 × blank value, no qualification is needed.	Yes [__]	**No [__]**	N/A [__]	Comments:

ACTION: If any blank has positive results, list all the concentrations detected and flagging level (flagging level = 5 × blank value) on the checklist. List all affected samples and their qualifiers.

5.0 Laboratory Control Standards

5.1 Was a laboratory control standard (LCS) run with each analytical batch of 20 samples or less?	**Yes [__]**	No [__]	N/A [__]	Comments:

ACTION: If no, call laboratory for LCS form submittal. If data is not available, use professional judgment to determine qualification actions for data associated with the batch.

5.2 Is a LCS Summary Form present?	**Yes [__]**	No [__]	N/A [__]	Comments:

ACTION: If no, contact lab for resubmission of missing data.

5.3 Is any wet chemistry analyte LCS recovery outside the control limits?	Yes [__]	**No [__]**	N/A [__]	Comments:

LCS Limits:

Alkalinity** ☐ = 80-120% Bicarbonate Alkalinity** ☐ = 80-120% Carbonate Alkalinity** ☐ = 80-120% Specific Conductivity *☐ = 80-120%

Total Organic Carbon** ☐ = 80-120% TDS** ☐ = 80-120% Ammonia Nitrogen as N* ☐ = 80-120%

COD Low* ☐ = 80-120% Nitrate Nitrogen as N**☐ = 80-120%% Nitrite Nitrogen as N** ☐= 80-120%

Hardness* ☐ = 80-120% Chloride* ☐ = 80-120% Sulfate (EPA 300.0)* ☐ = 80-120% pH* ☐ = 80-120% TSS* NA

Other parameter(list) _____%R = _____ ☐ Rec Limits= _____

Other parameter(list) _____ %R = _____ ☐ Rec Limits = _____

(*MADEP has not yet defined LCS recovery limits for wet chemistry analyses.)*

ACTION: If recovery is above the upper limit, qualify all positive sample results within the batch as (J). If recovery is below the lower limit, qualify all positive and no-detect results within the batch as (J). If LCS recovery is <10%, non-detect results are rejected (R).

6.0 Matrix Spikes

Matrix spikes may be collected at different frequencies based on monthly, quarterly, or task specific schedules. Confirm spike requirements for each set with the senior chemist.

6.1 Were project-specific MS/MSDs analyzed? List project samples that were spiked.

ACTION: If no, contact senior chemist to see if any were specified.

Yes [__] No [__] N/A [__] Comments:

6.2 Is the MS/MSD Recovery Form present?

ACTION: If no, contact lab for resubmission of missing data.

Yes [__] No [__] N/A [__] Comments:

6.3 Were matrix spikes analyzed at the required frequency of 1 per 20 samples per matrix? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If any matrix spike data is missing, call lab for resubmission.

6.4 Are any wet chemistry analyte spike recoveries outside of the QC limits?

Yes [__] **No [__]** N/A [__] Comments:

NOTE: $\%R = \frac{(SSR-SR)}{SA} \times 100\%$ Where: SSR = Spiked sample result
SR = Sample result
SA = Spike added

MS/MSD Recovery Limits:

Alkalinity* = NA Bicarbonate Alkalinity* = NA Carbonate alkalinity* = NA Ammonia* (LACHAT) ☐ = 74-125%

Chloride** ☐= 75-125% Specific Conductivity * = NA Total Organic Carbon* = NA TDS** = NA

COD Low* ☐ = 75-125% Nitrate Nitrogen as N** ☐ = 75-125%

Nitrite Nitrogen as N** ☐ = 75-125% Hardness* ☐ = 75-125% Sulfate (EPA 300.0)* ☐ = 75-125% pH* = NA TSS* = NA

Other parameter(list) _____% R = _____ ☐ Rec Limits = _____

* = Laboratory Limits ** = Olin QAPP Limits (*MADEP has not yet defined LCS recovery limits for wet chemistry analyses.*)

NOTES: 1) If only one of the recoveries for an MS/MSD pair is outside of the control limits, no qualification is necessary. Use professional judgment for the MS/MSD flags.
2) If the MS/MSD was performed by the laboratory on a non-project sample, no qualification is required.

ACTION: MS/MSD flags only apply to the sample spiked. Do not evaluate if sample concentration is > 4X spike. If the recoveries of the MS and MSD exceed the upper control limit, qualify positive results as estimated (J). If the recoveries of the MS and MSD are lower than the lower control limit but > 30%, qualify both positive results and non-detects (J). If the MS/MSD recovery is < 30% and the sample is non-detect, the results are considered unusable and flagged (R).

ACTION: Laboratory control limits apply when spiked sample results fall within the normal calibration range. If dilutions are required due to high sample concentrations, the data is evaluated, but no flags are applied.

6.5 Are any RPDs for MS/MSD recoveries outside of the QA/QC limits?

NOTE: $RPD = \frac{S - D}{(S + D)/2} \times 100\%$ Where S = MS result
D = MSD result

Yes [__] **No [__]** N/A [__] Comments:

MS/MSD RPD Limits:

Alkalinity* = NA	Bicarbonate Alkalinity* = NA	Carbonate alkalinity* = NA	Ammonia** (LACHAT) ☐ = 20%
Chloride**(SM 4500 Cl) ☐ = 20%	Specific Conductivity * = NA	Total Organic Carbon* = NA	TDS** = NA Total Organic Carbon* = NA
COD Low** ☐ = 20%	COD High** ☐ = 20%	Nitrate Nitrogen as N** ☐ 20%	Nitrite Nitrogen as N** ☐ = 20%
Hardness* = NA	Sulfate (EPA 300.0)* ☐ = 20%	pH* = NA	TSS* = NA

Other parameter(list) _____ RPD = _____ ☐ RPD limit = _____

Other parameter(list) _____ RPD = _____ ☐ RPD limit = _____

* = Laboratory Limits ** = Olin QAPP Limits

(*MADEP has not yet defined LCS recovery limits for wet chemistry analyses*.)

7.0 Laboratory Duplicate

Are the RPDs for the laboratory duplicates <20%? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If the RPD is greater than specified limits, qualify all results for that analyte as estimated (J).

8.0 Sampling Accuracy

The majority of ground water samples are collected directly from a tap, process stream, or with dedicated tubing. Rinse blanks will not be collected.

8.1 Were rinsate blanks collected? Prior to evaluating rinsate blanks, obtain a list of the associated samples from the senior chemist. **Yes [__]** No [__] N/A [__] Comments:

8.2 Do any rinsate blanks have positive results? Yes [__] **No [__]** N/A [__] Comments:

ACTION: Evaluate rinsate results vs. blank results to determine if contaminant may be laboratory-derived. If not lab-related, qualify according to the table below.

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

***NOTE**: MADEP does not require the collection of rinsate blanks.*

9.0 Field Duplicates

9.1 Were field duplicate samples collected? Obtain a list of samples and their associated field duplicates. **Yes [__]** No [__] N/A [__] Comments:

9.2 Were field duplicates collected per the required frequency? **Yes [__]** No [__] N/A [__] Comments:

SOW ☐ QAPP ☐ *MADEP Option 1(1 per 20)* ☐ *MADEP Option 3 (1 per 10)* ☐

9.3 Was the RPD ≤ 50% for soils or ≤30% for waters? Calculate the RPD for all results and attach to this review. **Yes [__]** No [__] N/A [__] Comments:

ACTION: RPD must be ≤50% for soil or ≤30% water. Qualify data (J) for both sample results if the RPD exceeds limit.

Was any of the data qualified? Yes [__] **No [__]** N/A [__] Comments:

If so, apply data qualifiers directly to the DQE copy of laboratory report and **flag pages** for entry in database.

REFERENCES

MACTEC, Quality Assurance Project Plan for Remedial Investigation/Feasibility Study – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting. Draft .October 2008.

STL-Westfield, 2002. "Olin – General Chemistry Control Limits (Soil & Water)," Severn Trent Laboratories, Inc., 53 Southampton Road, Westfield, MA, 01085.

OLIN CORPORATION
LEVEL I DATA QUALITY EVALUATION
STANDARD OPERATING PROCEDURE AND CHECKLIST
EXTRACTABLE PETROLEUM HYDROCARBONS BY METHOD MADEP-EPH-98-1

Reviewer/Date_____
Sr. Review/Date_____
Lab Report # _____
LAW Project #_____

1.0 Laboratory Deliverable Requirements

1.1 Laboratory Information: Was all of the following provided in the laboratory report? **Yes [__]** No [__] N/A [__] Comments:
Check items received.

☐ *Name of Laboratory* ☐ *Certification ID #* ☐ *Address* ☐ *Project ID* ☐ *Phone #* ☐ *Sample identification – Field and Laboratory*

Client Information: ☐ *Name* ☐ *Address* ☐ *Phone #* ☐ *Client Contact* *(IDs must be cross-referenced)*

ACTION: If no, contact lab for submission of missing or illegible information.

1.2 Laboratory Report Certification Statement ***Yes [__]*** *No [__]* *N/A [__]* *Comments:*

Does the laboratory report include a completed Analytical Report Certification in the required format?

***ACTION**: If no, contact lab for submission of missing certification or certification with correct format.*

1.3 Laboratory Case Narrative:** Are both of the following statements true?* ***Yes [__] *No [__]* *N/A [__]* *Comments:*

☐ *Narrative serves as an exception report for the project and method QA/QC performance.* ☐ *Narrative includes an explanation of each discrepancy on the Certification Statement.*

***ACTION**: If no, contact lab for submission of missing or illegible information.*

1.4 Chain of Custody (COC) **Yes [__]** No [__] N/A [__] Comments:

Does the laboratory report include the *original* Chain of Custody forms containing all samples in this SDG?

NOTE: Olin receives and maintains the *original* COC.

ACTION: If no, contact lab for submission of missing original COC.

1.5 Sample Receipt Information (*Cooler Receipt Form*): Were each of the following tasks completed and recorded upon receipt of the sample(s) into the laboratory? **Yes [__]** No [__] N/A [__] Comments:

☐ Sample temperature confirmed: must be 1° – 10° C. (If samples were sent by courier and delivered on the same day as collection, temperature requirement does not apply).

☐ Container type noted ☐ Custody seals checked ☐ Condition observed ☐ pH verified (where applicable) ☐ Field and lab IDs cross referenced

ACTION: If no, contact lab for submission of missing or incomplete documentation.

1.5.1 Were the correct bottles and preservatives used? **Yes [__]** No [__] N/A [__] Comments:

Water - 1 Liter amber bottle / 5 ml 1:1 HCl, cool to 4°C
Soil - 4 oz amber soil jar w/ Teflon lined cap / cool to 4°C

ACTION: If no, inform senior chemist. Document justification for change in container/volume (if applicable), qualify positive and non-detect data (J) data if cooler temperature exceeds 10°C. Rejection of data requires professional judgment

ACTION: If each VOA vial for a sample contains air bubbles or the VOA vial analyzed contained air bubbles, flag positives (J) and reject nondetects (R).

1.5.2 Were all samples delivered to the laboratory without breakage? **Yes [__]** No [__] N/A [__] Comments:

1.5.3 Does the *Cooler Receipt Form* or Lab Narrative indicate other problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? Yes [__] **No [__]** N/A [__] Comments:

1.6 Sample Results Section: *Was the follow ing information supplied in the laboratory report for each sample?* **Yes [__]** No [__] N/A [__] Comments:

OLIN CORPORATION
LEVEL I DATA QUALITY EVALUATION
STANDARD OPERATING PROCEDURE AND CHECKLIST
EXTRACTABLE PETROLEUM HYDROCARBONS BY METHOD MADEP

☐ Field ID and Lab ID ☐ *Date and time collected* ☐ Analyst Initials ☐ *Dilution Factor* ☐ % moisture or solids ☐ *Reporting limits* ☐ *Clean-up method* ☐ Analysis method ☐ *Preparation method* ☐ *Date of preparation/extraction/digestion clean-up and analysis, where applicable* ☐ Matrix ☐ Target analytes and concentrations ☐ Units (soils must be reported in dry weight)

ACTION: If no, contact lab for submission of missing or incomplete information.

1.7 QA/QC Information: Was the following information provided in the laboratory report for each sample batch? **Yes [__]** No [__] N/A [__] Comments:

☐ Method blank results ☐ LCS recoveries ☐ MS/MSD recoveries and RPDs ☐ Laboratory duplicate results (where applicable) ☐ Surrogate recoveries

ACTION: If no, contact lab for submission of missing or incomplete information.

2.0 Holding Times **Yes [__]** No [__] N/A [__] Comments:

Have any technical holding times, determined from date of collection to date of analysis, been exceeded? For water samples, the holding time is 14 days from sampling to extraction and 40 days from extraction to analysis. For soil samples, the holding time is 7 days from sampling to extraction and 40 days from extraction to analysis.

NOTE: List samples that exceeded hold time with number of days exceeded on checklist.

ACTION: If technical holding times are exceeded, qualify all positive results (J) and reject (R) all non-detect results.

3.0 Laboratory Method

3.1 Was the correct laboratory method used? **Yes [__]** No [__] N/A [__] Comments:

Extractable Petroleum Hydrocarbons MADEP-EPH-98-1

ACTION: If no, contact lab to provide justification for method change compared to the requested method. Contact senior chemist to inform Client of change or to request variance.

3.2 Are the practical quantitation limits the same as those specified by the **Yes [__]** No [__] N/A [__] Comments:

☐ SOW ☐ QAPP ☐ Lab?

ACTION: If no, evaluate change with respect to sample matrix, preparation, dilution, moisture, etc. If sample PQL is indeterminate, contact lab for explanation.

3.3 Are the appropriate parameter results present for each sample in the SDG? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, check Request for Analysis to verify if method was ordered and COC to verify that it was sent, and contact lab for resubmission of the missing data

3.4 If *dilutions were required, were dilution factors reported?* **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact the lab for submission.

4.0 Method Blanks

4.1 Is the Method Blank Summary present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, call the laboratory for submission of missing data.

4.2 Was a method blank analyzed for each analysis batch of EPH field samples of 20 or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, document discrepancy in case narrative and contact lab for justification. Consult senior chemist for action needed.

4.3 Is the method blank less than the PQL? (See attached table for PQLs) Yes [__] **No [__]** N/A [__] Comments:

4.4 Do any method blanks have positive results for EPHs? Qualify data according to the following: Yes [__] **No [__]** N/A [__] Comments:

If the sample concentration is < 5 × blank value, flag sample result "JB"

If the sample concentration is > 5 × blank value, no qualification is needed.

ACTION: If any blank has positive results, list all the concentrations detected and flagging level (flagging level = 5 × blank value) on the checklist. List all affected samples and their qualifiers.

5.0 Laboratory Control Standard

5.1 Was a laboratory control standard run with each analytical batch of 20 samples or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: Call laboratory for LCS form submittal. If data are not available, reject (R) data associated with that batch.

5.2 Is a LCS Summary Form present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact lab for resubmission of missing data.

5.3 Is the recovery of any analyte outside of control limits of 40-140%? Yes [__] **No [__]** N/A [__] Comments:

NOTE: *A second source LCS containing at least five target analytes from each fraction is required by MADEP.*

NOTE: *MADEP guidelines list LCS recovery limits as 40-140 for all analytes except C36. The laboratory must identify analytes that routinely exceed these limits. See the attached table for a listing of MADEP LCS control limits vs. the control limits listed in the QAPP.*

ACTION: If recovery is above the upper limit, qualify all positive sample results within the batch as (J). If recovery is below the lower limit but > 10%, qualify all positive and no-detect results within the batch as (J). If LCS recovery is <10%, positive and non-detect results are rejected (R) unless the QC limit for that compound is below 10% (flag as above).

5.4 Are 80% of LCS recoveries within laboratory control limits? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If 80% of LCS recoveries are not within limits, use professional judgment and consult Senior Chemist. If more than half of the recoveries are above control limits, qualify all positive results as (J). If more than half of the recoveries are below control limits, batch may require rejection and reanalysis

6.0 Matrix Spikes

6.1 Were project-specific MS/MSDs collected? List project samples that were spiked.

Yes [__] No [__] N/A [__] Comments:

ACTION: If no, contact senior chemist to see if any were specified.

6.2 Is the MS/MSD Recovery Form present?

Yes [__] No [__] N/A [__] Comments:

ACTION: If no, contact lab for resubmission of missing data.

6.3 Were matrix spikes analyzed at the required frequency of 1 per 20 samples per matrix?

Yes [__] No [__] N/A [__] Comments:

ACTION: If any matrix spike data is missing, call lab for resubmission.

6.4 Are any EPH spike recoveries outside of the QC limits?

Yes [__] **No [__]** N/A [__] Comments:

NOTE: $\%R = \frac{(SSR-SR)}{SA} \times 100\%$ Where: SSR = Spiked sample result, SR = Sample result, SA = Spike added

NOTE: *A second source MS/MSD containing at least five target analytes from each fraction is required by MADEP*.

NOTE: *MADEP guidelines list MS/MSD recovery limits as 40-140 for all analytes except C36. The laboratory must identify analytes that routinely exceed these limits. See the attached table for a listing of MADEP LCS control limits vs. the control limits listed in the QAPP*

NOTES: 1) If only one of the recoveries for an MS/MSD pair is outside of the control limits, no qualification is necessary. Use professional judgment for the MS/MSD flags.
2) If the MS/MSD was performed by the laboratory on a non-project sample, no qualification is required.

NOTE: Laboratory control limits apply when spiked sample results fall within the normal calibration range. If dilutions are required due to high sample concentrations, the data is evaluated, but no flags are applied.

ACTION: MS/MSD flags only apply to the sample spiked. If the recoveries of the MS and MSD exceed the upper control limit, qualify positive results as estimated (J). If the recoveries of the MS and MSD are lower than the lower control limit but > 30%, qualify both positive results and non-detects (J). If the MS/MSD recovery is < 30% and the sample is non-detect, the results are considered unusable and flagged (R).

6.5 Are any RPDs for MS/MSD recoveries outside of the QC limits of *≤ 50 for soil and ≤ 30 for water* ? Yes [__] **No [__]** N/A [__] Comments:

NOTE: $RPD = \frac{S-D}{(S+D)/2} \times 100\%$ Where: S = MS sample result
D = MSD sample result

NOTE: *MADEP guidelines list MS/MSD RPD lim its for water and soil as ≤ 50. See the attached table for a listing of MADEP MS/MSD RPD limits vs. the control lim its listed in the QAPP.*

NOTE: Laboratory control limits apply when spiked sample results fall within the normal calibration range. If dilutions are required due to high sample concentrations, the data are evaluated, but no flags are applied.

ACTION: If the RPD exceeds the control limit, qualify positive and non-detects results (J).

7.0 Surrogate Recoveries

Were one or more EPH surrogate recoveries outside of laboratory limits for any sample or method blank? If yes, were samples re-analyzed? Yes [__] **No [__]** N/A [__] Comments:

NOTE: %R = QD x 100% Where: S = MS sample result
D = MSD sample result

NOTE: *MADEP guidelines require at least two extraction and two fractionation surrogates, and lists recovery lim its as 40- 140% for all surrogates. See the attached table for a listing of MADEP surrogate limits vs. the control limits listed in the QAPP*

NOTE: If surrogate recoveries fail due to dilution, results are not flagged. Document on checklist and in the case narrative.

ACTION: If recoveries are >10%, but fail to meet QC criteria: (1) For recoveries below the QC limit, qualify non-detects and positives (J), and (2) For recoveries above the QC limit, qualify only positives (J). If any surrogate recovery is <10% (unless the QC limits are below 10%, in which case, results are flagged as stated above), flag positives (J) and reject nondetects (R).

8.0 Sampling Accuracy

8.1 Were rinsate blanks collected? Prior to evaluating rinsate blanks, obtain a list of the associated samples from the project chemist. Yes [__] **No [__]** N/A [__] Comments:

***NOTE**: MADEP does not specify the collection of rinsate blanks.*

8.2 Do any rinsate blanks have positive results? Yes [__] **No [__]** N/A [__] Comments:

ACTION: Evaluate rinsate results against other blank results to determine if contaminant may be laboratory-, ambient, or shipment-derived. If results are not lab-, ambient, or shipment related, qualify according to the table below.

Sample conc. < 5x blank value:	Sample conc. > 5x blank value:
Flag sample result "JB"	No qualification is needed

9.0 Field Duplicates

9.1 Were field duplicate samples collected? Obtain a list of the samples and their associated field duplicates.	Yes [__]	**No [__]**	N/A [__]	Comments:
9.2 Were field duplicates collected per the required frequency? ☐ SOW ☐ QAPP ☐ *MADEP Option 1(1 per 20)* ☐ *MADEP Option 3 (1 per 10)*	**Yes [__]**	No [__]	N/A [__]	Comments:
9.3 Was the RPD ≤ 50% for soils or waters? Calculate the RPD for all results and attach to this review.	**Yes [__]**	No [__]	N/A [__]	Comments:

ACTION: RPD must be ≤50% for soil and water. Qualify data (J) for both sample results if the RPD exceeds 50%.

10.0 Application of Validation Qualifiers

Was any of the data qualified?	Yes [__]	**No [__]**	N/A [__]	Comments:

If so, apply data qualifiers directly to the DQE copy of laboratory report and **flag pages** for entry in database.

REFERENCES

MACTEC, Quality Assurance Project Plan for Remedial Investigation/Feasibility Study – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting. Draft .October 2008.

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USEPA, 1988. "Laboratory Data Validation Functional Guidelines for Evaluating Organic and Inorganic Analysis," EPA/540/R-94-012 and EPA/540-R-94-013, July 1988.

MADEP, 2001. Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup, "Massachusetts Quality Assurance / Quality Control (QA / QC Requirements,**"** BWSC-CAM, Interim Final Draft, Revision No. 2, 5 October 2001.

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OLIN CORPORATION
LEVEL I DATA QUALITY EVALUATION
STANDARD OPERATING PROCEDURE AND CHECKLIST
VOLATILE PETROLEUM HYDROCARBONS BY METHOD MADEP

Reviewer/Date_____
Sr. Review/Date_____
Lab Report # _____
Project #_____

1.0 Laboratory Deliverable Requirements

1.1 Laboratory Information: Was all of the following provided in the laboratory report? **Yes [__]** No [__] N/A [__] Comments:
Check items received.

☐ *Name of Laboratory* ☐ *Certification ID #* ☐ *Address* ☐ *Project ID* ☐ *Phone #* ☐ *Sample identification – Field and Laboratory*

Client Information: ☐ *Name* ☐ *Address* ☐ *Client Contact* *(IDs must be cross-referenced)*

ACTION: If no, contact lab for submission of missing or illegible information.

1.2 Laboratory Report Certification Statement

Yes [__] *No [__]* *N/A [__]* *Comments:*

Does the laboratory report include a completed Analytical Report Certification in the required format?

***ACTION**: If no, contact lab for submission of missing certification or certification with correct format.*

1.3 Laboratory Case Narrative:

Yes [__] *No [__]* *N/A [__]* *Comments:*

☐ *Narrative serves as an exception report for the project and method QA/QC performance.* ☐ *Narrative includes an explanation of each discrepancy on the Certification Statement.*

***ACTION**: If no, contact lab for submission of missing or illegible information.*

1.4 Chain of Custody (COC)

Yes [__] No [__] N/A [__] Comments:

Does the laboratory report include a copy of the completed Chain of Custody forms containing all samples in this SDG?

NOTE: Olin receives and maintains the *original* COC.

ACTION: If no, contact lab for submission of completed COC.

1.5 Sample Receipt Information (*Cooler Receipt Form*): Were each of the following tasks completed and recorded upon receipt of the sample(s) into the laboratory? **Yes [__]** No [__] N/A [__] Comments:

☐ Sample temperature confirmed: must be 1° – 10° C. (If samples were sent by courier and delivered on the same day as collection, temperature requirement does not apply).

☐ Container type noted ☐ Condition observed ☐ Field and lab IDs cross referenced

ACTION: If no, contact lab for submission of missing or incomplete documentation.

1.5.1 Were the correct bottles and preservatives used? **Yes [__]** No [__] N/A [__] Comments:

Water - 40 mL VOA vial/HCL to pH<2, cool to 4°C
Soil - 5 gram Encore™/cool to 4°C or 40 mL VOA vial with field preservation of sodium bisulfate (low-level) or methanol (high-level) or field preservation in water if soils are reactive to sodium bisulfate (i.e. alkaline conditions, excessive humic acid content, etc.)

ACTION: If no, inform senior chemist. Document justification for change in container/volume (if applicable); qualify both positive data and non-detect data (J) if cooler temperature exceeds 10°C. Rejection of data requires professional judgment

ACTION: If each VOA vial for a sample contains air bubbles or the VOA vial analyzed contained air bubbles, flag positives (J) and reject nondetects (R).

1.5.2 Were all samples delivered to the laboratory without breakage? **Yes [__]** No [__] N/A [__] Comments:

1.5.3 Does the *Cooler Receipt Form* or Lab Narrative indicate other problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? Yes [__] **No [__]** N/A [__] Comments:

1.6 Sample Results Section: *Was the following information supplied in the laboratory report for each sample?* **Yes [__]** No [__] N/A [__] Comments:

☐ Field ID and Lab ID ☐ *Date and time collected* ☐ Analyst Initials ☐ *Dilution Factor* ☐ % moisture or solids ☐ *Reporting limits*

☐ Analysis method concentrations ☐ *Preparation method* ☐ *Date of preparation/extraction/digestion clean-up and analysis, where applicable* ☐ Matrix ☐ Target analytes and ☐ Units (soils must be reported in dry weight)

ACTION: If no, contact lab for submission of missing or incomplete information.

1.7 QA/QC Information: Was the following information provided in the laboratory report for each sample batch? **Yes [__]** No [__] N/A [__] Comments:

☐ Method blank results ☐ LCS recoveries ☐ MS/MSD recoveries and RPDs ☐ Surrogate recoveries

ACTION: If no, contact lab for submission of missing or incomplete information.

2.0 Holding Times

Have any technical holding times, determined from date of collection to date of analysis, been exceeded? Yes [__] **No [__]** N/A [__] Comments:

For water samples, the holding time is 7 days from sampling for unpreserved samples and 14 days for preserved samples.

For soil samples, methanol preservation required with a holding time of 14 days. If an Encore™ sampler was used, the lab must ***preserve*** the sample within 48 hours. Analytical holding time from time of preservation is 14 days.

NOTE: List samples that exceed hold time with # of days exceeded on checklist

ACTION: If technical holding times are exceeded, qualify all positive results (J). Use professional judgment to reject (R) data for grossly exceeded.

3.0 Laboratory Method

3.1 Was the correct laboratory method used? **Yes [__]** No [__] N/A [__] Comments:

Purge and Trap Water: 5030B Soil: 5035
Volatile Petroleum Hydrocarbons MADEP VPH 98-1

ACTION: If no, contact lab to provide justification for method change compared to the requested method. Contact senior chemist to inform Client of change or to request variance.

3.2 Are the practical quantitation limits the same as those specified by the ☐ SOW ☐ QAPP ☐ Lab ☐ MADEP **Yes [__]** No [__] N/A [__] Comments:

NOTE: *The MADEP QA/QC Guidelines provides PQLs for volatile petroleum hydrocarbons. See MADEP PQLs vs. the PQLs listed in the QAPP.*

ACTION: If no, evaluate change with respect to sample matrix, preparation, dilution, moisture, etc. If sample PQL is indeterminate, contact lab for explanation.

3.3 Are the appropriate parameter results present for each sample in the SDG? **Yes [__]** No [__] N/A [__] Comments:

NOTE: *The MADEP QA/QC Guidelines requires a minimum compound reporting list for volatile organic compounds.*

3.4 If *dilutions were required, were dilution factors reported?* **Yes [__]** No [__] N/A [__] Comments:

NOTE: *MADEP guidance states that if a diluted and an undiluted analysis is performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte.*

ACTION: If no, contact the lab for submission.

4.0 Method Blanks

4.1 Are the Method Blank Summaries present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, call the laboratory for submission of missing data.

4.2 Was a method blank analyzed for each analytical batch of 20 samples or less? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, document discrepancy in case narrative and contact lab for justification. Consult senior chemist for action needed.

4.3 Is the method blank less than the PQL? (See attached table for PQLs).	**Yes [__]**	No [__]	N/A [__]	Comments:
4.4 Do any method blanks have positive results for VPH parameters? Qualify data according to the following:	Yes [__]	**No [__]**	N/A [__]	Comments:

For VPH contaminants:

Review blank and sample chromatograms to evaluate the nature of the detection in the blank and associated samples. Use professional judgment. The following actions may be applied:

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed..

ACTION: If any blank has positive results, list all the concentrations detected and flagging level (flagging level = 10x or 5 × blank value) on the checklist. List all affected samples and their qualifiers.

5.0 Laboratory Control Standard

5.1 Was a laboratory control standard (LCS) run with each analytical batch of 20 samples or less?	**Yes [__]**	No [__]	N/A [__]	Comments:

ACTION: Call laboratory for LCS form submittal. If data are not available, reject (R) data associated with that batch.

5.2 Is a LCS Summary Form present?	**Yes [__]**	No [__]	N/A [__]	Comments:

ACTION: If no, contact lab for resubmission of missing data.

5.3 Is the recovery of any analyte outside of control limits of 70-130% in the QAPP? Yes [__] **No [__]** N/A [__] Comments:

NOTE: *A full target, second source LCS is required by MADEP.*

NOTE: *MADEP guidelines list LCS recovery limits as 70-130 except for naphthalene. The laboratory must identify any other analytes that routinely exceed 70-130 percent.*

ACTION: If recovery is above the upper limit, qualify all positive sample results within the batch as (J). If recovery is below the lower limit but > 10%, qualify all positive and no-detect results within the batch as (J). If LCS recovery is <10%, positive and non-detect results are rejected (R) unless the QC limit for that compound is below 10% (flag as above).

5.4 Are 80% of LCS recoveries within laboratory control limits? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If 80% of LCS recoveries are not within limits, use professional judgment and consult Senior Chemist. If more than half of the recoveries are above control limits, qualify all positive results as (J). If more than half of the recoveries are below control limits, batch may require rejection and reanalysis

6.0 Matrix Spikes

6.1 Were project-specific MS/MSDs collected? List project samples that were spiked. **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact senior chemist to see if any were specified.

6.2 Is the MS/MSD Recovery Form present? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If no, contact lab for resubmission of missing data.

6.3 Were matrix spikes analyzed at the required frequency of 1 per 20 samples per matrix? **Yes [__]** No [__] N/A [__] Comments:

ACTION: If any matrix spike data are missing, call lab for resubmission.

6.4 Are any VPH spike recoveries outside of the QC limits of 70-130%? Yes [__] **No [__]** N/A [__] Comments:

NOTE: $\%R = \frac{(SSR-SR)}{SA} \times 100\%$ Where: SSR = Spiked sample result, SR = Sample result, SA = Spike added

NOTE: *A full target, second source MS/MSD is required by MADEP.*

NOTE: *MADEP guidelines list MS/MSD recovery limits as 70-130 except for naphthalene.*

NOTES: 1) If only one of the recoveries for an MS/MSD pair is outside of the control limits, no qualification is necessary. Use professional judgment for the MS/MSD flags.
2) If the MS/MSD was performed by the laboratory on a non-project sample, no qualification is required.

ACTION: MS/MSD flags only apply to the sample spiked. If the recoveries of the MS and MSD exceed the upper control limit, qualify positive results as estimated (J). If the recoveries of the MS and MSD are lower than the lower control limit but > 30%, qualify both positive results and non-detects (J). If the MS/MSD recovery is < 30% and the sample is non-detect, the results are considered unusable and flagged (R).

ACTION: Laboratory control limits apply when spiked sample results fall within the normal calibration range. If dilutions are required due to high sample concentrations, the data is evaluated, but no flags are applied.

6.5 Are any RPDs for MS/MSD recoveries outside of the QC limits of 50 (water and soil)? Yes [__] **No [__]** N/A [__] Comments:

NOTE: $RPD = \frac{S-D}{(S+D)/2} \times 100\%$ Where: S = MS sample result, D = MSD sample result

NOTE: *MADEP guidelines list MS/MSD RPD limits for both water and soils as ≤ 50.*

ACTION: If the RPD exceeds the control limit, qualify positive results and non-detects (J).

ACTION: Laboratory control limits apply when spiked sample results fall within the normal calibration range. If dilutions are required due to high sample concentrations, the data are evaluated, but no flags are applied.

7.0 Surrogate Recoveries

Were VPH surrogate recoveries outside of the QAPP limits of 70-130% for any sample or method blank? If yes, were samples re-analyzed? Yes [__] **No [__]** N/A [__] Comments:

NOTE: %R = QD x 100% Where: S = MS sample result
D = MSD sample result

NOTE: *MADEP guidelines list surrogate lim its for both water and soils as 70-130% for both detectors.*

ACTION: If recoveries are >10%, but fail to meet QC criteria: (1) For recoveries below the QC limit, qualify non-detects and positives (J), and (2) For recoveries above the QC limit, qualify only positives (J). If any surrogate recovery is <10% (unless the QC limits are below 10%, in which case, results are flagged as stated above), flag positives (J) and reject nondetects (R).

NOTE: If surrogate recoveries fail due to dilution, results are not flagged. Document on checklist and in the case narrative.

8.0 Sampling Accuracy

8.1 Were trip blanks shipped with VOC samples and analyzed? **Yes [__]** No [__] N/A [__] Comments:

NOTE*: MADEP requires trip blanks per the following frequency:*

	Soil/Sediment	*Aqueous*	*Drinking Water*
Option 1	*Not Required*	*Not Required*	*1 per cooler VOAs/VPH*
Option 3	*1 per 10 samples*	*1 per 10 samples*	*1 per 10 samples*

8.2 Do any trip blanks have positive results? Yes [__] **No [__]** N/A [__] Comments:

ACTION: Prepare a list of samples shipped in the same cooler as the contaminated blank.

ACTION: Evaluate trip blank results against method blank results to determine if contaminant may be laboratory-derived. If results are not lab-related, qualify according to the table below.

If the sample concentration is < 5 × blank value, flag sample result non-detect "U" at the PQL or the concentration reported if greater than the PQL.

If the sample concentration is > 5 × blank value, no qualification is needed.

8.3 Were ambient blanks shipped with VPH samples and analyzed? **Yes [__]** No [__] N/A [__] Comments:

NOTE*: MADEP requires ambient (field) blanks per the following frequency:*

	Soil/Sediment	*Aqueous*	*Drinking Water*
Option 1	*Not Required*	*Not Required*	*Not Required*
Option 3	*1 per 10 samples*	*1 per 10 samples*	*1 per 10 samples*

8.4 Do any ambient blanks have positive results? Yes [__] **No [__]** N/A [__] Comments:

ACTION: Prepare a list of samples associated with the contaminated blank (all collected from the site on that day).

ACTION: Evaluate ambient blank results against method and trip blank results to determine if contaminant may be laboratory- and/or shipment-derived. If results are not lab- and/or shipment-related, qualify according to the table above (8.2).

8.5 Were rinsate blanks collected? Prior to evaluating rinsate blanks, obtain a list of the associated samples from the senior chemist. **Yes [__]** No [__] N/A [__] Comments:

NOTE*: MADEP does not specify the collection of rinsate blanks.*

8.6 Do any rinsate blanks have positive results? Yes [__] **No [__]** N/A [__] Comments:

ACTION: Evaluate rinsate results against blank results to determine if contaminant may be laboratory-, ambient-, or shipment-derived. If results are not lab-, ambient-, or shipment-related, qualify according to the table above (8.2).

9.0 Field Duplicates

9.1 Were field duplicate samples collected? Obtain a list of samples and their associated field duplicates. **Yes [__]** No [__] N/A [__] Comments:

9.2 Were field duplicates collected per the required frequency? **Yes [__]** No [__] N/A [__] Comments:

☐ SOW ☐ QAPP ☐ *MADEP Option 1(1 per 20)* ☐ *MADEP Option 3 (1 per 10)*

9.3 Was the RPD ≤ 50% for soils or ≤ 30% waters? Calculate the RPD for all results and attach to this review. **Yes [__]** No [__] N/A [__] Comments:

ACTION: RPD must be ≤50% for soil or ≤ 30% waters. Qualify data (J) for both sample results if the RPD is exceeded.

10.0 Application of Validation Qualifiers

Was any of the data qualified? **Yes [__]** No [__] N/A [__] Comments:

If so, apply data qualifiers directly to the DQE copy of laboratory report and **flag pages** for entry in database.

REFERENCES

MACTEC, Quality Assurance Project Plan for Remedial Investigation/Feasibility Study – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting. Draft .October 2008.

Massachusetts Department of Environmental Protection (MADEP), 1998. "Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)"; Division of Environmental Analysis; Office of Research and Standards; Bureau of Waste Site Cleanup; January 1998.

PESTICIDES/PCBs

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifications: Region I Organics (Draft 2/04)

TIER I / II / III (circle one)

SITE: Olin Chemical Project #:_____ SDG #:_____

LAB #: _____

Sample IDs: Attach tracking sheet and/or sample listing.

This checklist is designed to be used with USEPA Region I Validation Guidelines Part III (Draft 2/04). During Level III validation, calculation and transcription checks are completed for instrument tuning, surrogates, target compounds, spike recoveries, calibration data, and internal standards as specified in the guideline. These checks are documented on attached validation notes.

YES NO NA	
Data completeness ☐ ☐ ☐ All data summaries, QC forms and raw data available from hard copy or electronic data package ☐ ☐ ☐Data summaries match EDD	Contact lab if missing data. Lab to respond with 24 hours.
Holding Times and Preservation ☐ ☐ ☐Hold times met (Waters – Extract within 7 days, analyze within 40 days. Soils – extract within 14 days, analyze within 40 days)	
Initial Calibration ☐ ☐ ☐ %RSD less than or equal to 20% ☐ ☐ ☐ RTs within windows	
Continuing Calibration ☐ ☐ ☐ %D less than or equal to 25% ☐ ☐ ☐ RTs within windows	
Instrument Blank ☐ ☐ ☐ Analyzed at beginning of 12 hour clock and at end of analytical sequence	
Blank Contamination ☐ ☐ ☐ Instrument blank contamination ☐ ☐ ☐ Method blank contamination ☐ ☐ ☐ Equipment/Rinseate blank contamination	Evaluate all blanks for contamination. Highest contaminant level used for action level.
Surrogate Recoveries	

PESTICIDES/PCBs

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifications: Region I Organics (Draft 2/04)

TIER I / II / III (circle one)

☐ ☐ ☐ Surrogates percent recovery criteria met Pest/PCB for soil and water (30%-150%)	
Matrix Spikes and Laboratory Control Samples ☐ ☐ ☐ MS/MSD percent recovery criteria met (Pest - water and soil 30-150%) (PCB – water and soil 40-140%) ☐ ☐ ☐ MS/MSD RPD criteria met. PCBs (soil or water < or = 50%). Pest (soil or water < or = 30%). ☐ ☐ ☐ LCS percent recovery criteria met (Pest and PCB = 40%-140%)	
Field Duplicates ☐ ☐ ☐ RPD criteria met (Pest/PCB = soil <50% and water <30%)	
Target Compounds ☐ ☐ ☐ Reviewed narrative for anomalies	
PEST/PCB Identification ☐ ☐ ☐ Results reported from primary column ☐ ☐ ☐ PCB results < or = to 25% D between primary and confirmation column. ☐ ☐ ☐ Within RT windows	For positive results having %D between 25% and 100%, qualify result J. For positive results greater than reporting limit having %D > 100%, qualify result R or use professional judgment (NJ). For positive results less than reporting limit having %D > 100%, qualify result UJ at the reporting limit.

Validator's Signature:_____

Date: _____

Reference:

MACTEC, Project Operation Plan Volume IIB, Quality Assurance Project Plan for Remedial Investigation/Feasibility Study – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting. .April 2009.

SEMIVOLATILE ORGANICS

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifications: REGION I Organics Guideline (1996)

TIER I / II / III (circle one)

SITE: Olin Chemical Superfund Site Project #:_____ SDG #:_____

LAB #: _____

Sample IDs: Attach tracking sheet and/or sample listing.

This checklist is designed to be used with USEPA Region I Validation Guidelines Part II (12/1996). During Level III validation, calculation and transcription checks are completed for instrument tuning, surrogates, target compounds, spike recoveries, calibration data, and internal standards as specified in the guideline. These checks are documented on attached validation notes.

YES NO NA	
Data completeness ☐ ☐ ☐ All data summaries, QC forms and raw data available from hard copy or electronic data package ☐ ☐ ☐ Data summaries match EDD	Contact lab if missing data. Lab to respond with 24 hours.
Holding Times and Preservation ☐ ☐ ☐ Hold times met (Waters – Extract within 7 days, analyze within 40 days. Soils – extract within 14 days, analyze within 40 days)	
Instrument Performance Check (Tune) ☐ ☐ ☐ Tune available for each 12-hour period samples were analyzed ☐ ☐ ☐ Appropriate number of significant figures reported (at least 2) ☐ ☐ ☐ Mass/Charge list (m/z) criteria met	
Initial Calibration ☐ ☐ ☐ %RSD less than or equal to 30% ☐ ☐ ☐ RRF greater than or equal to 0.05	
Continuing Calibration ☐ ☐ ☐ %D less than or equal to 25% ☐ ☐ ☐ RRF greater than or equal to 0.05.	
Blank Contamination ☐ ☐ ☐ Method blank contamination ☐ ☐ ☐ Equipment/Rinseate blank contamination	Evaluate all blanks for contamination. Highest contaminant level used for action level.
Surrogate Recoveries	

SEMIVOLATILE ORGANICS

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifications: REGION I Organics Guideline (1996)

TIER I / II / III (circle one)

☐ ☐ ☐ Surrogates percent recovery criteria met: Soil = (base/neutral 30%-130%, acid 30%-130%) Water = (base/neutral 30%-130%, acid 15%-110%)	
Matrix Spikes and Laboratory Control Samples ☐ ☐ ☐ MS/MSD percent recovery criteria met Soil and Water = (base/neutral 40%-140%, acid 30%-130%) ☐ ☐ ☐ MS/MSD RPD criteria met (soils <50%, water <30%) ☐ ☐ ☐ LCS percent recovery criteria met soil/water (base 40%-140%, acid 30%-130%)	
Field Duplicates ☐ ☐ ☐ RPD criteria met (soils <50%, water <30%)	
Internal Standard ☐ ☐ ☐ Area counts within -50 to +100 percent of calib. std. ☐ ☐ ☐ Retention Time within 30 seconds of calib. std.	
Target Compounds ☐ ☐ ☐ Reviewed narrative for anomalies	
Tentatively Identified Compounds (TICs) ☐ ☐ ☐ TCL compounds reported as TICs	

Validator's Signature:_____

Date: _____

Reference:

MACTEC, Quality Assurance Project Plan for Remedial Investigation/Feasibility Study – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting. Draft .October 2008.

VOLATILE ORGANICS

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifications: REGION I Organics Guideline (1996)

TIER I / II / III (circle one)

SITE: Olin Chemical Project #:_____ SDG #:_____

LAB #: _____

Sample IDs: Attach tracking sheet and/or sample listing.

This checklist is designed to be used with USEPA Region I Validation Guidelines Part II (12/1996). During Level III validation, calculation and transcription checks are completed for instrument tuning, surrogates, target compounds, spike recoveries, calibration data, and internal standards as specified in the guideline. These checks are documented on attached validation notes.

YES NO NA	
Data completeness ☐ ☐ ☐ All data summaries, QC forms and raw data available from hard copy or electronic data package ☐ ☐ ☐ Data summaries match EDD	Contact lab if missing data. Lab to respond with 24 hours.
Holding Times and Preservation ☐ ☐ ☐ Hold times met (14 days with preservation) ☐ ☐ ☐ Preserved (waters HCL, soils methanol)	
Instrument Performance Check (Tune) ☐ ☐ ☐ Tune available for each 12-hour period samples were analyzed ☐ ☐ ☐ Appropriate number of significant figures reported (at least 2) ☐ ☐ ☐ Mass/Charge list (m/z) criteria met	
Initial Calibration ☐ ☐ ☐ %RSD less than or equal to 30% ☐ ☐ ☐ RRF greater than or equal to 0.05	
Continuing Calibration ☐ ☐ ☐ %D less than or equal to 25% ☐ ☐ ☐ RRF greater than or equal to 0.05.	
Blank Contamination ☐ ☐ ☐ Method blank contamination ☐ ☐ ☐ Trip blank contamination ☐ ☐ ☐ Equipment/Rinseate blank contamination	Evaluate all blanks for contamination. Highest contaminant level used for action level.
Surrogate Recoveries	

VOLATILE ORGANICS

REGION I VALIDATION CHECKLIST for
OLIN CHEMICAL SUPERFUND SITE
Criteria and Qualifications: REGION I Organics Guideline (1996)

TIER I / II / III (circle one)

☐ ☐ ☐ Surrogates percent recovery criteria met (water and soil: 70%-130%)	
Matrix Spikes and Laboratory Control Samples ☐ ☐ ☐ MS/MSD percent recovery criteria met (water and soil: 70%-130%) ☐ ☐ ☐ MS/MSD RPD criteria met (water and soil <30%) ☐ ☐ ☐ LCS percent recovery criteria (water and soil: 70%-130%)	
Field Duplicates ☐ ☐ ☐ RPD criteria (water <30%, soils <50%) met	
Internal Standard ☐ ☐ ☐ Area counts within -50 to +100 percent of calib. std. ☐ ☐ ☐ Retention Time within 30 seconds of calib. std.	
Target Compounds ☐ ☐ ☐ Reviewed narrative for anomalies	
Tentatively Identified Compounds (TICs) ☐ ☐ ☐ TCL compounds reported as TICs	

Validator's Signature:_____

Date: _____

Reference:

MACTEC, Quality Assurance Project Plan for Remedial Investigation/Feasibility Study – Olin Chemical Superfund Site, Wilmington Property, 51 Eames Street, Wilmington, MA", MACTEC Engineering and Consulting. Draft .October 2008.

APPENDIX E
LABORATORY CERTIFICATION DOCUMENTS

NELAP RECOGNIZED
ACCREDITING
nelap
AUTHORITY

FLORIDA DEPARTMENT OF
HEALTH

State of Florida
Department of Health, Bureau of Laboratories

This is to certify that

E87604

KATAHDIN ANALYTICAL SERVICES, INC.
600 TECHNOLOGY WAY
SCARBOROUGH, ME 04074

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER - MICROBIOLOGY, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, DRINKING WATER - SYNTHETIC ORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY, BIOLOGICAL TISSUE - PESTICIDES-HERBICIDES-PCB'S

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2008 THROUGH June 30, 2009

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E87604-09-07/01/2008
Supersedes all previously issued certificates

The Commonwealth of Massachusetts

Department of Environmental Protection

Division of Environmental Analysis

Senator William X. Wall Experiment Station

certifies

M-ME019

KATAHDIN ANALYTICAL SERVICES INC
600 TECHNOLOGY WAY
SCARBOROUGH, ME 04074-0000

Laboratory Director: DEBORAH J. NADEAU

for the analysis of POTABLE WATER (CHEMISTRY)
NON POTABLE WATER (CHEMISTRY)

pursuant to 310 CMR 42.00

This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.

This certificate is valid only when accompanied by the latest dated Certified Parameter List as issued by the Massachusetts D.E.P. Contact the Division of Environmental Analysis to verify the current certification status of the laboratory.

Certification is no guarantee of the validity of the data. This certification is subject to unannounced laboratory inspections.

Issued: 01 JUL 2008

Expires: 30 JUN 2009

Oscar C. Jacobs

Director, Division of Environmental Analysis

The Commonwealth of Massachusetts

Department of Environmental Protection

Division of Environmental Analysis

Senator William X. Wall Experiment Station

certifies

M-MA014

TESTAMERICA WESTFIELD
53 SOUTHAMPTON RD
WESTFIELD, MA 01085-5308

Laboratory Director: **STEVEN HARTMANN**

for the analysis of

POTABLE WATER (MICROBIOLOGY)
POTABLE WATER (CHEMISTRY)
NON POTABLE WATER (CHEMISTRY)

pursuant to 310 CMR 42.00

This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.

This certificate is valid only when accompanied by the latest dated Certified Parameter List as issued by the Massachusetts D.E.P. Contact the Division of Environmental Analysis to verify the current certification status of the laboratory.

Certification is no guarantee of the validity of the data. This certification is subject to unannounced laboratory inspections.

Issued: **01 JUL 2008**

Expires: **30 JUN 2009**

Oscar C. Parcarbo

Director, Division of Environmental Analysis

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
RICHARD F. DAINES, M.D.

Expires 12:01 AM April 01, 2010
Issued April 01, 2009

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. STEVEN HARTMANN
TESTAMERICA WESTFIELD
53 SOUTHAMPTON ROAD
WESTFIELD, MA 01085

NY Lab Id No: 10843
EPA Lab Code: MA00014

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES POTABLE WATER All approved analytes are listed below:

Drinking Water Metals I

Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Drinking Water Metals II

Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
Nickel, Total	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4

Drinking Water Metals II

Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Drinking Water Metals III

Boron, Total	EPA 200.7 Rev. 4.4
Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4

Drinking Water Miscellaneous

Methyl tert-butyl ether	EPA 524.2
Organic Carbon, Total	SM 18-21 5310B (00)

Drinking Water Non-Metals

Alkalinity	SM 18-21 2320B (97)
Calcium Hardness	EPA 200.7 Rev. 4.4
Fluoride, Total	EPA 300.0 Rev. 2.1
Hydrogen Ion (pH)	SM 18-21 4500-H B (00)
Nitrate (as N)	EPA 300.0 Rev. 2.1
Nitrite (as N)	EPA 300.0 Rev. 2.1
Orthophosphate (as P)	SM 18-21 4500-P E
Solids, Total Dissolved	SM 18-21 2540C (97)
Specific Conductance	SM 18-21 2510B (97)

Volatile Aromatics

1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2

Serial No.: 39079

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NELAP Recognized

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All approved analytes are listed below:

Volatile Aromatics

1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2

Volatile Halocarbons

1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

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NEW YORK STATE DEPARTMENT OF HEALTH
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Expires 12:01 AM April 01, 2010
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MR. STEVEN HARTMANN
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WESTFIELD, MA 01085

NY Lab Id No: 10843
EPA Lab Code: MA00014

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 624
	EPA 8260B
Acrylonitrile	EPA 624
	EPA 8260B
Amines	
2-Nitroaniline	EPA 8270C
3-Nitroaniline	EPA 8270C
4-Chloroaniline	EPA 8270C
4-Nitroaniline	EPA 8270C
Aniline	EPA 8270C
Carbazole	EPA 8270C
Pyridine	EPA 8270C
Benzidines	
3,3'-Dichlorobenzidine	EPA 625
Benzidine	EPA 625
	EPA 8270C
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 608
	EPA 8081A
4,4'-DDE	EPA 608
	EPA 8081A
4,4'-DDT	EPA 608
	EPA 8081A
Aldrin	EPA 608
	EPA 8081A
alpha-BHC	EPA 608

Chlorinated Hydrocarbon Pesticides	
alpha-BHC	EPA 8081A
alpha-Chlordane	EPA 8081A
beta-BHC	EPA 608
	EPA 8081A
Chlordane Total	EPA 608
	EPA 8081A
delta-BHC	EPA 608
	EPA 8081A
Dieldrin	EPA 608
	EPA 8081A
Endosulfan I	EPA 608
	EPA 8081A
Endosulfan II	EPA 608
	EPA 8081A
Endosulfan sulfate	EPA 608
	EPA 8081A
Endrin	EPA 608
	EPA 8081A
Endrin aldehyde	EPA 608
	EPA 8081A
Endrin Ketone	EPA 8081A
gamma-Chlordane	EPA 8081A
Heptachlor	EPA 608
	EPA 8081A
Heptachlor epoxide	EPA 608
	EPA 8081A
Lindane	EPA 608
Toxaphene	EPA 608

Serial No.: 39080

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Chlorinated Hydrocarbon Pesticides	
Toxaphene	EPA 8081A
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260B
1,2,4,5-Tetrachlorobenzene	EPA 8270C
1,2,4-Trichlorobenzene	EPA 625
	EPA 8260B
	EPA 8270C
2-Chloronaphthalene	EPA 625
Hexachlorobenzene	EPA 625
	EPA 8270C
Hexachlorobutadiene	EPA 625
	EPA 8270C
Hexachlorocyclopentadiene	EPA 625
	EPA 8270C
Hexachloroethane	EPA 625
	EPA 8270C
Demand	
Biochemical Oxygen Demand	SM 18-20 5210B (01)
Chemical Oxygen Demand	EPA 410.4 Rev. 2.0
Fuel Oxygenates	
ethyl tert-butyl ether	EPA 8260B
Methyl tert-butyl ether	EPA 8260B
tert-amyl methyl ether	EPA 8260B
Haloethers	
4-Bromophenylphenyl ether	EPA 625
	EPA 8270C
Haloethers	
4-Chlorophenylphenyl ether	EPA 625
	EPA 8270C
Bis (2-chloroisopropyl) ether	EPA 625
Bis(2-chloroethoxy)methane	EPA 625
	EPA 8270C
Bis(2-chloroethyl)ether	EPA 625
	EPA 8270C
Mineral	
Alkalinity	SM 18-21 2320B (97)
Calcium Hardness	EPA 200.7 Rev. 4.4
	SM 18-20 2340B (97)
Chloride	EPA 300.0 Rev. 2.1
Hardness, Total	EPA 200.7 Rev. 4.4
Sulfate (as SO4)	EPA 300.0 Rev. 2.1
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 625
	EPA 8270C
2,6-Dinitrotoluene	EPA 625
	EPA 8270C
Isophorone	EPA 625
	EPA 8270C
Nitrobenzene	EPA 625
	EPA 8270C
Nitrosoamines	
N-Nitrosodimethylamine	EPA 625
	EPA 8270C

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All approved analytes are listed below:

Nitrosoamines	
N-Nitrosodi-n-propylamine	EPA 625
	EPA 8270C
N-Nitrosodiphenylamine	EPA 625
	EPA 8270C
Nutrient	
Ammonia (as N)	LACHAT 10-107-06-1-B
Kjeldahl Nitrogen, Total	LACHAT 10-107-06-2
Nitrate (as N)	EPA 300.0 Rev. 2.1
	LACHAT 10-107-04-1-C
Nitrite (as N)	SM 18-21 4500-NO2 B (00)
Orthophosphate (as P)	SM 18-21 4500-P E
Phosphorus, Total	SM 18-21 4500-P E
Phthalate Esters	
Benzyl butyl phthalate	EPA 625
	EPA 8270C
Bis(2-ethylhexyl) phthalate	EPA 625
	EPA 8270C
Diethyl phthalate	EPA 625
	EPA 8270C
Dimethyl phthalate	EPA 625
	EPA 8270C
Di-n-butyl phthalate	EPA 625
	EPA 8270C
Di-n-octyl phthalate	EPA 625
	EPA 8270C
Polychlorinated Biphenyls	
PCB-1016	EPA 608
	EPA 8082
PCB-1221	EPA 608
	EPA 8082
PCB-1232	EPA 608
	EPA 8082
PCB-1242	EPA 608
	EPA 8082
PCB-1248	EPA 608
	EPA 8082
PCB-1254	EPA 608
	EPA 8082
PCB-1260	EPA 608
	EPA 8082
PCB-1262	EPA 8082
PCB-1268	EPA 8082
Polynuclear Aromatics	
Acenaphthene	EPA 625
	EPA 8270C
Acenaphthylene	EPA 625
	EPA 8270C
Anthracene	EPA 625
	EPA 8270C
Benzo(a)anthracene	EPA 625
	EPA 8270C
Benzo(a)pyrene	EPA 625
	EPA 8270C
Benzo(b)fluoranthene	EPA 625

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Polynuclear Aromatics	
Benzo(b)fluoranthene	EPA 8270C
Benzo(ghi)perylene	EPA 625
	EPA 8270C
Benzo(k)fluoranthene	EPA 625
	EPA 8270C
Chrysene	EPA 625
	EPA 8270C
Dibenzo(a,h)anthracene	EPA 625
	EPA 8270C
Fluoranthene	EPA 625
	EPA 8270C
Fluorene	EPA 625
	EPA 8270C
Indeno(1,2,3-cd)pyrene	EPA 625
	EPA 8270C
Naphthalene	EPA 625
	EPA 8260B
	EPA 8270C
Phenanthrene	EPA 625
	EPA 8270C
Pyrene	EPA 625
	EPA 8270C

Priority Pollutant Phenols	
2,4,5-Trichlorophenol	EPA 8270C
2,4,6-Trichlorophenol	EPA 625
	EPA 8270C
2,4-Dichlorophenol	EPA 625
	EPA 8270C

Priority Pollutant Phenols	
2,4-Dimethylphenol	EPA 625
	EPA 8270C
2,4-Dinitrophenol	EPA 625
	EPA 8270C
2-Chlorophenol	EPA 625
	EPA 8270C
2-Methyl-4,6-dinitrophenol	EPA 625
	EPA 8270C
2-Methylphenol	EPA 8270C
2-Nitrophenol	EPA 625
	EPA 8270C
3-Methylphenol	EPA 8270C
4-Chloro-3-methylphenol	EPA 625
	EPA 8270C
4-Methylphenol	EPA 8270C
4-Nitrophenol	EPA 625
	EPA 8270C
Cresols, Total	EPA 8270C
Pentachlorophenol	EPA 625
	EPA 8270C
Phenol	EPA 625
	EPA 8270C

Purgeable Aromatics	
1,2,4-Trimethylbenzene	EPA 8260B
1,2-Dichlorobenzene	EPA 624
	EPA 8260B
	EPA 8270C
1,3,5-Trimethylbenzene	EPA 8260B

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All approved analytes are listed below:

Purgeable Aromatics	
1,3-Dichlorobenzene	EPA 624
	EPA 8260B
	EPA 8270C
1,4-Dichlorobenzene	EPA 624
	EPA 625
	EPA 8260B
	EPA 8270C
Benzene	EPA 624
	EPA 8260B
Chlorobenzene	EPA 624
	EPA 8260B
Ethyl benzene	EPA 624
	EPA 8260B
Isopropylbenzene	EPA 8260B
n-Butylbenzene	EPA 8260B
n-Propylbenzene	EPA 8260B
p-Isopropyltoluene (P-Cymene)	EPA 8260B
sec-Butylbenzene	EPA 8260B
Styrene	EPA 8260B
Toluene	EPA 624
	EPA 8260B
Total Xylenes	EPA 624
	EPA 8260B

Purgeable Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260B
1,1,1-Trichloroethane	EPA 624
	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 624

Purgeable Halocarbons	
1,1,2,2-Tetrachloroethane	EPA 8260B
1,1,2-Trichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethane	EPA 624
	EPA 8260B
1,1-Dichloroethene	EPA 624
	EPA 8260B
1,1-Dichloropropene	EPA 8260B
1,2-Dichloroethane	EPA 624
	EPA 8260B
1,2-Dichloropropane	EPA 624
	EPA 8260B
1,3-Dichloropropane	EPA 8260B
2,2-Dichloropropane	EPA 8260B
2-Chloroethylvinyl ether	EPA 624
	EPA 8260B
Bromochloromethane	EPA 8260B
Bromodichloromethane	EPA 624
	EPA 8260B
Bromoform	EPA 624
	EPA 8260B
Bromomethane	EPA 624
	EPA 8260B
Carbon tetrachloride	EPA 624
	EPA 8260B
Chloroethane	EPA 624
	EPA 8260B
Chloroform	EPA 624

Serial No.: 39080

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NELAP Recognized

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
RICHARD F. DAINES, M.D.

Expires 12:01 AM April 01, 2010
Issued April 01, 2009

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. STEVEN HARTMANN
TESTAMERICA WESTFIELD
53 SOUTHAMPTON ROAD
WESTFIELD, MA 01085

NY Lab Id No: 10843
EPA Lab Code: MA00014

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Purgeable Halocarbons	
Chloroform	EPA 8260B
Chloromethane	EPA 624
	EPA 8260B
cis-1,2-Dichloroethene	EPA 624
	EPA 8260B
cis-1,3-Dichloropropene	EPA 624
	EPA 8260B
Dibromochloromethane	EPA 624
	EPA 8260B
Dibromomethane	EPA 8260B
Dichlorodifluoromethane	EPA 624
	EPA 8260B
Methylene chloride	EPA 624
	EPA 8260B
Tetrachloroethene	EPA 624
	EPA 8260B
trans-1,2-Dichloroethene	EPA 624
	EPA 8260B
trans-1,3-Dichloropropene	EPA 624
	EPA 8260B
trans-1,4-Dichloro-2-butene	EPA 8260B
Trichloroethene	EPA 624
	EPA 8260B
Trichlorofluoromethane	EPA 624
	EPA 8260B
Vinyl chloride	EPA 624
	EPA 8260B

Purgeable Organics	
2-Butanone (Methylethyl ketone)	EPA 8260B
2-Hexanone	EPA 8260B
4-Methyl-2-Pentanone	EPA 8260B
Acetone	EPA 8260B
Carbon Disulfide	EPA 8260B
Vinyl acetate	EPA 8260B
Residue	
Solids, Total Dissolved	SM 18-21 2540C (97)
Solids, Total Suspended	SM 18-20 2540D (97)
Semi-Volatile Organics	
2-Methylnaphthalene	EPA 8270C
Acetophenone	EPA 8270C
Benzoic Acid	EPA 8270C
Benzyl alcohol	EPA 8270C
Dibenzofuran	EPA 8270C
Wastewater Metals I	
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Calcium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

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Wastewater Metals I	
Chromium, Total	EPA 6010B
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Iron, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Lead, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Magnesium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Potassium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Sodium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Strontium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Wastewater Metals II	
Aluminum, Total	EPA 200.7 Rev. 4.4

Wastewater Metals II	
Aluminum, Total	EPA 200.8 Rev. 5.4
	EPA 6010B
Antimony, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Chromium VI	EPA 7196A
	SM 18-19 3500-Cr D
Mercury, Total	EPA 245.1 Rev. 3.0
	EPA 7470A
Selenium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Wastewater Metals III	
Cobalt, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B

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Wastewater Metals III	
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Thallium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
Titanium, Total	EPA 200.7 Rev. 4.4
Wastewater Miscellaneous	
Boron, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Cyanide, Total	EPA 9012A
	LACHAT 10-204-00-1-A
Hydrogen Ion (pH)	EPA 9040B
	SM 18-21 4500-H B (00)
Organic Carbon, Total	SM 18-21 5310B (00)
Phenols	LACHAT 10-210-00-1-A
Silica, Dissolved	EPA 6010B
Specific Conductance	SM 18-21 2510B (97)
Sulfide (as S)	SM 18 4500-S E
Sample Preparation Methods	
	EPA 3005A
	EPA 3010A
	EPA 3020A
	EPA 3510C
	EPA 5030B
	EPA 9030B

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ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved subcategories and/or analytes are listed below:

Nutrient

Nitrite (as N)	EPA 300.0 Rev. 2.1

Serial No.: 39081

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All approved analytes are listed below:

Amines	
1,2-Diphenylhydrazine	EPA 8270C
2-Nitroaniline	EPA 8270C
3-Nitroaniline	EPA 8270C
4-Chloroaniline	EPA 8270C
4-Nitroaniline	EPA 8270C
Aniline	EPA 8270C
Carbazole	EPA 8270C
Characteristic Testing	
Corrosivity	EPA 9045C
Ignitability	EPA 1010
Chlorinated Hydrocarbon Pesticides	
Aldrin	EPA 8081A
alpha-BHC	EPA 8081A
alpha-Chlordane	EPA 8081A
beta-BHC	EPA 8081A
delta-BHC	EPA 8081A
Endosulfan sulfate	EPA 8081A
Endrin	EPA 8081A
Endrin aldehyde	EPA 8081A
Endrin Ketone	EPA 8081A
gamma-Chlordane	EPA 8081A
Heptachlor	EPA 8081A
Heptachlor epoxide	EPA 8081A
Lindane	EPA 8081A
Methoxychlor	EPA 8081A
Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA 8260B
	EPA 8270C
2-Chloronaphthalene	EPA 8270C
Hexachlorobenzene	EPA 8270C
Hexachlorobutadiene	EPA 8270C
Hexachlorocyclopentadiene	EPA 8270C
Hexachloroethane	EPA 8270C
Haloethers	
4-Bromophenylphenyl ether	EPA 8270C
4-Chlorophenylphenyl ether	EPA 8270C
Bis (2-chloroisopropyl) ether	EPA 8270C
Bis(2-chloroethoxy)methane	EPA 8270C
Metals I	
Barium, Total	EPA 6010B
Cadmium, Total	EPA 6010B
Calcium, Total	EPA 6010B
Chromium, Total	EPA 6010B
Copper, Total	EPA 6010B
Iron, Total	EPA 6010B
Lead, Total	EPA 6010B
Magnesium, Total	EPA 6010B
Manganese, Total	EPA 6010B
Nickel, Total	EPA 6010B
Potassium, Total	EPA 6010B
Silver, Total	EPA 6010B
Sodium, Total	EPA 6010B

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Metals II	
Aluminum, Total	EPA 6010B
Antimony, Total	EPA 6010B
Arsenic, Total	EPA 6010B
Beryllium, Total	EPA 6010B
Chromium VI	EPA 7196A
Mercury, Total	EPA 7471A
Selenium, Total	EPA 6010B
Vanadium, Total	EPA 6010B
Zinc, Total	EPA 6010B
Metals III	
Cobalt, Total	EPA 6010B
Molybdenum, Total	EPA 6010B
Thallium, Total	EPA 6010B
Tin, Total	EPA 6010B
Miscellaneous	
Cyanide, Total	EPA 9012A
Hydrogen Ion (pH)	EPA 9045C
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 8270C
2,6-Dinitrotoluene	EPA 8270C
Isophorone	EPA 8270C
Nitrobenzene	EPA 8270C
Pyridine	EPA 8270C
Nitrosoamines	
N-Nitrosodi-n-propylamine	EPA 8270C
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270C
Bis(2-ethylhexyl) phthalate	EPA 8270C
Diethyl phthalate	EPA 8270C
Dimethyl phthalate	EPA 8270C
Di-n-butyl phthalate	EPA 8270C
Di-n-octyl phthalate	EPA 8270C
Polychlorinated Biphenyls	
PCB-1016	EPA 8082
PCB-1221	EPA 8082
PCB-1232	EPA 8082
PCB-1242	EPA 8082
PCB-1248	EPA 8082
PCB-1254	EPA 8082
PCB-1260	EPA 8082
PCB-1262	EPA 8082
PCB-1268	EPA 8082
Polynuclear Aromatic Hydrocarbons	
Acenaphthene	EPA 8270C
Acenaphthylene	EPA 8270C
Anthracene	EPA 8270C
Benzo(a)anthracene	EPA 8270C
Benzo(a)pyrene	EPA 8270C
Benzo(b)fluoranthene	EPA 8270C
Benzo(ghi)perylene	EPA 8270C
Benzo(k)fluoranthene	EPA 8270C
Chrysene	EPA 8270C
Dibenzo(a,h)anthracene	EPA 8270C

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Polynuclear Aromatic Hydrocarbons	
Fluoranthene	EPA 8270C
Fluorene	EPA 8270C
Indeno(1,2,3-cd)pyrene	EPA 8270C
Naphthalene	EPA 8260B
	EPA 8270C
Phenanthrene	EPA 8270C
Pyrene	EPA 8270C
Priority Pollutant Phenols	
2,4,5-Trichlorophenol	EPA 8270C
2,4,6-Trichlorophenol	EPA 8270C
2,4-Dichlorophenol	EPA 8270C
2,4-Dimethylphenol	EPA 8270C
2,4-Dinitrophenol	EPA 8270C
2-Chlorophenol	EPA 8270C
2-Methyl-4,6-dinitrophenol	EPA 8270C
2-Methylphenol	EPA 8270C
2-Nitrophenol	EPA 8270C
3-Methylphenol	EPA 8270C
4-Chloro-3-methylphenol	EPA 8270C
4-Methylphenol	EPA 8270C
4-Nitrophenol	EPA 8270C
Pentachlorophenol	EPA 8270C
Phenol	EPA 8270C
Purgeable Aromatics	
1,2,4-Trimethylbenzene	EPA 8260B
1,2-Dichlorobenzene	EPA 8260B
	EPA 8270C

Purgeable Aromatics	
1,3-Dichlorobenzene	EPA 8260B
	EPA 8270C
1,4-Dichlorobenzene	EPA 8260B
	EPA 8270C
2-Chlorotoluene	EPA 8260B
4-Chlorotoluene	EPA 8260B
Benzene	EPA 8260B
Bromobenzene	EPA 8260B
Chlorobenzene	EPA 8260B
Ethyl benzene	EPA 8260B
Isopropylbenzene	EPA 8260B
n-Butylbenzene	EPA 8260B
n-Propylbenzene	EPA 8260B
p-Isopropyltoluene (P-Cymene)	EPA 8260B
sec-Butylbenzene	EPA 8260B
Styrene	EPA 8260B
tert-Butylbenzene	EPA 8260B
Toluene	EPA 8260B
Total Xylenes	EPA 8260B
Purgeable Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260B
1,1,1-Trichloroethane	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 8260B
1,1,2-Trichloroethane	EPA 8260B
1,1-Dichloroethane	EPA 8260B
1,1-Dichloroethene	EPA 8260B
1,1-Dichloropropene	EPA 8260B
1,2,3-Trichloropropane	EPA 8260B

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Purgeable Halocarbons	
1,2-Dibromo-3-chloropropane	EPA 8260B
1,2-Dichloroethane	EPA 8260B
1,2-Dichloropropane	EPA 8260B
1,3-Dichloropropane	EPA 8260B
2,2-Dichloropropane	EPA 8260B
2-Chloroethylvinyl ether	EPA 8260B
Bromochloromethane	EPA 8260B
Bromodichloromethane	EPA 8260B
Bromoform	EPA 8260B
Bromomethane	EPA 8260B
Carbon tetrachloride	EPA 8260B
Chloroethane	EPA 8260B
Chloroform	EPA 8260B
Chloromethane	EPA 8260B
cis-1,2-Dichloroethene	EPA 8260B
cis-1,3-Dichloropropene	EPA 8260B
Dibromochloromethane	EPA 8260B
Dibromomethane	EPA 8260B
Dichlorodifluoromethane	EPA 8260B
Methylene chloride	EPA 8260B
Tetrachloroethene	EPA 8260B
trans-1,3-Dichloropropene	EPA 8260B
trans-1,4-Dichloro-2-butene	EPA 8260B
Trichloroethene	EPA 8260B
Trichlorofluoromethane	EPA 8260B
Vinyl chloride	EPA 8260B
Purgeable Organics	
1,4-Dioxane	EPA 8260B

Purgeable Organics	
2-Butanone (Methylethyl ketone)	EPA 8260B
2-Hexanone	EPA 8260B
4-Methyl-2-Pentanone	EPA 8260B
Acetone	EPA 8260B
Acetonitrile	EPA 8260B
Carbon Disulfide	EPA 8260B
Methyl tert-butyl ether	EPA 8260B
tert-Butyl alcohol	EPA 8260B
Vinyl acetate	EPA 8260B
Semi-Volatile Organics	
2-Methylnaphthalene	EPA 8270C
Acetophenone	EPA 8270C
Benzoic Acid	EPA 8270C
Benzyl alcohol	EPA 8270C
Dibenzofuran	EPA 8270C
Sample Preparation Methods	
	EPA 3020A
	EPA 3050B
	EPA 3550B
	EPA 5035

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Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081A
4,4'-DDE	EPA 8081A
4,4'-DDT	EPA 8081A
Dieldrin	EPA 8081A
Endosulfan I	EPA 8081A
Endosulfan II	EPA 8081A

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NELAP RECOGNIZED
ACCREDITING
nelap
AUTHORITY

FLORIDA DEPARTMENT OF
HEALTH

State of Florida
Department of Health, Bureau of Laboratories

This is to certify that

E81005

TESTAMERICA TALLAHASSEE
2846 INDUSTRIAL PLAZA DRIVE
TALLAHASSEE, FL 32301

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, DRINKING WATER - SYNTHETIC ORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2008 THROUGH June 30, 2009

GREAT SEAL OF THE STATE OF FLORIDA
IN GOD WE TRUST

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E81005-18-07/01/2008
Supersedes all previously issued certificates

NELAP - RECOGNIZED

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM BRANCH

CERTIFICATE OF NELAP ACCREDITATION

Is hereby granted to

TESTAMERICA WEST SACRAMENTO

880 RIVERSIDE PARKWAY

WEST SACRAMENTO, CA 95605

Scope of the Certificate is limited to the
"NELAP Fields of Accreditation"
which accompany this Certificate.

Continued accredited status depends on successful
ongoing participation in the program.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **01119CA**

Expiration Date: **01/31/2010**

Effective Date: **01/31/2009**

Richmond, California
subject to forfeiture or revocation

George C. Kulasingam, Ph.D., Chief
Environmental Laboratory Accreditation Program Branch

CALIFORNIA DEPARTMENT OF PUBLIC HEALTH

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM - NELAP RECOGNIZED

NELAP Fields of Accreditation

TESTAMERICA WEST SACRAMENTO **Lab Phone** (916) 373-5600

880 RIVERSIDE PARKWAY
WEST SACRAMENTO, CA 95605

Certificate No: 01119CA Renew Date: 01/31/2010

102 - Inorganic Chemistry of Drinking Water			
102.045	001	EPA 314.0	Perchlorate
102.047	001	EPA 331.0	Perchlorate
102.510	006	SM3120B	Hardness (calc.)
102.520	001	EPA 200.7	Calcium
102.520	002	EPA 200.7	Magnesium
102.520	003	EPA 200.7	Potassium
102.520	004	EPA 200.7	Silica
102.520	005	EPA 200.7	Sodium
102.520	006	EPA 200.7	Hardness (calc.)
103 - Toxic Chemical Elements of Drinking Water			
103.130	001	EPA 200.7	Aluminum
103.130	003	EPA 200.7	Barium
103.130	004	EPA 200.7	Beryllium
103.130	005	EPA 200.7	Cadmium
103.130	007	EPA 200.7	Chromium
103.130	008	EPA 200.7	Copper
103.130	009	EPA 200.7	Iron
103.130	011	EPA 200.7	Manganese
103.130	012	EPA 200.7	Nickel
103.130	015	EPA 200.7	Silver
103.130	017	EPA 200.7	Zinc
103.140	001	EPA 200.8	Aluminum
103.140	002	EPA 200.8	Antimony
103.140	003	EPA 200.8	Arsenic
103.140	004	EPA 200.8	Barium
103.140	005	EPA 200.8	Beryllium
103.140	006	EPA 200.8	Cadmium
103.140	007	EPA 200.8	Chromium
103.140	008	EPA 200.8	Copper
103.140	009	EPA 200.8	Lead
103.140	010	EPA 200.8	Manganese
103.140	011	EPA 200.8	Mercury
103.140	012	EPA 200.8	Nickel

103.140	013	EPA 200.8	Selenium
103.140	014	EPA 200.8	Silver
103.140	015	EPA 200.8	Thallium
103.140	016	EPA 200.8	Zinc
103.160	001	EPA 245.1	Mercury
105 - Semi-volatile Organic Chemistry of Drinking Water			
105.230	000	EPA 1613	Dioxins
105.230	001	EPA 1613	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
108 - Inorganic Chemistry of Wastewater			
108.020	001	EPA 120.1	Conductivity
108.090	001	EPA 160.4	Residue, Volatile
108.110	001	EPA 180.1	Turbidity
108.112	001	EPA 200.7	Boron
108.112	002	EPA 200.7	Calcium
108.112	003	EPA 200.7	Hardness (calc.)
108.112	004	EPA 200.7	Magnesium
108.112	005	EPA 200.7	Potassium
108.112	006	EPA 200.7	Silica
108.112	007	EPA 200.7	Sodium
108.120	001	EPA 300.0	Bromide
108.120	002	EPA 300.0	Chloride
108.120	003	EPA 300.0	Fluoride
108.120	004	EPA 300.0	Nitrate
108.120	005	EPA 300.0	Nitrite
108.120	006	EPA 300.0	Nitrate-nitrite
108.120	007	EPA 300.0	Phosphate, Ortho
108.120	008	EPA 300.0	Sulfate
108.141	001	EPA 310.2	Alkalinity
108.183	001	EPA 335.4	Cyanide, Total
108.200	001	EPA 350.1	Ammonia
108.211	001	EPA 351.2	Kjeldahl Nitrogen
108.232	001	EPA 353.2	Nitrate-nitrite
108.232	002	EPA 353.2	Nitrite
108.264	001	EPA 365.3	Phosphate, Ortho
108.265	001	EPA 365.3	Phosphorus, Total
108.266	001	EPA 365.4	Phosphorus, Total
108.323	001	EPA 410.4	Chemical Oxygen Demand
108.362	001	EPA 420.4	Phenols, Total
108.381	001	EPA 1664A	Oil and Grease
108.410	001	SM2320B	Alkalinity
108.420	001	SM2340B	Hardness (calc.)

108.430	001	SM2510B	Conductivity
108.440	001	SM2540B	Residue, Total
108.441	001	SM2540C	Residue, Filterable
108.442	001	SM2540D	Residue, Non-filterable
108.443	001	SM2540F	Residue, Settleable
108.472	001	SM4500-CN E	Cyanide, Total
108.473	001	SM4500-CN G	Cyanide, amenable
108.490	001	SM4500-H+ B	pH
108.580	001	SM4500-S= D	Sulfide
108.611	001	SM5310C	Total Organic Carbon
109 - Toxic Chemical Elements of Wastewater			
109.010	001	EPA 200.7	Aluminum
109.010	002	EPA 200.7	Antimony
109.010	003	EPA 200.7	Arsenic
109.010	004	EPA 200.7	Barium
109.010	005	EPA 200.7	Beryllium
109.010	007	EPA 200.7	Cadmium
109.010	009	EPA 200.7	Chromium
109.010	010	EPA 200.7	Cobalt
109.010	011	EPA 200.7	Copper
109.010	012	EPA 200.7	Iron
109.010	013	EPA 200.7	Lead
109.010	015	EPA 200.7	Manganese
109.010	016	EPA 200.7	Molybdenum
109.010	017	EPA 200.7	Nickel
109.010	019	EPA 200.7	Selenium
109.010	021	EPA 200.7	Silver
109.010	023	EPA 200.7	Thallium
109.010	024	EPA 200.7	Tin
109.010	025	EPA 200.7	Titanium
109.010	026	EPA 200.7	Vanadium
109.010	027	EPA 200.7	Zinc
109.020	001	EPA 200.8	Aluminum
109.020	002	EPA 200.8	Antimony
109.020	003	EPA 200.8	Arsenic
109.020	004	EPA 200.8	Barium
109.020	005	EPA 200.8	Beryllium
109.020	006	EPA 200.8	Cadmium
109.020	007	EPA 200.8	Chromium
109.020	008	EPA 200.8	Cobalt
109.020	009	EPA 200.8	Copper

109.020	010	EPA 200.8	Lead
109.020	011	EPA 200.8	Manganese
109.020	012	EPA 200.8	Molybdenum
109.020	013	EPA 200.8	Nickel
109.020	014	EPA 200.8	Selenium
109.020	015	EPA 200.8	Silver
109.020	016	EPA 200.8	Thallium
109.020	017	EPA 200.8	Vanadium
109.020	018	EPA 200.8	Zinc
109.190	001	EPA 245.1	Mercury
111 - Semi-volatile Organic Chemistry of Wastewater			
111.111	000	EPA 1613B	Dioxins
111.111	001	EPA 1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
111.111	002	EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
111.111	003	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	004	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	005	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	006	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
111.111	007	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
111.111	008	EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
111.111	009	EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
111.111	010	EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
111.111	011	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	012	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	013	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
111.111	014	EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	015	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
111.111	016	EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
111.111	017	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
111.111	018	EPA 1613B	Total TCDD
111.111	019	EPA 1613B	Total PeCDD
111.111	020	EPA 1613B	Total HxCDD
111.111	021	EPA 1613B	Total HpCDD
111.111	022	EPA 1613B	Total TCDF
111.111	023	EPA 1613B	Total PeCDF
111.111	024	EPA 1613B	Total HxCDF
111.111	025	EPA 1613B	Total HpCDF
111.273	001	EPA 1664A	Oil and Grease
114 - Inorganic Chemistry of Hazardous Waste			
114.010	001	EPA 6010B	Antimony
114.010	002	EPA 6010B	Arsenic

114.010	003	EPA 6010B	Barium
114.010	004	EPA 6010B	Beryllium
114.010	005	EPA 6010B	Cadmium
114.010	006	EPA 6010B	Chromium
114.010	007	EPA 6010B	Cobalt
114.010	008	EPA 6010B	Copper
114.010	009	EPA 6010B	Lead
114.010	010	EPA 6010B	Molybdenum
114.010	011	EPA 6010B	Nickel
114.010	012	EPA 6010B	Selenium
114.010	013	EPA 6010B	Silver
114.010	014	EPA 6010B	Thallium
114.010	015	EPA 6010B	Vanadium
114.010	016	EPA 6010B	Zinc
114.010	026	EPA 6010B	Silica
114.010	027	EPA 6010B	Sodium
114.020	001	EPA 6020	Antimony
114.020	002	EPA 6020	Arsenic
114.020	003	EPA 6020	Barium
114.020	004	EPA 6020	Beryllium
114.020	005	EPA 6020	Cadmium
114.020	006	EPA 6020	Chromium
114.020	007	EPA 6020	Cobalt
114.020	008	EPA 6020	Copper
114.020	009	EPA 6020	Lead
114.020	010	EPA 6020	Molybdenum
114.020	011	EPA 6020	Nickel
114.020	012	EPA 6020	Selenium
114.020	013	EPA 6020	Silver
114.020	014	EPA 6020	Thallium
114.020	015	EPA 6020	Vanadium
114.020	016	EPA 6020	Zinc
114.103	001	EPA 7196A	Chromium (VI)
114.140	001	EPA 7470A	Mercury
114.141	001	EPA 7471A	Mercury
114.221	001	EPA 9012A	Cyanide, Total
114.240	001	EPA 9040B	Corrosivity - pH Determination
114.241	001	EPA 9045C	Corrosivity - pH Determination
114.250	001	EPA 9056	Fluoride
114.270	001	EPA 9214	Fluoride

115 - Extraction Test of Hazardous Waste

115.021	001	EPA 1311	TCLP Inorganics
115.022	001	EPA 1311	TCLP Extractables
115.030	001	CCR Chapter11, Article 5, Appendix II	Waste Extraction Test (WET)
116 - Volatile Organic Chemistry of Hazardous Waste			
116.080	001	EPA 8260B	Acetone
116.080	003	EPA 8260B	Acrolein
116.080	004	EPA 8260B	Acrylonitrile
116.080	005	EPA 8260B	Allyl Alcohol
116.080	006	EPA 8260B	Allyl Chloride
116.080	007	EPA 8260B	Benzene
116.080	010	EPA 8260B	Bromochloromethane
116.080	011	EPA 8260B	Bromodichloromethane
116.080	012	EPA 8260B	Bromoform
116.080	013	EPA 8260B	Bromomethane
116.080	015	EPA 8260B	Carbon Disulfide
116.080	016	EPA 8260B	Carbon Tetrachloride
116.080	018	EPA 8260B	Chlorobenzene
116.080	019	EPA 8260B	Chloroethane
116.080	020	EPA 8260B	2-Chloroethyl Vinyl Ether
116.080	021	EPA 8260B	Chloroform
116.080	022	EPA 8260B	Chloromethane
116.080	023	EPA 8260B	Chloroprene
116.080	026	EPA 8260B	Dibromochloromethane
116.080	027	EPA 8260B	Dibromochloropropane
116.080	028	EPA 8260B	1,2-Dibromoethane
116.080	029	EPA 8260B	Dibromofluoromethane
116.080	030	EPA 8260B	Dibromomethane
116.080	031	EPA 8260B	1,2-Dichlorobenzene
116.080	032	EPA 8260B	1,3-Dichlorobenzene
116.080	033	EPA 8260B	1,4-Dichlorobenzene
116.080	035	EPA 8260B	trans-1,4-Dichloro-2-butene
116.080	036	EPA 8260B	Dichlorodifluoromethane
116.080	037	EPA 8260B	1,1-Dichloroethane
116.080	038	EPA 8260B	1,2-Dichloroethane
116.080	039	EPA 8260B	1,1-Dichloroethene
116.080	040	EPA 8260B	trans-1,2-Dichloroethene
116.080	041	EPA 8260B	cis-1,2-Dichloroethene
116.080	042	EPA 8260B	1,2-Dichloropropane
116.080	043	EPA 8260B	1,3-Dichloropropane
116.080	044	EPA 8260B	2,2-Dichloropropane
116.080	045	EPA 8260B	1,1-Dichloropropene

116.080	046	EPA 8260B	cis-1,3-Dichloropropene
116.080	047	EPA 8260B	trans-1,3-Dichloropropene
116.080	050	EPA 8260B	1,4-Dioxane
116.080	053	EPA 8260B	Ethylbenzene
116.080	055	EPA 8260B	Ethyl Methacrylate
116.080	056	EPA 8260B	Hexachlorobutadiene
116.080	058	EPA 8260B	2-Hexanone (MBK)
116.080	059	EPA 8260B	Iodomethane
116.080	060	EPA 8260B	Isobutyl Alcohol
116.080	062	EPA 82608	Methacrylonitrile
116.080	064	EPA 8260B	Methyl tert-butyl Ether (MTBE)
116.080	065	EPA 8260B	Methylene Chloride
116.080	066	EPA 8260B	Methyl Ethyl Ketone
116.080	067	EPA 8260B	Methyl Methacrylate
116.080	068	EPA 8260B	4-Methyl-2-pentanone (MIBK)
116.080	069	EPA 8260B	Naphthalene
116.080	078	EPA 8260B	Propionitrile
116.080	081	EPA 8260B	1,1,1,2-Tetrachloroethane
116.080	082	EPA 8260B	1,1,2,2-Tetrachloroethane
116.080	083	EPA 8260B	Tetrachloroethene
116.080	084	EPA 8260B	Toluene
116.080	086	EPA 8260B	1,2,3-Trichlorobenzene
116.080	087	EPA 8260B	1,2,4-Trichlorobenzene
116.080	088	EPA 8260B	1,1,1-Trichloroethane
116.080	089	EPA 8260B	1,1,2-Trichloroethane
116.080	090	EPA 8260B	Trichloroethene
116.080	091	EPA 8260B	Trichlorofluoromethane
116.080	092	EPA 8260B	1,2,3-Trichloropropane
116.080	093	EPA 8260B	Vinyl Acetate
116.080	094	EPA 8260B	Vinyl Chloride
116.080	095	EPA 8260B	Xylenes, Total
116.080	096	EPA 8260B	tert-Amyl Methyl Ether (TAME)
116.080	097	EPA 8260B	tert-Butyl Alcohol (TBA)
116.080	098	EPA 8260B	Ethyl tert-butyl Ether (ETBE)
116.080	099	EPA 8260B	Bromobenzene
116.080	100	EPA 8260B	n-Butylbenzene
116.080	101	EPA 8260B	sec-Butylbenzene
116.080	102	EPA 8260B	tert-Butylbenzene
116.080	103	EPA 8260B	2-Chlorotoluene
116.080	104	EPA 8260B	4-Chlorotoluene
116.080	105	EPA 8260B	Isopropylbenzene

116.080	106	EPA 8260B	N-propylbenzene
116.080	107	EPA 8260B	Styrene
116.080	108	EPA 8260B	1,2,4-Trimethylbenzene
116.080	109	EPA 8260B	1,3,5-Trimethylbenzene
116.080	120	EPA 8260B	Oxygenates
116.100	001	LUFT GC/MS	Total Petroleum Hydrocarbons - Gasoline
116.100	002	LUFT GC/MS	Benzene
116.100	003	LUFT GC/MS	Toluene
116.100	004	LUFT GC/MS	Xylenes
116.100	005	LUFT GC/MS	Methyl tert-butyl Ether (MTBE)
116.100	010	LUFT GC/MS	BTEX and MT8E

117 - Semi-volatile Organic Chemistry of Hazardous Waste

117.010	001	EPA 8015B	Diesel-range Total Petroleum Hydrocarbons
117.016	001	LUFT	Diesel-range Total Petroleum Hydrocarbons
117.110	000	EPA 8270C	Extractable Organics
117.110	001	EPA 8270C	Acenaphthene
117.110	002	EPA 8270C	Acenaphthylene
117.110	003	EPA 8270C	Acetophenone
117.110	004	EPA 8270C	2-Acetylaminofluorene
117.110	006	EPA 8270C	4-Aminobiphenyl
117.110	007	EPA 8270C	Aniline
117.110	008	EPA 8270C	Anthracene
117.110	009	EPA 8270C	Aramite
117.110	010	EPA 8270C	Benzidine
117.110	011	EPA 8270C	Benz(a)anthracene
117.110	012	EPA 8270C	Benzo(b)fluoranthene
117.110	013	EPA 8270C	Benzo(k)fluoranthene
117.110	014	EPA 8270C	Benzo(g,h,i)perylene
117.110	015	EPA 8270C	Benzo(a)pyrene
117.110	016	EPA 8270C	Benzoic Acid
117.110	018	EPA 8270C	Benzyl Alcohol
117.110	019	EPA 8270C	Benzyl Butyl Phthalate
117.110	020	EPA 8270C	Bis(2-chloroethoxy)methane
117.110	021	EPA 8270C	Bis(2-chloroethyl) Ether
117.110	022	EPA 8270C	Bis(2-chloroisopropyl) Ether
117.110	023	EPA 8270C	Di(2-ethylhexyl) Phthalate
117.110	024	EPA 8270C	4-Bromophenyl Phenyl Ether
117.110	025	EPA 8270C	Carbazole
117.110	026	EPA 8270C	4-Chloroaniline
117.110	027	EPA 8270C	4-Chloro-3-methylphenol
117.110	028	EPA 8270C	1-Chloronaphthalene

117.110	029	EPA 8270C	2-Chloronaphthalene
117.110	030	EPA 8270C	2-Chlorophenol
117.110	031	EPA 8270C	4-Chlorophenyl Phenyl Ether
117.110	032	EPA 8270C	Chrysene
117.110	035	EPA 8270C	Dibenz(a,j)acridine
117.110	036	EPA 8270C	Dibenz(a,h)anthracene
117.110	037	EPA 8270C	Dibenzofuran
117.110	039	EPA 8270C	1,2-Dichlorobenzene
117.110	040	EPA 8270C	1,3-Dichlorobenzene
117.110	041	EPA 8270C	1,4-Dichlorobenzene
117.110	042	EPA 8270C	3,3'-Dichlorobenzidine
117.110	043	EPA 8270C	2,4-Dichlorophenol
117.110	044	EPA 8270C	2,6-Dichlorophenol
117.110	045	EPA 8270C	Diethyl Phthalate
117.110	050	EPA 8270C	p-Dimethylaminoazobenzene
117.110	051	EPA 8270C	7,12-Dimethylbenz(a)anthracene
117.110	052	EPA 8270C	a,a-Dimethylphenethylamine
117.110	053	EPA 8270C	2,4-Dimethylphenol
117.110	054	EPA 8270C	Dimethyl Phthalate
117.110	055	EPA 8270C	Di-n-butyl phthalate
117.110	056	EPA 8270C	Di-n-octyl phthalate
117.110	058	EPA 8270C	1,3-Dinitrobenzene
117.110	059	EPA 8270C	1,4-Dinitrobenzene
117.110	060	EPA 8270C	2,4-Dinitrophenol
117.110	061	EPA 8270C	2,4-Dinitrotoluene
117.110	062	EPA 8270C	2,6-Dinitrotoluene
117.110	063	EPA 8270C	Diphenylamine
117.110	064	EPA 8270C	1,2-Diphenylhydrazine
117.110	066	EPA 8270C	Ethyl Methanesulfonate
117.110	067	EPA 8270C	Fluoranthene
117.110	068	EPA 8270C	Fluorene
117.110	069	EPA 8270C	Hexachlorobenzene
117.110	070	EPA 8270C	Hexachlorobutadiene
117.110	071	EPA 8270C	Hexachlorocyclopentadiene
117.110	072	EPA 8270C	Hexachloroethane
117.110	074	EPA 8270C	Hexachloropropene
117.110	075	EPA 8270C	Indeno(1,2,3-c,d)pyrene
117.110	076	EPA 8270C	Isophorone
117.110	077	EPA 8270C	Isosafrole
117.110	079	EPA 8270C	3-Methylcholanthrene
117.110	080	EPA 8270C	2-Methyl-4,6-dinitrophenol

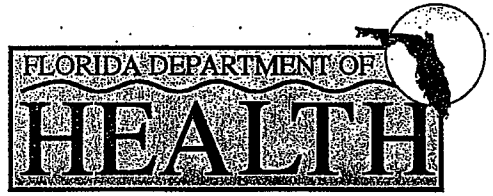
117.110	082	EPA 8270C	Methyl Methanesulfonate
117.110	083	EPA 8270C	2-Methylnaphthalene
117.110	084	EPA 8270C	2-Methylphenol
117.110	085	EPA 8270C	3-Methylphenol
117.110	086	EPA 8270C	4-Methylphenol
117.110	087	EPA 8270C	Naphthalene
117.110	088	EPA 8270C	1,4-Naphthoquinone
117.110	089	EPA 8270C	1-Naphthylamine
117.110	090	EPA 8270C	2-Naphthylamine
117.110	092	EPA 8270C	2-Nitroaniline
117.110	093	EPA 8270C	3-Nitroaniline
117.110	094	EPA 8270C	4-Nitroaniline
117.110	095	EPA 8270C	Nitrobenzene
117.110	096	EPA 8270C	2-Nitrophenol
117.110	097	EPA 8270C	4-Nitrophenol
117.110	098	EPA 8270C	N-nitrosodi-n-butylamine
117.110	099	EPA 8270C	N-nitrosodiethylamine
117.110	100	EPA 8270C	N-nitrosodimethylamine
117.110	101	EPA 8270C	N-nitrosodi-n-propylamine
117.110	102	EPA 8270C	N-nitrosodiphenylamine
117.110	103	EPA 8270C	N-nitrosomethylethylamine
117.110	104	EPA 8270C	N-nitrosomorpholine
117.110	105	EPA 8270C	N-nitrosopiperidine
117.110	106	EPA 8270C	N-nitrosopyrrolidine
117.110	107	EPA 8270C	5-Nitro-o-toluidine
117.110	108	EPA 8270C	Pentachlorobenzene
117.110	109	EPA 8270C	Pentachloronitrobenzene
117.110	110	EPA 8270C	Pentachlorophenol
117.110	111	EPA 8270C	Phenacetin
117.110	112	EPA 8270C	Phenanthrene
117.110	113	EPA 8270C	Phenol
117.110	114	EPA 8270C	1,4-Phenylenediamine
117.110	116	EPA 8270C	2-Picoline
117.110	119	EPA 8270C	Pyrene
117.110	120	EPA 8270C	Pyridine
117.110	122	EPA 8270C	Safrole
117.110	124	EPA 8270C	1,2,4,5-Tetrachlorobenzene
117.110	125	EPA 8270C	2,3,4,6-Tetrachlorophenol
117.110	128	EPA 8270C	o-Toluidine
117.110	129	EPA 8270C	1,2,4-Trichlorobenzene
117.110	130	EPA 8270C	2,4,5-Trichlorophenol

117.110	131	EPA 8270C	2,4,6-Trichlorophenol
117.110	132	EPA 8270C	1,3,5-Trinitrobenzene
117.111	015	EPA 8270C	Chlorobenzilate
117.111	021	EPA 8270C	Diallate
117.111	025	EPA 8270C	Dimethoate
117.111	039	EPA 8270C	Isodrin
117.111	054	EPA 8270C	Parathion Ethyl
117.111	055	EPA 8270C	Parathion Methyl
117.111	056	EPA 8270C	Phorate
117.111	058	EPA 8270C	Sulfotepp
117.111	061	EPA 8270C	O,O,O-triethyl Phosphorothioate
117.111	062	EPA 8270C	Trifluralin
117.111	073	EPA 8270C	Polynuclear Aromatic Hydrocarbons
117.111	074	EPA 8270C	Adipates
117.111	075	EPA 8270C	Phthalates
117.111	076	EPA 8270C	Other Extractables
117.120	000	EPA 8280A	Dioxins and Dibenzofurans
117.120	001	EPA 8280A	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
117.120	002	EPA 8280A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.120	003	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	004	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	005	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	006	EPA 8280A	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.120	007	EPA 8280A	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.120	008	EPA 8280A	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.120	009	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	010	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	011	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.120	012	EPA 8280A	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	013	EPA 8280A	Total TCDD
117.120	014	EPA 8280A	Total PeCDD
117.120	015	EPA 8280A	Total HxCDD
117.120	016	EPA 8280A	Total TCDF
117.120	017	EPA 8280A	Total PeCDF
117.120	018	EPA 8280A	Total HxCDF
117.120	019	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.120	020	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.120	021	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.120	022	EPA 8280A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.120	023	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
117.120	024	EPA 8280A	Total HpCDD

117.120	025	EPA 8280A	Total HpCDF
117.130	000	EPA 8290	Dioxins and Dibenzofurans
117.130	001	EPA 8290	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
117.130	002	EPA 8290	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.130	003	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	004	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	005	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	006	EPA 8290	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.130	007	EPA 8290	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.130	008	EPA 8290	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.130	009	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	010	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	011	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.130	012	EPA 8290	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	013	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.130	014	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.130	015	EPA 8290	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.130	016	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.130	017	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
117.140	000	EPA 8310	Polynuclear Aromatic Hydrocarbons
117.140	001	EPA 8310	Acenaphthene
117.140	002	EPA 8310	Acenaphthylene
117.140	003	EPA 8310	Anthracene
117.140	004	EPA 8310	Benz(a)anthracene
117.140	005	EPA 8310	Benzo(a)pyrene
117.140	006	EPA 8310	Benzo(b)fluoranthene
117.140	007	EPA 8310	Benzo(k)fluoranthene
117.140	008	EPA 8310	Benzo(g,h,i)perylene
117.140	009	EPA 8310	Chrysene
117.140	010	EPA 8310	Dibenz(a,h)anthracene
117.140	011	EPA 8310	Fluoranthene
117.140	012	EPA 8310	Fluorene
117.140	013	EPA 8310	Indeno(1,2,3-c,d)pyrene
117.140	014	EPA 8310	Naphthalene
117.140	015	EPA 8310	Phenanthrene
117.140	016	EPA 8310	Pyrene
117.170	000	EPA 8330	Nitroaromatics and Nitramines
117.170	001	EPA 8330	4-Amino-2,6-dinitrotoluene
117.170	002	EPA 8330	2-Amino-4,6-dinitrotoluene
117.170	003	EPA 8330	1,3-Dinitrobenzene
117.170	004	EPA 8330	2,4-Dinitrotoluene

117.170	005	EPA 8330	2,6-Dinitrotoluene
117.170	006	EPA 8330	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
117.170	007	EPA 8330	Methyl-2,4,6-trinitrophenylnitramine
117.170	008	EPA 8330	Nitrobenzene
117.170	009	EPA 8330	2-Nitrotoluene
117.170	010	EPA 8330	3-Nitrotoluene
117.170	011	EPA 8330	4-Nitrotoluene
117.170	012	EPA 8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
117.170	013	EPA 8330	1,3,5-Trinitrobenzene
117.170	014	EPA 8330	2,4,6-Trinitrotoluene
117.171	000	EPA 8330A	Nitroaromatics and Nitramines
117.171	001	EPA 8330A	4-Amino-2,6-dinitrotoluene
117.171	002	EPA 8330A	2-Amino-4,6-dinitrotoluene
117.171	003	EPA 8330A	1,3-Dinitrobenzene
117.171	004	EPA 8330A	2,4-Dinitrotoluene
117.171	005	EPA 8330A	2,6-Dinitrotoluene
117.171	006	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
117.171	007	EPA 8330A	Methyl-2,4,6-trinitrophenylnitramine
117.171	008	EPA 8330A	Nitrobenzene
117.171	009	EPA 8330A	2-Nitrotoluene
117.171	010	EPA 8330A	3-Nitrotoluene
117.171	011	EPA 8330A	4-Nitrotoluene
117.171	012	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
117.171	013	EPA 8330A	1,3,5-Trinitrobenzene
117.171	014	EPA 8330A	2,4,6-Trinitrotoluene
117.210	000	EPA 8081A	Organochlorine Pesticides
117.210	001	EPA 8081A	Aldrin
117.210	002	EPA 8081A	a-BHC
117.210	003	EPA 8081A	b-BHC
117.210	004	EPA 8081A	d-BHC
117.210	005	EPA 8081A	g-BHC (Lindane)
117.210	006	EPA 8081A	Captafol
117.210	007	EPA 8081A	a-Chlordane
117.210	008	EPA 8081A	g-Chlordane
117.210	009	EPA 8081A	Chlordane (tech.)
117.210	010	EPA 8081A	Chlorobenzilate
117.210	013	EPA 8081A	4,4'-DDD
117.210	014	EPA 8081A	4,4'-DDE
117.210	015	EPA 8081A	4,4'-DDT
117.210	016	EPA 8081A	Diallate
117.210	020	EPA 8081A	Dieldrin

117.210	021	EPA 8081A	Endosulfan I
117.210	022	EPA 8081A	Endosulfan II
117.210	023	EPA 8081A	Endosulfan Sulfate
117.210	024	EPA 8081A	Endrin
117.210	025	EPA 8081A	Endrin Aldehyde
117.210	026	EPA 8081A	Endrin Ketone
117.210	027	EPA 8081A	Heptachlor
117.210	028	EPA 8081A	Heptachlor Epoxide
117.210	031	EPA 8081A	Isodrin
117.210	033	EPA 8081A	Methoxychlor
117.210	039	EPA 8081A	Toxaphene
117.220	000	EPA 8082	PCBs
117.220	001	EPA 8082	PCB-1016
117.220	002	EPA 8082	PCB-1221
117.220	003	EPA 8082	PCB-1232
117.220	004	EPA 8082	PCB-1242
117.220	005	EPA 8082	PCB-1248
117.220	006	EPA 8082	PCB-1254
117.220	007	EPA 8082	PCB-1260



State of Florida

Department of Health, Bureau of Laboratories

This is to certify that

E87667

TESTAMERICA DENVER
4955 YARROW STREET
ARVADA, CO 80002

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - MICROBIOLOGY, DRINKING WATER - PESTICIDES-HERBICIDES-PCB'S, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2008 THROUGH June 30, 2009

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E87667-12-07/01/2008
Supersedes all previously issued certificates