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APPENDIX 15

STANDARD OPERATING PROCEDURE

NORTHEAST ANALYTICAL, INC.

NE205_01.SOP

REVISION NUMBER: 01

**STANDARD OPERATING PROCEDURE
FOR THE PREPARATION AND ANALYSIS OF SAMPLES FOR TOTAL
ORGANIC CARBON BY US-EPA LLOYD KAHN METHOD
AND TEKMAR-DOHRMANN APPLICATION NOTE TOC-011,**

AUGUST 20, 2002

COPY #

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STANDARD OPERATING PROCEDURE

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Northeast Analytical, Inc.
Issuing section: Inorganics Laboratory
SOP Name: NE205_01.DOC
Date: 8/20/2002
Revision: 01

1.0 TITLE Standard Operating Procedure for the preparation and analysis of samples for Total Organic Carbon (TOC) according to Determination of Total Organic Carbon in sediment, Lloyd Kahn, U.S.E.P.A. Region II, Edison NJ 1988 and Tekmar-Dohrmann application note TOC-011.

2.0 PURPOSE

- 2.1 Procedures for the preparation and analyses of aqueous samples for particulate organic carbon by the use of a Boat Sampler Module are provided.
- 2.2 Procedures for the preparation and analyses of solid samples for total organic carbon by the use of a Boat Sampler Module are provided.

3.0 SCOPE/APPLICABLE MATRICES

- 3.1 Methods in this procedure are used for soils, sediments, and solids for total organic carbon.

4.0 SUMMARY OF METHOD

- 4.1 Principle: Depending upon the configuration, TOC can be measured by ultra-violet promoted persulfate oxidation or high-temperature combustion, followed by infrared detection. The combustion method is used for solid samples.
- 4.2 Organic carbon is converted to carbon dioxide (CO₂) by catalytic combustion or wet chemical oxidation. The CO₂ formed can be measured directly by an infrared detector. The amount of CO₂ is directly proportional to the concentration of carbonaceous material in the sample.
- 4.3 The fractions of total carbon (TC) are defined as:
 - 4.3.1 inorganic carbon (IC)-the carbonate, bicarbonate, and dissolved CO₂;
 - 4.3.2 total organic carbon (TOC)-all carbon atoms covalently bonded in organic molecules;
 - 4.3.3 dissolved organic carbon (DOC)-the fraction of TOC that passes through a 0.45- μ m -pore-diameter filter,
 - 4.3.4 particulate organic carbon (POC)-also referred to as non dissolved organic carbon, the fraction of TOC retained by a 0.45- μ m filter.

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- 4.4 TOC and POC in solid and sludge can be measured by utilizing the combustion-infrared method. The sample is homogenized and treated with acid and then heated to remove IC. The treated sample is placed into a heated reaction chamber packed with an oxidative catalyst such as cobalt oxide. The organic carbon is oxidized to CO₂ and H₂O. The sludge and sediment sampler combusts samples at 800°C in an oxygen atmosphere so that solids as well as liquids can be analyzed.
- 4.5 The sampler consists of a magnetically coupled boat inlet system that delivers the sample to the high temperature furnace. Two ports are provided for sample introduction, a septum port for liquid injections, and a flip-top port for solid samples. The CO₂ from the oxidation of organic carbon is transported in the carrier-gas stream and is measured by means of a nondispersive infrared analyzer (NDIR).

5.0 COMMENTS

- 5.1 The detection limit for samples is dependent on the amount of sample analyzed.

6.0 SAMPLE STORAGE AND HANDLING

- 6.1 Samples are stored in the walk-in cooler.
- 6.2 Solid samples require no preservation prior to analysis other than storage at 4 °C and have a holding time of 14 days.

7.0 INTERFERENCES

- 7.1 Volatile organics in sediments may be lost in the decarbonation step resulting in a low bias.
- 7.2 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4° C and analyzing within the specified holding times.

8.0 DOCUMENTATION

- 8.1 The following information is documented in logbooks:
- 8.1.1 Document maintenance or replacement of parts to the infrared analyzer in the General laboratory equipment maintenance logbook. Record problems, steps taken to repair instrument and the names of representatives of instrument vendor in General laboratory equipment maintenance logbook.
- 8.1.2 Document the following information during the preparation and analysis of samples in the Inorganics laboratory logbook:
- 8.1.2.1 The method, analysts' initials, and the dates of extraction and analyses.
- 8.1.2.2 The standard codes of solutions used during the course of analysis.
- 8.1.2.3 Record the NEA sample identification numbers, initial sample weights or volumes, post extraction dilution factors, Infrared sample area counts, boat number and any relevant comments.

9.0 SAFETY

- 9.1 Safety glasses, lab coat or lab apron, and disposable gloves must be worn when handling chemicals and samples.

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- 9.2 Personnel should familiarize themselves with the necessary safety precautions by reading MSDS information covering any chemicals used to perform SOP.
- 9.3 Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Specifically, concentrated nitric, sulfuric, and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood and if skin contact occurs, flush with large volumes of water.
- 9.4 Ultra-violet radiation can cause damage to the eyes. Do not open the door to the UV persulfate module without turning the lamp off.

10.0EQUIPMENT

- 10.1 Apparatus and Equipment. Located in the Inorganics laboratory and Main Extraction Laboratory.
 - 10.1.1 Dohrmann IR-I NDIR detector module. Located in the main laboratory.
 - 10.1.2 Dohrmann sludge/sediment sampler. Dohrmann (p/n 832-222). Located in the main laboratory.
 - 10.1.3 250 and 1000 μ L Rainin autopipets. Rainin (p/n EP-250 and EP-1000).
 - 10.1.4 250 and 1000 μ L pipette tips. Rainin (p/n RT-96 and RT-200).
 - 10.1.5 1-5 ml Eppendorf digital pipette with pipette tips. Located in the Inorganics laboratory.
 - 10.1.6 Quartz boats. Dohrmann (p/n 899-624). Located in the main laboratory.
 - 10.1.7 Quartz wool. Dohrmann (p/n 511-735). Located in the main laboratory.
 - 10.1.8 Drying oven.
 - 10.1.9 Propane tank with torch assembly. Located in the main laboratory.
 - 10.1.10 Tweezers and steel spatula. Located in the main laboratory.
 - 10.1.11 50, 100 and 250 μ l syringe. Located in the main laboratory.
 - 10.1.12 High purity oxygen tank with regulator. Located in the main laboratory.
 - 10.1.13 Aluminum weighing boats. Located in the main laboratory.
 - 10.1.14 UV-Persulfate Reaction Module. Located in the main laboratory.
 - 10.1.15 Teflon sleeve reactor, taper joint. Dohrmann (p/n 070-627). Located in the main laboratory.
 - 10.1.16 Lamp, Ultra-violet. Dohrmann (p/n 512-092). Located in the main laboratory.
 - 10.1.17 High purity nitrogen tank with regulator. Located in the main laboratory.
 - 10.1.18 20-mesh tin. Dohrmann (p/n 511-876). Located in the main laboratory and used for tin/copper scrubber.
 - 10.1.19 Copper. Dohrmann (p/n 511-895). Located in the main laboratory and used for tin/copper scrubber.

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- 10.1.20 Pyrex wool. Dohrmann (p/n 511-895). Located in the main laboratory and used for tin/copper scrubber.
- 10.1.21 Class A volumetric flasks at volumes of 10, 25, 50, 100, and 200 ml.
- 10.1.22 Analytical balance. The vendor is Mettler and the model number is AG204. Capable of weighing to 0.0001 grams.
- 10.1.23 Graduated cylinders.
- 10.1.24 Disposable 1, 5, and 10 ml pipettes.
- 10.1.25 TOC logbook.
- 10.1.26 Rinse bottle. Filled with laboratory grade water. Located in the Inorganics laboratory.
- 10.1.27 Inorganics department standard preparation logbook.
- 10.1.28 Gray septum. Dohrmann (p/n 517-807). Located in the main laboratory.

10.2 Reagents

10.2.1 Preparation of TOC working standard solution.

10.2.1.1 ERA p/n 516 or other suitable vendor.

10.2.1.2 Determine total ug of TOC in stock standard = {[standard conc. (mg/L)]*[dilution volume (ml)]}

10.2.1.2.1 For example, the instructions supplied with the solution stipulate that 5 ml of the vial be diluted to 1000 ml. The standard concentration stated in the 'Certificate of Analysis' will be 72.6 mg/L. Thus, the total amount of TOC in the vial is 14,520 ug.(200X)

10.2.2 Preparation of TOC (solids) calibration standards:

10.2.2.1 Prepare 5 calibration standards ranging in concentration from approximately 70 mg/L to 12,000 mg/L.

10.2.2.2 Use the following formula to assist in preparing the calibration standards.
 Cal. Std. (mg/L) =
$$\frac{\{[\text{Conc. Of working std. (ug/ml)}] * \{\text{vol. of stock std. (ml)}\}}{\{\text{Final vol. (ml)}\}}$$

10.2.2.3 Using information from the previous examples to prepare a calibration standard.

$$\frac{\{[14,520 \text{ (ug/ml)}] * [0.05 \text{ (ml)}]\}}{(10 \text{ ml})} = 72.6 \text{ mg/L calibration standard}$$

11.0 METHOD PERFORMANCE

11.1 Statistics for method performance are maintained by the Quality Assurance unit and are available for review.

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12.0 PROCEDURE

- 12.1 Operation and maintenance of the Dohrmann IR-I NDIR detector module.
 - 12.1.1 Refer to the instrument manual for specific instructions and part numbers for all components.
 - 12.1.2 To prepare the tin/copper scrubber;
 - 12.1.2.1 fit one end of the Pyrex scrubber tube with a cored gray septum.
 - 12.1.2.2 Insert a tuft of Pyrex wool and then about 2 inches of tin in the other end. Secure the tin with another tuft of Pyrex wool.
 - 12.1.2.3 Fill the remaining half of the scrubber tube with an equal amount of copper. Secure the copper with a third tuft of Pyrex wool. Insert a cored gray septum.
 - 12.1.2.4 Each day of use, inspect the tin/copper scrubber and change the contents of the tube when one-half of the tin is discolored.
 - 12.1.3 The detector must be on for several hours in order to achieve equilibrium. It is recommended that the detector be turned on the day before the analysis is to be performed. Power up the detector and the main unit.
 - 12.1.4 Verify that the printer has sufficient amount of paper before starting the analysis. Reset the printer so that the number "1" will be printed for the first analysis performed for that day.
 - 12.1.5 Select the "TOC" and the "DET" positions. For the detector, select position "3" for high concentrations, "2" for medium concentrations, and "1" for low concentrations of TOC.
 - 12.1.6 The module will not light the green "ready" light if the baseline is above 0.05. Adjust the "zero" control until the baseline is less than 0.02. The "CALIB" light must be off during analysis.
- 12.2 Operation and maintenance of the Dohrmann sludge/sediment sampler.
 - 12.2.1 Refer to the instrument manual for specific instructions and part numbers for all components.
 - 12.2.2 A portion of sample is weighed into a quartz boat where it is acidified and dried. The boat is placed in the boat carriage of the sampler and it is moved into the combustion chamber. Gas from the combustion tube flows into the flask to the right where it passes through acidified water.
 - 12.2.3 The gas travels to the flask to the left where excess water is removed before traveling to the detector module. The gas passes through the tin and copper scrubber and into the detector.
 - 12.2.4 Before turning on the solid sampler, carefully examine individual components for sign of wear.
 - 12.2.4.1 Adjust the flow of oxygen to 30 psi.
 - 12.2.4.2 The level of acidified water in the right flask must be above the fritted sparging finger. A vigorous flow of gas emitting from the sparging finger should be easily observed, if not, check the gas lines and connections for leaks.
 - 12.2.4.3 The water collection flask should be emptied on a daily basis.
 - 12.2.5 Turn on the furnace unit.

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- 12.2.6 When using the module for the first time or after a long period of inactivity, the furnace should be monitored with a voltmeter to verify that the temperature is at 800°C.
 - 12.2.6.1 Place the black (ground) probe in the "com" port.
 - 12.2.6.2 Place the red (positive) probe in the "monitor", set the voltmeter to "volts".
 - 12.2.6.3 The voltage reading should read "0.80", if not, place the red probe in the "adj" port.
 - 12.2.6.4 The voltage reading should read "0.80", if not, adjust the voltage by turning the setscrew until the correct voltage is achieved.
- 12.2.7 If the gray septum (p/n 517-807) at either end of the combustion tube have corroded and require replacement, the furnace must be turned off before replacing the septum.
- 12.3 Calibration of Dohrmann sludge/sediment sampler and IR-I NDIR detector module.
 - 12.3.1 A new calibration curve must be generated:
 - 12.3.1.1 Every four months;
 - 12.3.1.2 if either the ICV or CCV are outside acceptance criteria or
 - 12.3.1.3 If the detector has been rebuilt.
 - 12.3.2 The calibration curve is based on 'µg of carbon' versus 'area'. The calibration standards require duplicate injections. The average blank area is subtracted from the average area for each calibration standard.
 - 12.3.3 Press 'Reset' on printer to reset the number log for the printer.
 - 12.3.4 A fresh tuft of quartz wool is inserted into a quartz boat. The boat is placed inside the sediment sampler module. Hook the loop of the boat with the end of the magnetic boat carriage.
 - 12.3.5 Remove contaminates from the boat by placing it in the furnace until the baseline has started to decrease. Pull the boat out of the furnace.
 - 12.3.6 After the boat has cooled (approximately 30 seconds), place the boat underneath the injection port. Remove septum and inject 0.070 ml of the blank or calibration standard onto the boat. Replace septum.
 - 12.3.7 After the baseline has stabilized, place the boat in the furnace. Press the "Start" button. After the signal has started to decrease, pull the boat out of the furnace.
 - 12.3.8 Repeat injection of the standard until consecutive measurements are obtained that are reproducible to within $\pm 10\%$.
 - 12.3.9 Repeat for the remaining calibration standards.
 - 12.3.10 Every standard must be within the scale of the detector.
 - 12.3.11 If the needle in the IR meter goes past '95' or if the red error light has lit after injecting the stock standard or a sample, inject a smaller volume of the standard or prepare a smaller amount of sample and reanalyze.
 - 12.3.12 Enter the injection number, standard label, date analyzed, injection volume, and the area printed by the printer in the TOC logbook.

- 12.3.13 After the calibration curve has been completed, enter the area and the ug of carbon for each standard in the appropriate fields in the TOC solids Excel spreadsheet. The slope for the calibration curve, the average area for the blank and the average area for the lowest calibration standard are entered into a formula in LIMS that converts the average area for each sample into the average ug of carbon.
- 12.4 Preparation of solid samples.
- 12.4.1 Between 1.0 and 50 mg of material can be placed in a boat depending on the percent of carbon in the sample. Solid samples are analyzed once..(If Client requires triplicate analysis see NE177.SOP).
- 12.4.2 The concentration of the samples must be within the range of the calibration curve. If the sample concentration of the sample is outside the range of the calibration curve, repeat the analysis of the sample.
- 12.4.3 If the sample concentration is too high, repeat the sample preparation and analysis with a lower sample weight (minimum 1.0 mg).
- 12.4.4 Place each quartz boat in a numbered aluminum weigh boat.
- 12.4.5 Homogenize a portion of the sample.
- 12.4.6 Place one aluminum boat with a quartz boat on the analytical balance and tare the balance.
- 12.4.7 Transfer an aliquot of the sample to the quartz boat and record the NEA #, weight and the boat number in the TOC logbook.
- 12.4.8 Add 2 to 3 drops of 1+1 nitric acid to each sample.
- 12.4.9 Place the aluminum weigh boats in the oven. Remove the sample when dried (minimum of 10 minutes).
- 12.4.10 Place the boat in the raceway. After the baseline has stabilized, place the boat in the furnace and press the 'Start' button.
- 12.4.11 Copy the TOC area from the printer into the TOC logbook.
- 12.4.12 After each sample analysis, rinse the boat with LGR water, scrape any remaining material from the boat and place the boat in the flame of the propane torch to remove any contaminants.
- 12.5 Percent total solids determination.
- 12.5.1 Determine the percent total solids for each sample as described in NE090.SOP
- 12.6 Sample calculations using Microsoft Excel 4.0.
- 12.6.1 The calibration curve and samples are calculated by using an Excel spreadsheet. The spreadsheet requires the entry of the absorbencies of the standards and samples, sample extract volumes, sample weights or volumes, percent solids and dilution factors.
- 12.6.2 Sample values are computed by comparing response with the standard curve.
- 12.7 Archiving of data.
- 12.7.1 Print a copy of spreadsheet and place in the data folder for that client.
- 12.8 Transfer of Data to LIMS (TOCS)

- 12.8.1 After the calibration curve has been completed, give the LIMs manager a copy of the Exel spreadsheet for the calibration curve including values for the slope and instrument intensities for blank and the lowest calibration standard.
- 12.8.2 Log into LIMS. Click “Win Results” or “Results” from LIMS toolbar. Select the appropriate samples by either typing in the sample ID’s or selecting the Login Record File.
- 12.8.3 Choose the result entry template “TOCSOL”, then click “OK”. A result entry spreadsheet will then be created with the following columns: TOCSO, TOCI_A, TOCI_1, TOC_1SW, %SOLIDS.
- 12.8.4 The data for samples should be entered into the columns as follows:
 - 12.8.4.1 TOCI_1 = Area Counts for Sample
 - 12.8.4.2 TOC_1SW = Sample weight in grams.
 - 12.8.4.3 %SOLIDS = % Total Solids for Sample (Enter as a percentage, not a decimal).
 - 12.8.4.4 TOCI_A = Area Counts for Sample (Fills in automatically).
 - 12.8.4.5 TOCSO = Result for TOC in Solids (Fills in automatically).
- 12.8.5 Once the field TOCSO has been filled in by the computer, right click on that field and select “detailed edit” from the pull down menu. Confirm that the MDL and the date analyzed for the sample are correct. Proceed to the next sample.
- 12.8.6 Each method has tests for the required QC parameters.
- 12.8.7 Add QC parameters to the assigned samples by QC batching.
 - 12.8.7.1 QC samples are added as individual tests to predetermined samples in LIMs. Access LIMs and go to ‘QC Batching’.
 - 12.8.7.2 Enter the required information into the database.
 - 12.8.7.3 Once the data has been entered for all samples, go the QC section of the spreadsheet. If batching was performed correctly there should be some of these fields displayed in white. If not, right click on the dark gray fields in that same row so that all appropriate QC tests have been added.
 - 12.8.7.3.1 For example, the data fields for the sample duplicate must be selected (white) before entering data into the spreadsheet fields.

13.0 Calculation of data.

- 13.1 Data solids are reported in "mg/kg" units.
- 13.2 Matrix spike recovery:

$$\%REC = \frac{[{\text{M.S. sample}}] - {\text{sample}}}{\text{Spike Added}} * 100$$
- 13.3 Relative Percent Difference:

$$\%RPD = \frac{\text{Abs. } [{\text{Result1}}] - {\text{Result2}}}{\text{Result1} + {\text{Result2}}} * 200$$

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13.4 %Recovery for IPC and QCS solutions:
%REC.= $\frac{\{\text{Found value}\}}{\{\text{True value}\}} * 100$

13.5%Recovery for LCSS samples:
%REC.= $\frac{\{\text{found value}\}}{\{\text{True Value}\}} * 100$

14.0 Data submission

- 14.1 The information that is required on the client's certificate is the NEA identification number, client identification, method code, sample results, detection limits, concentration units, and analysis dates.
- 14.2 The units used for reporting solid samples are mg/kg. Results are rounded to three significant figures.
- 14.3 For soil samples, if the sample analyses is off scale and the minimum sample weight of 1.0 mg was used, calculate the maximum concentration of TOC based on the μg of carbon of the highest calibration standard, average sample weight, and the percent total solids. Report the results as greater than the calculated maximum sample concentration.

15.0 Equipment maintenance.

- 15.1 Record the replacement of parts in the General Laboratory maintenance book.
- 15.2 Replace the drying tube if water droplets are observed inside the tube.
- 15.3 Replace the material in the scrubber tube when discolored.
- 15.4 Replace septa and tubing when worn.
- 15.5 If the baseline consistently rises with each day of use the IR detector must be repaired by Horiba.
 - 15.5.1 Remove the detector from the housing and wrap in bubble wrap. Place in a secure box with packing peanuts.
 - 15.5.2 Type the PO# required for repair, the company name, company contact, type of service required, analyst name and phone number, and if necessary a return authorization number on company stationary and place in an envelope. Place the envelope in the box.
 - 15.5.3 Two weeks are required for shipping and repair of the detector.
- 15.6 If the temperature for boat module oven fluctuates, either the oven or the thermocouple must be replaced.

16.0 QUALITY CONTROL

- 16.1 Initial Demonstration of Performance
 - 16.1.1 Perform Method Detection Limit studies for solid matrices.
 - 16.1.1.1 MDLs should be determined annually or when in the judgment of the analyst, a change in instrument hardware or operating conditions would dictate they need to be redetermined.
- 16.2 Certification for analysts.

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- 16.2.1 Analysts must read, understand, and use the latest version of the laboratory's SOPs that relate to their job responsibilities. The SOPs must be read during their initial training, annually, and when the SOPs are revised.
- 16.2.2 When possible, analysts should attend training courses related to their job responsibilities.
- 16.2.3 For each matrix processed with this method, the analyst must be capable to perform the following tasks:
 - 16.2.3.1 Prepare and analyze four consecutive laboratory control samples within method quality control limits or within the limits supplied with the purchased material.
- 16.2.4 For each matrix processed with this method, the analyst must be capable of analyzing a blind sample successfully.
 - 16.2.4.1 A blind sample must be analyzed during their initial training and annually thereafter.
- 16.3 Sample preparation quality control.
 - 16.3.1 Laboratory reagent Blank/Preparation blank : Prepare and analyze at least one LRB with batch of 20 or fewer samples of the same matrix or batch.
- 16.4 Instrument and analysis quality control.
 - 16.4.1 ICV/CCV:
 - 16.4.1.1 Analyze ICV immediately after calibrating the instrument.
 - 16.4.1.2 The source of the standards must be different than the source for the calibration standards.
 - 16.4.1.3 Prepare at a concentration approximately equal to the midpoint of the calibration curve.
 - 16.4.1.4 Analyze CCV every ten samples and at the beginning and the end of the analytical run.
 - 16.4.1.5 Continuing Precision and Accuracy. To initiate a control chart, a representative sample of a well mixed sediment is analyzed 15 times to determine the analytical precision. A control chart displaying a control limit of three times the measured standard deviation for the 15 replicates is created.

For every sample batch (20 or less) take one sample and run in quadruplicate. Calculate the standard deviation and compare with the control chart limit.

If the sample being run in quadruplicate exceeds the three standard deviation limit, identify the error and rerun the samples in that batch along with the quadruplicate samples.

17.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QC MEASURES

- 17.1 Initial review of documentation
 - 17.1.1 After the completion of the analysis run, the analyst reviews the logbook(s) and analytical data for completion and completes the required documentation.
 - 17.1.2 The supervisor reviews the logbook(s) and data and records their initials and review dates on the appropriate documents.

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- 17.1.3 Each QC measurement is reviewed and compared to the appropriate acceptance criteria.
- 17.1.4 The following section supplies the corrective action and or contingencies if the criteria for the QC measure are unacceptable.
- 17.2 Calibration curves:
 - 17.2.1 Acceptance criteria: Correlation coefficient must be ≥ 0.997 .
- 17.3 Linear dynamic range for samples:
 - 17.3.1 Acceptance criteria: Determined sample concentrations that are greater than the highest calibration standard must be diluted and reanalyzed. Report the results from the diluted sample.
- 17.4 Laboratory reagent Blank/Preparation blank:
 - 17.4.1 Acceptance criteria: The absolute value of the concentration must not exceed the PQL/PRDL of the analyte.
 - 17.4.1.1 Not applicable if the sample concentration is $> 10X$ blank level,
 - 17.4.1.2 Or if positive result is reported for the blank but the analyte is not in the sample.
- 17.5 Laboratory fortified matrix/matrix spike accuracy.
 - 17.5.1 Acceptance criteria: The spike recovery limits are 75 to 125%.
 - 17.5.2 Not applicable if sample concentration is greater than 1/5 spike added.
- 17.6 Laboratory Duplicates/matrix spike and matrix spike duplicates precision
 - 17.6.1 Acceptance criteria: A %RPD limit of $\pm 20\%$ is used for analytes $> 5X$ PQL or $\pm PQL$ limit is used if the analyte concentration in the sample or duplicate is $< 5X$ PQL.
- 17.7 ICV/CCV and Initial Precision and Accuracy:
 - 17.7.1 Acceptance criteria: A % recovery limit of 90-110% is used.

18.0 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

- 18.1 Calibration curves:
 - 18.1.1 Corrective action: If QC measure not within acceptance criteria, determine source of problem, correct problem and recalibrate instrument.
- 18.2 Laboratory reagent Blank/Preparation blank:
 - 18.2.1 Corrective action: If QC measure not within acceptance criteria;
 - 18.2.1.1 Reanalyze preparation blank and;
 - 18.2.1.2 If still out, reanalyze all samples associated with the LRB.
- 18.3 Laboratory fortified blank/Laboratory control sample:
 - 18.3.1 Corrective action: If QC measure not within acceptance criteria;

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18.3.1.1 If LCS is high and sample results <PQL, no corrective action, ELSE,

18.3.1.2 Reanalyze LCS/LFB and;

18.3.1.3 If still out, re-extract and reanalyze all samples associated with the LCS.

18.4 Laboratory fortified matrix/matrix spike

18.4.1 Corrective action: Determine problem, re-analyze samples based upon analyst judgement.

18.5 Laboratory Duplicates

18.5.1 Corrective action: Determine problem, re-analyze samples based upon analyst judgement.

18.6 QCS/ICV/IPC/CCV:

18.6.1 Corrective action: If QC measures not within acceptance criteria stop the analysis, determine source of problem, correct problem and verify calibration and reanalyze all samples since last compliant QC measurement.

19.0 Contingencies for handling out of control or unacceptable data.

19.1 If the acceptance criteria for QC measures has been exceeded for requested analytes and the data is to be reported, the following procedures must be implemented:

19.1.1 The Quality assurance officer must be notified.

19.1.2 The data must be flagged with the appropriate qualifiers and case narrative.

19.1.3 The client must be notified about the data.

20.0 DEFINITIONS

20.1 Analytical Batch – The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples that are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g. groundwater, sludge, ash, etc.)

20.2 Calibration – The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid and reagents or concentration of acids as used in the sample preparation.

20.3 Calibration Blank-A volume of reagent water acidified with the acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the instrument.

20.4 Calibration Standard (CAL) - A solution prepared from the dilution of stock standards solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

20.5 Calibration curve - If the correlation coefficient is < 0.997 or if the calculated recoveries for any of the calibration standards are not within 10% of the true value (except for the lowest standard), repeat analysis of the outlying standards until curve is within acceptance criteria.

20.6 Correlation Coefficient – The correlation coefficient for the calibration curve must be greater than or equal to 0.997 according to NYSDOH requirements.

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- 20.7 Continuing Check blank (CCB) - Analyze the CCB solution after each ICV/CCV solution. If the absolute value of the CCB is \geq the PQL, stop the analysis, correct problem, recalibrate the instrument and reanalyze all samples since the last compliant CCB.
- 20.8 Instrument Performance Check (IPC) Solution – Also known as CCV. A solution of method analytes, used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
- 20.9 Laboratory Duplicates (LD1 and LD2) – Two aliquots of the same sample taken in the laboratory and analyzed with identical procedures. An analysis of LD1 and LD2 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
- 20.10 Laboratory Fortified Blank (LFB) – Also known as a LCSW or LCSS. An aliquot of LRB to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.
- 20.11 Laboratory Fortified Sample Matrix (LFM) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.
- 20.12 Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.
- 20.13 Linear Dynamic Range (LDR) – The concentration range where the instrument response to an analyte is linear.
- 20.14 Matrix – The predominant material of which the sample to be analyzed is composed.
- 20.15 Matrix Spike – An aliquot of the sample is spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given matrix.
- 20.16 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.
- 20.17 MSDS – Material safety data sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs. The thrust of the law is to have those who make, distribute, and use hazardous materials responsible for effective communication.
- 20.18 PQL – The Practical quantitation limit (PQL) is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.
- 20.19 Quality Control Sample (QCS) – Also known as ICV: A solution of method analytes of known concentrations, which is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check either laboratory or instrument performance.
- 20.20 Relative Percent Difference (RPD) – To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

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- 20.21 Replicate – Repeated operation occurring within an analytical procedure. Two or more analyses for the same constituent in an extract of a single sample constitute replicate extract analyses.
- 20.22 RCRA – Resource Conservation and Recovery Act, PL 94-580. Found at 40 CFR 240-271. EPA has jurisdiction. Enacted November 21, 1976, and amended since. RCRA’s major emphasis is the control of hazardous waste disposal. It controls all soil-waste disposal and encourages recycling and alternative energy sources.
- 20.23 Reagent Water – Water in which an interferent is not observed at or above the minimum quantitation limit of the parameters of interest.
- 20.24 Rounding Rules – If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by:
- 20.24.1 If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number.
- 20.24.2 If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.
- 20.25 Sample Delivery Group (SDG) – Unit within a single case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day data turnaround contracts). Data from all samples in an SDG are due concurrently.
- 20.26 Stock Standard solution - A concentrated solution containing one or more method analytes prepared in the laboratory or purchased from a reputable commercial source.
- 20.27 Water Sample – For the purpose of this method, a sample taken from one of the following sources: drinking, surface, ground, storm runoff, industrial or domestic wastewater.

21.0 POLLUTION PREVENTION/WASTE MANAGEMENT

- 21.1 Refer to NEA168.SOP for instructions for pollution prevention.
- 21.2 Refer to NEA089.SOP and NEA054.SOP for instructions for the disposal of waste generated during the procedures previously mentioned.

22.0 DETECTION LIMIT

- 22.1 The most recent MDL studies and PQLs are maintained by the Quality Assurance unit and are available for review.

23.0 REFERENCES

- 23.1 "Determination of Total Organic carbon in sediment," Lloyd Kahn, U.S.E.P.A. Region II, Edison NJ 1988.
- 23.2 Application Note: TOC-011 "Analysis of sludges and solids for carbon," Tekmar-Dohrmann, Cincinnati, OH 10/95.
- 23.3 NYS-OH ELAP manual item #271.

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