

# CASTNET

## Appendix 4: CASTNET Laboratory Standard Operating Procedures

---

**Clean Air Status and Trends Network**

**Quality Assurance Project Plan**

Revision 8.3

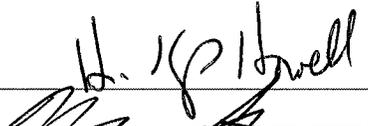
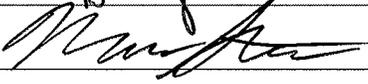
**Appendix 4:**

**CASTNET Laboratory Standard Operating Procedures**

October 2015

---

| <b>Laboratory Instrumentation</b> |  |
|-----------------------------------|--|
| GLM3180-001                       | Determination of Inorganic Anions in Water and Air Filters by Ion Chromatography for Acid Deposition Projects (EPA Method 300.0) |
| GLM3180-004                       | Ammonia for Dry and Wet Deposition (Modified EPA Method 350.1)   |
| GLM3180-005                       | Inductively Coupled Plasma-Atomic Emission Spectrometry: Perkin Elmer Optima 3000 DV (Modified EPA Method 6010B)                 |
| GLM3180-007                       | Hydrogen Ion (pH) in Water (EPA Method 150.1)  |
| GLM3180-008                       | Specific Conductance for Wet Deposition Samples (EPA Method 120.1)   |
| <b>Laboratory - Management</b>    |  |
| GLO3180-001                       | Sample Container Preparation for Dry and Wet Deposition  |
| GLO3180-002                       | Washing of Filter Pack Equipment, Denuders, and Filter Tweezers  |
| GLO3180-003                       | Central Sample Storage Organization for Dry and Wet Deposition Samples   |
| GLO3180-010                       | Whatman Paper Filter Preparation with Potassium Carbonate  |
| GLO3180-011                       | Coating of Ambient Air Denuder Tubes   |
| GLO3180-012                       | Clean Air Status and Trends Network (CASTNET) Three-Stage Ambient Air Filter Pack Receipt, Disassembly, and Login                |
| GLO3180-013                       | Preparing and Shipping the CASTNET Three-Stage Filter Packs  |
| GLO3180-015                       | Procedure for Sample Information Changes and Additions   |
| GLO3180-016                       | Procedure for Data Entry Error Correction  |
| GLO3180-022                       | Deionized Water Quality  |
| GLO3180-035                       | Element Batch Preparation  |
| GLO3180-036                       | Procedure for Generation of Control Charts   |
| GLO3180-037                       | Procedure for Labeling and Expiration of Standards   |
| GLO3180-038                       | Procedure for Calibration of Pipettors   |

| <b>Authorizations</b> |                   |  |             |
|-----------------------|-------------------|--|-------------|
| <b>Title</b>          | <b>Name</b>       | <b>Signature</b>   | <b>Date</b> |
| Project Manager       | H. Kemp Howell    |  | 10/16/15    |
| QA Supervisor         | Marcus O. Stewart |  | 10/16/15    |

## Revision / Review History

| Revision Number  | Change Description   | Reviewed by | Date  |
|--|--|-------------|-------|
| GLM3180-004.R6   | Updated to reflect new instrumentation (TRAACS to AA3) and reagent formula   | M. Stewart  | 2/05  |
| GLM3180-005.R5   | Updated to reflect CASTNET 3 requirement of SP +/- 5 percent.  | M. Stewart  | 2/05  |
| GLM3180-008  | Nomenclature change: mhos to Siemens; and clarification to use of automatic temperature compensation                           | M. Stewart  | 10/09 |
|  | All other laboratory SOPs save those below have been updated to indicate the current laboratory manager.                       | M. Stewart  | 2/05  |
| GLM3180-001.R6<br>GLM3180-004.R7<br>GLM3180-005.R6<br>GLO3180-010.R3<br>GLO3180-012.R4<br>GLO3180-013.R5<br>GLO3180-015.R7<br>GLO3180-022.R5                   | Updated to reflect the use of a new Laboratory Information Management System (LIMS)  | M. Stewart  | 10/06 |
| GLM3180-001.R7<br>GLM3180-004.R9<br>GLM3180-005.R7<br>GLM3180-007.R8<br>GLM3180-008.R7   | Updated to reflect the use of new laboratory instrumentation and reformatted to include more figures.                          | M. Stewart  | 10/10 |
| GLO3180-002.R5<br>GLO3180-003.R7<br>GLO3180-010.R4<br>GLO3180-011.R3<br>GLO3180-012.R5<br>GLO3180-013.R6<br>GLO3180-015.R7<br>GLO3180-016.R5<br>GLO3180-022.R5 | Reformatted to include more figures.   | M. Stewart  | 10/10 |
| <b>The following SOP have been archived</b>  |  |             |       |
| GLM3180-010.R3   | Acidity for Wet Deposition Samples (EPA Method 305.2)  | M. Stewart  | 2/05  |
| GLO3180-005.R2   | Wet Deposition Bucket Blank Sample   | M. Stewart  | 2/05  |
| GLO3180-006.R2   | Wet Deposition Field Bottle Blank Sample Preparation   | M. Stewart  | 2/05  |
| GLO3180-008.R2   | Receiving, Unpacking, and Login of One-Stage Filter Packs and Denuders   | M. Stewart  | 2/05  |
| GLO3180-009.R2   | Preparing and Shipping the One-Stage Filter Packs and Denuders   | M. Stewart  | 2/05  |
| GLO3180-020.R2   | Procedure for Generating the Atmospheric Concentration Reports   | M. Stewart  | 2/05  |
| GLO3180-021.R3   | Data Submittal for Wet and Dry Deposition Samples  | M. Stewart  | 2/05  |
| GLO3180-007.R3   | Procedure for Receiving, Assigning Sample Numbers and Shipping CASTNET Wet Deposition Sampling Kits                            | M. Stewart  | 10/06 |
| GLO3180-014.R3   | Finalization/Definalization of Data Batches  | M. Stewart  | 10/06 |
| GLO3180-017.R5   | Computer Sample Login and Sample Arrival Notification  | M. Stewart  | 10/06 |
| GLO3180-018.R6   | Available Numbers Distribution   | M. Stewart  | 10/06 |
| GLO3180-023.R3   | Setting Up Chemical Laboratory Analysis and Scheduling System (CLASS™) Field Groups and Preparing Laboratory Sample Kit Labels | M. Stewart  | 10/06 |
| GLO3180-004.R5   | Filtering Wet Deposition Samples   | M. Stewart  | 10/11 |
| GLO3180-019.R5   | Laboratory Information Management System Enterprise Backup System  | M. Stewart  | 10/11 |

**Notes:** GLM = Gainesville Laboratory Method.

GLO = Gainesville Laboratory Operations. Rx = Revision Number.

**TITLE: DETERMINATION OF INORGANIC ANIONS IN WATER  
AND AIR FILTERS BY ION CHROMATOGRAPHY FOR  
ACID DEPOSITION PROJECTS  
(EPA METHOD 300.0, MODIFIED)**

Effective

Date:

12/2/15

Prepared by: Katherine W. Barry  
Laboratory Operations  
Manager



Reviewed by: Marcus O. Stewart  
QA Manager



**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |        |       |            |
|---------------|--------|-------|------------|
| Reviewed by:  | Title: | Date: | Signature: |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |

# **DETERMINATION OF INORGANIC ANIONS IN WATER AND AIR FILTERS BY ION CHROMATOGRAPHY FOR ACID DEPOSITION PROJECTS (EPA METHOD 300.0, MODIFIED)**

## **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the determination of inorganic anions in air filters, water, and annular denuders by ion chromatography (IC). The anions of interest include particulate chloride (Cl<sup>-</sup>), particulate nitrite (NO<sub>2</sub><sup>-</sup>), particulate nitrate (NO<sub>3</sub><sup>-</sup>), nitric acid (HNO<sub>3</sub>); and gaseous sulfur dioxide (SO<sub>2</sub>) as particulate sulfate (SO<sub>4</sub><sup>2-</sup>), depending on filter type; and particulate sulfate (SO<sub>4</sub><sup>2-</sup>).

## **2.0 SCOPE**

This method is used for acid deposition projects and covers the determination of selected inorganic ions by IC in rainwater, cloud water, lake water, snow, and in various filter media as dry deposition.

## **3.0 SUMMARY OF METHOD**

An aliquot of a water sample or an aliquot of an extract (filter or denuder) is passed through a series of ion exchangers. The anions of interest are separated on the basis of their relative affinities for a strongly basic anion exchanger (analytical column) and then converted to their highly conductive acid form by membrane suppression. Detection is accomplished by electrical conductivity. The resulting chromatographic peaks are identified on the basis of retention time compared to known standards. Quantitation is performed by the comparison of the peak area to the calibration curve areas.

### **3.1 Method Interferences**

Interferences can be caused by substances with retention times that are similar to and overlap with those of the anion(s) of interest. Column overloading can lead to peak tailing, poor peak resolution, and/or carryover into an adjacent downfield peak.

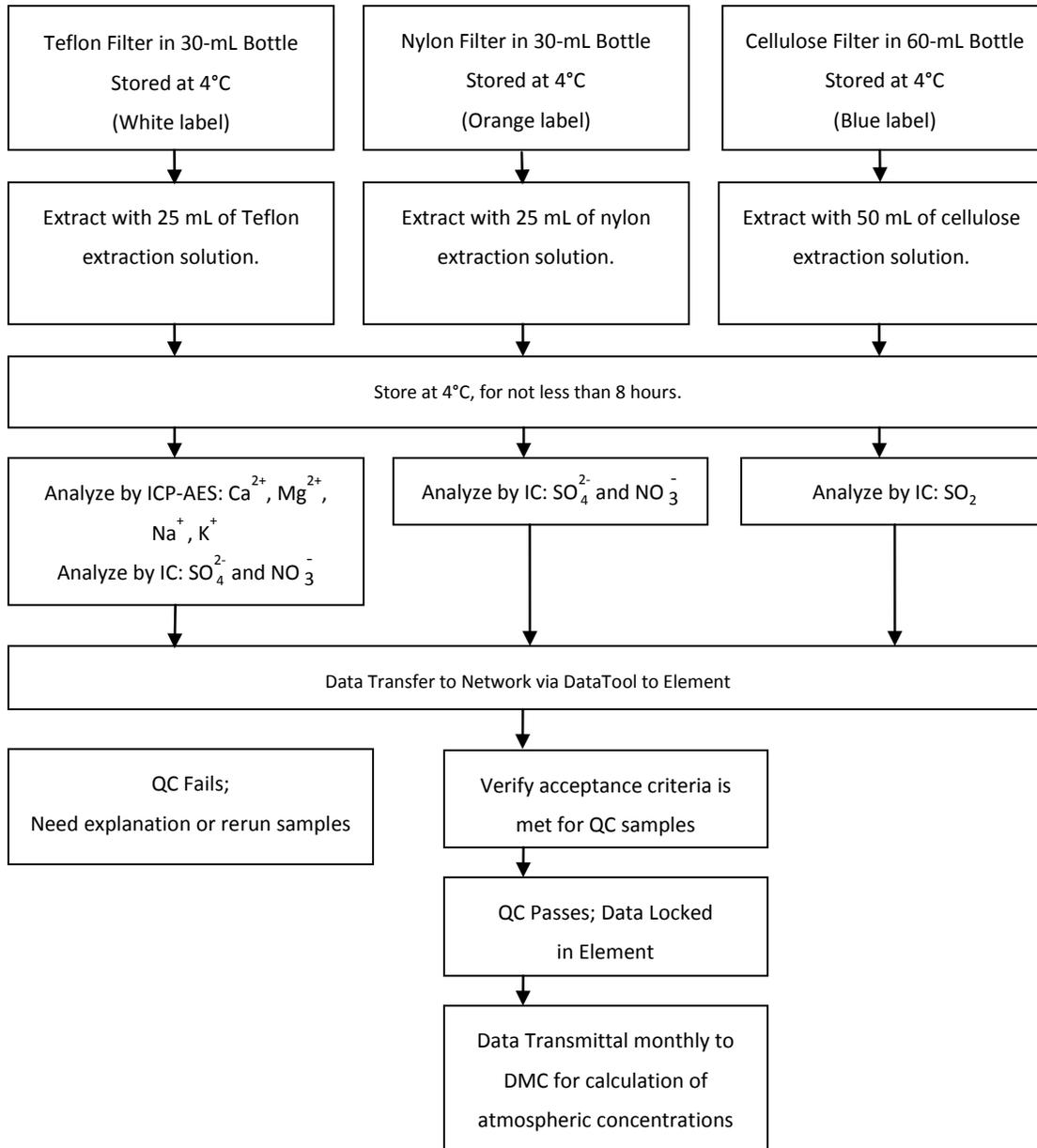
Method interferences may be caused by contaminants in the reagent water, reagents, glassware, or other sample processing accessories that lead to discrete artifacts or an elevated baseline in ion chromatograms.

Samples that contain particles larger than 0.45 micrometers (µm) and reagent solutions that contain particles larger than 0.20 µm require filtration to prevent damage to instrument columns and flow systems.

### 3.2 Deviations from the Method

A general overview of the steps described in this SOP is depicted in Figure 1. Deviations from the analytical method described in this SOP are not permitted.

**Figure 1** Overview – Laboratory Operations Procedures for Filter Packs

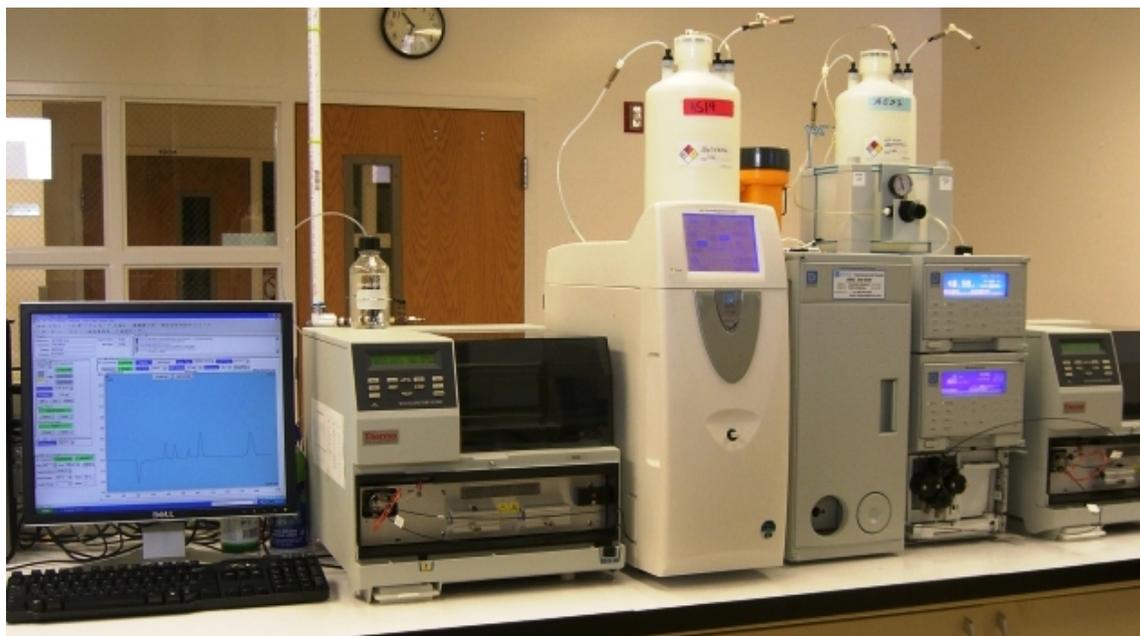


## 4.0 MATERIALS

### 4.1 Apparatus

- Analytical balance capable of accurately weighing to the nearest 0.01 gram (g)
- Ion chromatograph: Dionex ICS-1600 or equivalent (see Figure 2)
- Anion guard column: Dionex AG14, AG22, AG22-Fast or equivalent
- Anion analytical column: Dionex, AS14, AS22, AS22-Fast or equivalent
- Anion micro-membrane suppressor: AMMS 300, or equivalent
- Anion self-regenerating suppressor: ASRS-300, or equivalent
- Conductivity cell detector: Approximately 1.25 microliter ( $\mu\text{L}$ )-internal volume or equivalent
- Appropriate autosampler vials and caps
- Eppendorf variable or fixed volume pipettors or equivalent with appropriate disposable tips
- Disposable plastic beakers
- Shaker tables
- Ultrasonic baths

**Figure 2** Dionex ICS-1600 and 500X Ion Chromatographs



## 4.2 Reagents

### 4.2.1 Water and Single Compounds

- Reagent water: deionized (DI) water of resistivity of 15 mega ohms (MΩ) or greater derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 μm.
- Sodium hydroxide (NaOH) 2 normal (N) solution, American Chemical Society (ACS) reagent grade or better. Chemical Abstracts Service Registry Number (CAS#) 1310-73-2
- Sodium bicarbonate (NaHCO<sub>3</sub>), ACS reagent grade or better. CAS# 144-55-8
- Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), ACS reagent grade or better. CAS# 497-19-8
- Concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), ACS reagent grade or better. CAS# 7664-93-9
- Concentrated hydrochloric acid (HCl), ACS reagent grade or better. CAS# 7647-01-0
- Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), 30 percent volume-to-volume (v/v), ACS reagent grade or better. CAS# 7722-84-1

### 4.2.2 Mixtures

- Eluent (mobile phase) solutions: A solution of a suitable Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> buffer prepared as determined by the choice of analytical column. The solutions used for the AS22, AS22-Fast and AS14 columns follow.
  - AS22 and AS22-Fast concentrated eluent solution: 600 millimolar (mM) NaHCO<sub>3</sub> /400 mM Na<sub>2</sub>CO<sub>3</sub>: Dissolve 100.59 g NaHCO<sub>3</sub> and 84.28 g Na<sub>2</sub>CO<sub>3</sub> in reagent water and dilute to 2 liters (L).
  - AS14 concentrated eluent solution #1: 60 mM NaHCO<sub>3</sub>/480 mM Na<sub>2</sub>CO<sub>3</sub>: Dissolve 10.08g NaHCO<sub>3</sub> and 101.74g Na<sub>2</sub>CO<sub>3</sub> in reagent water and dilute to 2 L
  - AS 14 concentrated eluent solution #2: 100 mM NaHCO<sub>3</sub>/350 mM Na<sub>2</sub>CO<sub>3</sub>: Dissolve 16.78g NaHCO<sub>3</sub> and 73.96g Na<sub>2</sub>CO<sub>3</sub> in reagent water and dilute to 2 L
- Working eluent solutions: Dilute 40 milliliters (mL) of the appropriate concentrated eluent solution to 4 L with reagent water. The final concentrations of the listed working eluent solutions are as follows:
  - AS22 and AS22-Fast– 4.0/ 6.0 mM CO<sub>3</sub><sup>2-</sup>/HCO<sub>3</sub><sup>-</sup>
  - AS14 #1– 4.8/0.6 mM CO<sub>3</sub><sup>2-</sup>/HCO<sub>3</sub><sup>-</sup>
  - AS14 #2- 3.5/1.0 mM CO<sub>3</sub><sup>2-</sup>/HCO<sub>3</sub><sup>-</sup>
- Regeneration solution (for the membrane suppressor): 0.025 N H<sub>2</sub>SO<sub>4</sub>. Dilute 2.8 mL concentrated H<sub>2</sub>SO<sub>4</sub> to 4 L with DI water. Self-regenerating suppressors may be used with this solution in the chemical regeneration mode. External water and recycling modes of operation are also available. Select the suppression mode most suited to the sample matrix. See the instrument manuals for further information.
- Calibration stock solutions are purchased as National institute of Standards and Technology (NIST)-traceable solutions. A Certificate of Traceability (Figure 3) and

an expiration date will be provided with each stock. These solutions are purchased as a set of nine from AccuStandard. NO<sub>2</sub> and NO<sub>3</sub> are expressed in terms of nitrogen. A minimum of five points shall be used for each calibration curve. The concentrations of the daily curve are listed in Table 1 in units of micrograms (µg) per mL:

**Table 1** Concentration Curves (µg/mL)

| IC Curve | [Cl <sup>-</sup> ] | [NO <sub>2</sub> as N] | [Br <sup>-</sup> ] | [NO <sub>3</sub> as N] | [SO <sub>4</sub> <sup>2-</sup> ] |
|----------|--------------------|------------------------|--------------------|------------------------|----------------------------------|
| STD 1    | 0                  | 0                      | 1.0                | 0                      | 0                                |
| STD 2    | 0.008              | 0.004                  | 1.0                | 0.008*                 | 0.040*                           |
| STD 3    | 0.020*             | 0.010*                 | 1.0                | 0.020                  | 0.10                             |
| STD 4    | 0.040              | 0.020                  | 1.0                | 0.040                  | 0.20                             |
| STD 5    | 0.10               | 0.050                  | 1.0                | 0.10                   | 0.50                             |
| STD 6    | 0.30               | 0.150                  | 1.0                | 0.30                   | 1.50                             |
| STD 7    | 0.60               | 0.30                   | 1.0                | 0.60                   | 3.0                              |
| STD 8    | 1.0                | 0.50                   | 1.0                | 1.0                    | 5.0                              |
| STD 9    | 2.0                | 1.0                    | 1.0                | 2.0                    | 10.0                             |
| STD 10   | 3.0                | NA                     | 1.0                | 3.0                    | 15.0                             |

**Notes:** \*Indicates concentration of reporting limit  
STD = calibration standard

- Control standards used as continuing calibration verification (CCV) spikes are purchased from High Purity Standards (HPS). A Certificate of Traceability and an expiration date is provided with each lot. To ensure calibration accuracy during each analytical run, a CCV is used to verify accuracy.
- Standard Reference Material (SRM) reference solution is purchased as a ready-to-use NIST-traceable standard solution containing analytes of interest (e.g., Simulated Rainwater #2 from HPS or equivalent) with a certificate of analysis and an expiration date. Note that the SRM is used to verify the calibration and must be from a source other than that of the calibration standards.
- Bromide stock solution, 1,000 milligrams (mg) per L, is made by adding 2.06 g of Rubidium Bromide (RbBr) to a liter of DI water. CAS# 7789-39-1

Figure 3 Certificate of Traceability

|  |  |  |
|--|--|--|
| 125 Market Street<br>New Haven, CT 06513<br>USA  |  <b>AccuStandard, Inc.</b>                    | Tel (203) 786-5290<br>Fax (203) 786-5287<br>Website AccuStandard.com |
| <b>CERTIFICATE OF ANALYSIS</b>   |  |  |
| <b>Acculon™ Reference Standard</b>   |  |  |
| Item Number: IS-7966B-R2   |  |  |
| Mix Name: Custom IC Standard   | Lot Number: 210015038  |  |
| Matrix: Water, tr. CHCl <sub>3</sub>   | Expiration Date: Jul / 2011  |  |
| Concentration in µg/mL   |  |  |
| Br   | <u>1000</u>  | 3184   |
| Cl   | <u>8</u>   | 3182   |
| NO <sub>3</sub> -N   | <u>8</u>   | 3185   |
| SO <sub>4</sub>  | <u>40</u>  | 3181   |
| <p>The total maximum uncertainty on the certified value(s) is ±2%. See reverse side of certificate for details.</p> <p>RESULTS: This solution standard was certified for accuracy of the listed components via methodology traceable to primary or well characterized secondary standards.</p> <p>This standard was prepared gravimetrically to contain the concentrations shown above. Balances, used in the preparation, are calibrated regularly using NIST-traceable weights. All glassware used in preparation is Class A.</p> <p>We use the highest purity raw materials available, ASTM type I 18 megohm deionized water, and typically 99.999%+ starting materials to minimize impurity levels in the final solution. All solutions are filtered through a 0.2 µm filter prior to being bottled.</p> <p>Use good laboratory procedure when diluting this product. Shake bottle prior to use and do not pipette directly out of the bottle. Use only cleaned Class A volumetric glassware.</p> <p>We certify the accuracy of this standard to be ± 0.5% of the stated value until the expiration date listed above, provided it is kept tightly capped and stored under normal laboratory conditions.</p> |  |  |
| Rec. 01/20/10<br>KSB   | <br>Lydia Snyder<br>Inorganic QC Supervisor |  |
| AccuStandard is accredited to ISO/IEC 17025:2005 and certified to ISO 9001:2000  |  |  |
| <small>DR-ORISO-001<br/>Rev. 9/08</small>  |  |  |

- Preparation of extraction solutions:
  - Extraction solution for cellulose filters: To a 4-L glass bottle add approximately 2 L of DI water; 4 mL of the 1,000 mg/L bromide stock; 6.7 mL 30 percent H<sub>2</sub>O<sub>2</sub>; and 10 mL concentrated HCl. Bring to 4 L with DI water.
  - Extraction solution for nylon filters: To a 4-L glass bottle add approximately 2 L of DI water; 4 mL of the 1,000 mg/L bromide stock; 6.7 mL 30 percent H<sub>2</sub>O<sub>2</sub>; and 6 mL of 2 N NaOH. Bring to 4 L with DI water.
  - Extraction solution for Teflon filters and denuders: To a 4-L glass bottle add approximately 2 L of DI water and 4 mL of the 1,000 mg/L bromide stock. Bring to 4 L with DI water.
- Acceptance criteria for extraction solutions:
  - Prepare extraction solution acceptance test samples as method blanks (MB) (see section 6.1.2 of this SOP), but do not take them through the extraction procedure.

Prior to use in extraction, each solution and the lowest concentration calibration standard (STD1) are analyzed by IC. In order to pass acceptance criteria, the responses for the target analytes of each solution must be less than the responses of STD1. Each of the target analytes must pass. If not, the solution is remade and reanalyzed.

- See GLO3180-10 for cellulose filter impregnation solution

## 5.0 SAFETY

**The analyst must be aware of the hazards associated with the chemicals used in this method.** Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

## 6.0 PROCEDURE

### 6.1 Sample Extraction

- 6.1.1 Pull the appropriate number of samples from the cold room. **Note:** There is no extraction for water samples.
- 6.1.2 Prepare a method blank (BLK). For Teflon and nylon filters, the BLK is an empty, labeled bottle. For cellulose filters, add 600  $\mu\text{L}$  of the cellulose filter impregnation fluid to an empty, labeled bottle. BLK is extracted as regular samples.
- 6.1.3 A blank spike (BS) or laboratory control sample (LCS) is prepared by adding the appropriate extraction fluid to the bottle, removing 1 mL, and adding 1 mL of anion BS solution with concentrations of 5 parts per million (ppm) for  $\text{Cl}^-$ , 2.5 ppm for  $\text{NO}_3^-$  as N, and 12.5 ppm for  $\text{SO}_4^{2-}$ . The extracted instrument targets are 0.2  $\mu\text{g}/\text{mL}$  for  $\text{Cl}^-$ , 0.1  $\mu\text{g}/\text{mL}$  for  $\text{NO}_3^-$ , and 0.5  $\mu\text{g}/\text{mL}$  for  $\text{SO}_4^{2-}$ . For Teflon extractions, a cation BS is also included. For cellulose, 2 mL is used since the extraction volume is 50 mL. A 600  $\mu\text{L}$  aliquot of the cellulose filter impregnation fluid is added to the cellulose BS prior to removing the 2 mL. Both the anion and cation BS solutions are custom made by HPS.
- 6.1.4 Select the proper extraction fluid for the extraction.
- 6.1.5 Calibrate a repipettor to 25 mL for Teflon and nylon, 50 mL for cellulose, and 20 mL for denuders. Tare the first bottle and a bottle every 10 samples. Pipette the extraction solution and reweigh the bottle to check the reproducibility of the repipettor. The acceptable limits are  $25.00 \pm 0.10$  g for Teflon and nylon,  $50.00 \pm 0.20$  g for cellulose,

and  $20.00 \pm 0.10$  g for denuders. Record the checked weights in the “Notes” section of the run log.

- 6.1.6 Extract the samples according to filter type (see Figure 1). Visually confirm that the proper filter type is in the bottle with each sample.
- Teflon and cellulose: 15 minutes shake, 30 minutes sonication, and 15 minutes shake. Teflon filters use the “high” shaker setting. Cellulose filters use the “low” setting.
  - Nylon: 30 minutes high-speed shake.
  - Denuders: 20 inversions and rotations (done by hand). After extraction, the denuder extract is transferred to a 30-mL Nalgene bottle.
- 6.1.7 A peer review will be done to verify that the sample IDs on the extract bottles match the extraction worksheet.

*Note: Sample extracts must be stored at  $4 \pm 2$  degrees Celsius ( °C) for at least 8 hours prior to analysis. This step ensures the reaction between the extraction fluid and the sample filter is complete.*

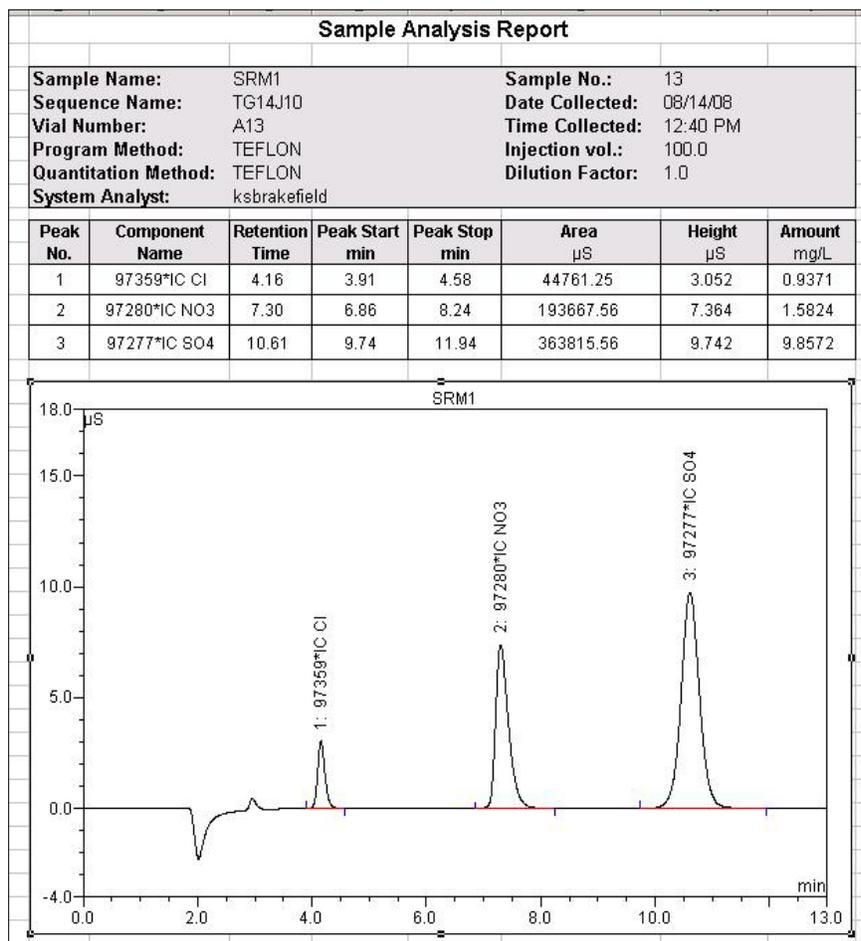
## 6.2 Sample Analysis

- 6.2.1 Establish a stable baseline by pumping the mobile phase (Section 4.2.2 of this SOP) through the instrument for at least 30 minutes. The operator’s manuals provided by the manufacturer contain detailed information regarding optimization of instrument performance and optimum operating criteria.
- 6.2.2 Load the auto-sampler vials beginning with the calibration curve followed by the extracted method blank (BLK1), the initial control standard (CCV1), the initial reference sample (SRM1), and extracted samples. Additional control standards (CCV2 through CCV9, CCVA, and CCVB) should be run every 10 samples and at the end of the analytical run. A second RF (SRM2) should also be run preceding the end-of-run CCVx.
- Note: Verify that the run-log printout auto-sampler positions are correct and the extract bottle IDs match the runlog; initial when complete.*
- 6.2.3 Each auto-sampler vial cap is scored with a razor knife. The razor knife is rinsed with Deionized water and wiped with a Kim wipe prior to scoring the cap. A number of auto-sampler vial caps will be scored in advance and stored in a clean container for future use. This additional step prevents the auto-sampler needle from getting bent causing a bad injection/replacement. Samples that have responses exceeding the highest standard must be diluted and reanalyzed in the next available run.

### 6.3 Data Analysis

- 6.3.1 Data files are processed using algorithms contained in the data collection software. Thermo-Fisher Dionex Chromeleon 7.2 software is currently in use. Parameters are adjusted as dictated by instrument performance.
- 6.3.2 Examine all chromatograms visually (Figure 4). Note any anomalies in the data batch narrative.

**Figure 4** IC Chromatogram



6.3.3 Prepare the Element upload file (Figure 5) using the batch processing feature of the PeakNet software, and generate an upload file in final concentration as described in SOP GLO3180-35, Element Batch Preparation.

6.3.4 Generate data batch in Element and DataTool using the uploaded file (Figure 6). The analyst can note any analytical anomalies in a separate batch narrative. Print a hard copy of the data batch to be included in the data batch folder.

**Figure 5** Element Auto Batch

| SampleID   | Analysis         | Analyte          | Rpt | Lock | Status  | Qualifier | IResult | FResult      | Type |
|------------|------------------|------------------|-----|------|---------|-----------|---------|--------------|------|
| 0824006-01 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | -0.0001 | ND [0.00250] | PR   |
| 0824006-01 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0824006-01 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0828001-15 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0828001-15 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0.0118  | 0.2950       | PR   |
| 0828001-15 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 3.4391  | 95.98        | PR   |
| 0830001-28 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0.0023  | ND [0.05750] | PR   |
| 0830001-28 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0.0058  | ND [0.1450]  | PR   |
| 0830001-28 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 0.151   | 3.775        | PR   |
| 0830004-15 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0.0006  | ND [0.01500] | PR   |
| 0830004-15 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0830004-15 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0830004-26 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0.0005  | ND [0.01250] | PR   |
| 0830004-26 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0830004-26 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0830004-28 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0.0021  | ND [0.05250] | PR   |
| 0830004-28 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0.0037  | ND [0.09250] | PR   |
| 0830004-28 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 0.0167  | ND [0.4175]  | PR   |
| 0830004-35 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0830004-35 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0830004-35 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0831001-01 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0831001-01 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0.0074  | ND [0.1850]  | PR   |
| 0831001-01 | SO4-TEF          | SO4-TEF          | X   |      | Batched |           | 1.5382  | 38.46        | PR   |
| 0831001-02 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | X   |      | Batched |           | 0       | ND [0.000]   | PR   |
| 0831001-02 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | X   |      | Batched |           | 0.0065  | ND [0.1625]  | PR   |

**Figure 6** Element Data Transfer Completion

| SampleID   | Analysis         | Analyte          | Analyzed       | Result  | Type | Units |
|------------|------------------|------------------|----------------|---------|------|-------|
| 0824006-01 | CHLOR            |                  | 08/14/08 13:10 | -0.0001 | PR   | ug/mL |
| 0824006-01 | NO3 AS           |                  | 08/14/08 13:10 | 0       | PR   | ug/mL |
| 0824006-01 | SO4-TE           |                  | 08/14/08 13:10 | 0       | PR   | ug/mL |
| 0828001-15 | CHLOR            |                  | 08/14/08 13:25 | 0       | PR   | ug/mL |
| 0828001-15 | NO3 AS           |                  | 08/14/08 13:25 | 0.0118  | PR   | ug/mL |
| 0828001-15 | SO4-TE           |                  | 08/14/08 13:25 | 3.4391  | PR   | ug/mL |
| 0830001-28 | CHLOR            |                  | 08/14/08 13:40 | 0.0023  | PR   | ug/mL |
| 0830001-28 | NO3 AS           |                  | 08/14/08 13:40 | 0.0058  | PR   | ug/mL |
| 0830001-28 | SO4-TE           |                  | 08/14/08 13:40 | 0.151   | PR   | ug/mL |
| 0830004-15 | CHLOR            |                  | 08/14/08 13:54 | 0.0006  | PR   | ug/mL |
| 0830004-15 | NO3 AS           |                  | 08/14/08 13:54 | 0       | PR   | ug/mL |
| 0830004-15 | SO4-TEF          | SO4-TEF          | 08/14/08 13:54 | 0       | PR   | ug/mL |
| 0830004-26 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | 08/14/08 14:09 | 0.0005  | PR   | ug/mL |
| 0830004-26 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | 08/14/08 14:09 | 0       | PR   | ug/mL |
| 0830004-26 | SO4-TEF          | SO4-TEF          | 08/14/08 14:09 | 0       | PR   | ug/mL |
| 0830004-28 | CHLORIDE, TEFLON | CHLORIDE,TEFLON  | 08/14/08 14:24 | 0.0021  | PR   | ug/mL |
| 0830004-28 | NO3 AS N, TEFLON | NO3 AS N, TEFLON | 08/14/08 14:24 | 0.0037  | PR   | ug/mL |

- 6.3.5 Assemble the data batch folder, including the Element data batch, copies of all run logs, certificates of analyses and processing methods, hard copies of each chromatogram, and any other necessary documentation.
- 6.3.6 Complete the batch folder checklists (Attachments B and C), and submit it for review and finalization.
- 6.3.7 Archive all electronic files generated from data collection and processing in condensed form on the AMEC network located on P drive: \LAB\PROJECTS\ICDATA.
- 6.3.8 If data are exported to the Clean Air Status and Trends Network database, the BLK, SRM, and DUP prefixes will be changed to MB, RF, and RP respectively upon ingestion.

#### **6.4 Quality Control**

- 6.4.1 One BLK is analyzed with each extraction unless specified differently by a project. The BLK is deionized water for wet deposition samples. The BLK for extracted samples is the applicable volume of extraction solution followed by the appropriate extraction procedure. The BLK results must be less than or equal to two times the reporting limit for the analytes of concern.
- 6.4.2 A CCV is analyzed at a frequency of 10 percent for every analytical batch, unless specified differently by a project, as well as at the beginning and end of the run. The measured value of the CCV must be within  $\pm 5$  percent of the certified value.
- 6.4.3 A SRM is used for an initial and a final calibration verification. The measured value of the reference sample must be within  $\pm 5$  percent of the certified value.
- 6.4.4 Sample replicates (DUP1, DUP2) are analyzed at a frequency of  $\pm 5$  percent. For samples greater than five times the reporting limit, the relative percent difference (RPD) of the replicate samples must be with  $\pm 20$  percent. For samples with concentrations less than or equal to five times the reporting limit, the absolute difference between sample and replicate must be less than the reporting limit.
- 6.4.5 All curves must contain a minimum of five points for quadratic calculations and have a correlation coefficient greater than or equal to 0.995.

#### **6.5 Calculations**

- 6.5.1 All calculations are performed with data reduction algorithms that reside in the instrument software.
- 6.5.2 Separate calibration curves are prepared for each anion of interest by plotting the response (peak area) of standards against concentration values using quadratic regression in the instrument software. Sample concentrations are calculated using the quadratic equation for the curve. The analyst may eliminate points to improve accuracy

throughout the range of calibration but at least 5 points plus a blank must remain. The final concentration is uploaded into Element via DataTool.

- 6.5.3 Each Element analyses matrix includes the extraction volume conversion in a static data table. Any dilution factors are written in the electronic instrument analysis schedule, noted on the chromatogram, and used to calculate final concentration for single analyte IC analyses. For multi-analyte analyses, single analyte dilutions are manually entered in the data entry section of Element Autobatch and noted on the chromatogram.

## **6.6 Corrective actions**

Attachment A shows the corrective actions taken when the QC Samples are not within acceptance criteria.

## **6.7 Data review**

The checklist used by the analyst, peer reviewer, and supervisor in reviewing the analytical data are attached as Attachments B and C.

## **7.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0. Prepared for U.S. Environmental Protection Agency (EPA), Washington, D.C. Contract No. EP-W-09-028. Gainesville, FL.

Dionex Corporation. 2009. ICS-1600 Ion Chromatography System Operator's Manual, Rev. 01, Document No. 065290. March 2009

Dionex Corporation. 2006. Chromeleon Tutorial & User Manual, Version 6.80. June 2006.

Dionex Corporation. 1998. DX-500 Maintenance and Troubleshooting. Document Number 031116-Rev 02 1998.

U.S. Environmental Protection Agency (EPA). 1995. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/R-96/027, November.

U.S. Environmental Protection Agency (EPA). 1993. Methods for Chemical Analysis of Water and Wastes (EA 300.0) EPA-600/4-79-020, Revised August.

## **8.0 ATTACHMENTS**

Attachment A – Corrective Actions

Attachment B – Batch Folder Checklist

Attachment C – Batch Peer Review Checklist

Attachment D – Revisions Table

**ATTACHMENT A**  
**Summary of Corrective Action Procedures for Ion Chromatography**

| <b>Quality Control</b>   | <b>Acceptance Criteria</b>                                  | <b>Corrective Action</b>   |
|--|---|--|
| Calibration curve correlation coefficient  | $\geq 0.995$  | Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument, or document why data are acceptable. |
| Calibration curve responses  | Brackets all samples  | Dilute and reanalyze samples exceeding the calibration curve range, or document why data are acceptable.   |
| Reference standard (SRM)   | $\pm 5\%$ of the certified true value                       | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples, or document why data are acceptable.                                  |
| Control standard (CCV)   | $\pm 5\%$ of the certified true value                       | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV, or document why data are acceptable.    |
| MB (BLK)   | $\leq 2$ times the DL                                       | Determine the cause of blank problem. Reanalyze the sample, if necessary, or document why data are acceptable.   |
| Blank Spike (BS)   | $\pm 20\%$ of target  | Not established  |
| Sample Replicate (DUP)   | $\pm 20\%$ RPD if the sample is greater than 5 times the DL | Determine the cause of the problem. Reanalyze the sample, if necessary, or document why data are acceptable.   |
| <b>Notes:</b> DL = Detection limit<br>RPD = Replicate percent difference<br><br>Source: AMEC |   |  |

### ATTACHMENT B Batch Folder Checklist

L010020

|               |                                 |
|---------------|---------------------------------|
| Initial or NA |                                 |
| <u>KSB</u>    | CLASS batch printout            |
| <u>KSB</u>    | Method reference                |
| <u>KSB</u>    | Instrument logbook copies       |
| <u>KSB</u>    | Reagent/Standard prep logsheets |
| <u>KSB</u>    | Original raw data               |

|                                     |                         |
|-------------------------------------|-------------------------|
| <u>Kent Brahefield</u><br>Analyst   | <u>10/8/10</u><br>Date  |
| <u>Amil C. Lucas</u><br>Reviewed by | <u>10-08-10</u><br>Date |

Notes: ✓

[Signature] 10/8/10

### ATTACHMENT C



**Batch Folder and ELEMENT Batch Review Checklist**

Batch L010020

**Section 1.**  
 This section is to be completed by the analyst before submitting the batch and counter-signed during peer review. Peer reviewer also checks each statement for accuracy (Indicated by a check preceding the item). Any discrepancies must be corrected before the batch can be accepted. Any "No" response must have a comment.

|  | Yes                                 | No                                  | Comments                                       |
|--|-------------------------------------|-------------------------------------|--|
| <input checked="" type="checkbox"/> 1. All samples uploaded correctly.             | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 2. All duplicate sources entered correctly.    | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 3. All QC results within established criteria: |                                     |                                     |  |
| <input checked="" type="checkbox"/> MB   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> BS   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> CCV  | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> SRM  | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> DUP  | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 4. All curve coefficients $\geq 0.995$ .       | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 5. All responses within calibration range.     | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 6. All dilution factors entered correctly.     | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input type="checkbox"/> 7. Other electronic flags in the ELEMENT batch?           | <input type="checkbox"/>            | <input checked="" type="checkbox"/> | If Yes, describe in Additional Comments below. |

Analyst: *Scott Brakefield* Date: 10/8/10

Reviewed By: *Samuel C. Lessor* Date: 10-08-10

**Section 2.**  
 This section is to be completed by the reviewer. Initial each item. All items must be found to be true and be initialed before the batch can be accepted.

1. Instrument run log matches ELEMENT batch. (Exceptions allowed with sufficient documentation) DC
2. All appropriate documentation present. (includes, but is not limited to, standard prep logs, COA, code sheets, method print-outs, schedule print-outs, and run log copies.) DC
3. Raw data (chromatograms, spectra intensities, etc.) is acceptable. (Additional documentation may be required.) DC

Reviewed By: *Samuel C. Lessor* Date: 10-08-10

Additional Comments:

## ATTACHMENT D Revisions Table

Table - Revisions

| Document Revision No. | Section      | Revision   |
|-----------------------|--------------|--|
| 11.0                  | Attachment D | Addition of this table.  |
|                       | 6.2.2        | Inserted – <i>“Note: Verify that the run-log printout auto-sampler positions are correct; initial when complete.”</i>                                      |
| 12.0                  | 4.2.2        | Added – <i>“as N”</i> to NO <sub>2</sub> and NO <sub>3</sub> on Table 1. Also updated standard numbers to 1 through 10.                                    |
|                       | 6.1.7        | Added new section: <i>“A peer review will be done to verify the sample IDs on the extract bottles match the IDs recorded on the extraction worksheet.”</i> |
|                       | 6.2.2        | Added – <i>“and the extract bottle IDs match the runlog.”</i>  |
|                       | 6.2.3        | Inserted – <i>“A number of auto-sampler vial caps will be scored in advance and stored in a clean container for future use.”</i>                           |
|                       | 6.3.1        | Inserted – <i>“Thermo-Fisher Dionex Chromeleon 7.2 software is currently in use.”</i>  |
|                       | 6.3.7        | Inserted – <i>“... on the AMEC network located on P drive: \LAB\PROJECTS\ICDATA”</i>   |

**TITLE: AMMONIA AS NITROGEN FOR DRY AND WET DEPOSITION  
(MODIFIED EPA METHOD 350.1)**

Effective

Date:

April 2, 2015

Prepared by: Kathy W. Barry  
Laboratory Chemist

Kathy W Barry

Reviewed by: Garry L. Price  
Laboratory Operations Manager

Garry Price

Reviewed by: Marcus O. Stewart  
QA Manager

Marcus O. Stewart

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |        |       |            |
|---------------|--------|-------|------------|
| Reviewed by:  | Title: | Date: | Signature: |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |

## **AMMONIA AS NITROGEN FOR DRY AND WET DEPOSITION (MODIFIED EPA METHOD 350.1)**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the determination of ammonia plus ammonium ( $\text{NH}_3 + \text{NH}_4^+$ ) as nitrogen (N) in air filter, denuder, and water samples.

### **2.0 SCOPE**

This method is applicable to the determination of  $\text{NH}_3 + \text{NH}_4^+$  in wet and dry deposition samples for CASTNET and CASTNET-like projects. The method used to collect and handle samples will determine the proportion of  $\text{NH}_3$  and  $\text{NH}_4^+$ . It is assumed that the CASTNET sampling method collects particulate  $\text{NH}_4^+$  using an open-faced filter pack.

The analytical concentration range is 0.02 to 2.0 micrograms per milliliter ( $\mu\text{g}/\text{mL}$ ) of  $\text{NH}_4^+$  as nitrogen ( $\text{NH}_4^+\text{-N}$ ). The upper concentration limit may be extended by sample dilution. The reported detection limit is 0.02  $\mu\text{g}/\text{mL}$   $\text{NH}_4^+\text{-N}$ .

### **3.0 SUMMARY OF METHOD**

This automated procedure for the determination of  $\text{NH}_3 + \text{NH}_4^+$  as N uses the Berthelot Reaction, where the formation of a blue-colored compound believed to be closely related to indophenol forms after a solution of an ammonium salt is added to sodium phenoxide followed by the addition of sodium hypochlorite. A solution of ethylenediamine tetraacetic acid (EDTA) is added to the sample stream to eliminate the precipitation of the hydroxides of calcium and magnesium. Sodium nitroprusside is added to intensify the blue color which is then measured colorimetrically.

Reduced volume versions of this method using the same reagents and the same molar ratios are acceptable as long as all quality control (QC) and performance requirements outlined in this SOP are met.

#### **3.1 Method Interferences**

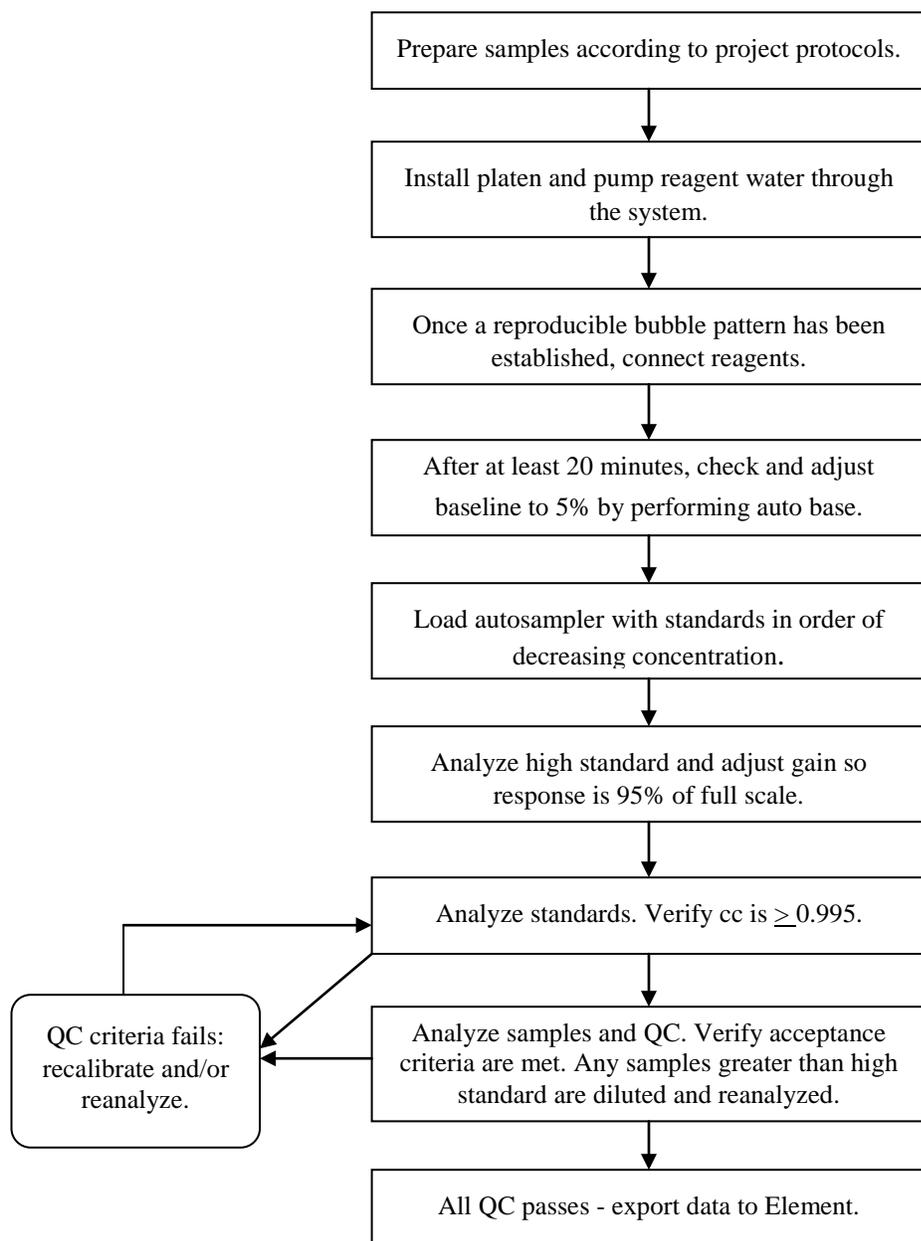
Ammonia is ubiquitous in the environment and may easily contaminate samples. Floor strippers and waxes commonly contain  $\text{NH}_3$  and are, therefore, a potential contamination source. Their use should be prohibited in any laboratory area where  $\text{NH}_3 + \text{NH}_4^+$  analyses are performed. To help avoid contamination, wash sample cups with deionized water prior to use and rinse the cups again with a portion of the standard or sample to be analyzed. Avoid allowing sample cups that contain any solutions to sit for prolonged periods prior to analysis.

High concentrations of calcium and magnesium ions can cause precipitation problems. A 4 percent EDTA solution helps suppress precipitation by chelating these metal cations.

### 3.2 Deviations from the Method

A general overview of the steps described in this SOP is depicted Figure 1. Deviations from the analytical method described in this SOP are not permitted.

**Figure 1** Overview – Laboratory Operations Procedures for Determination of Ammonia

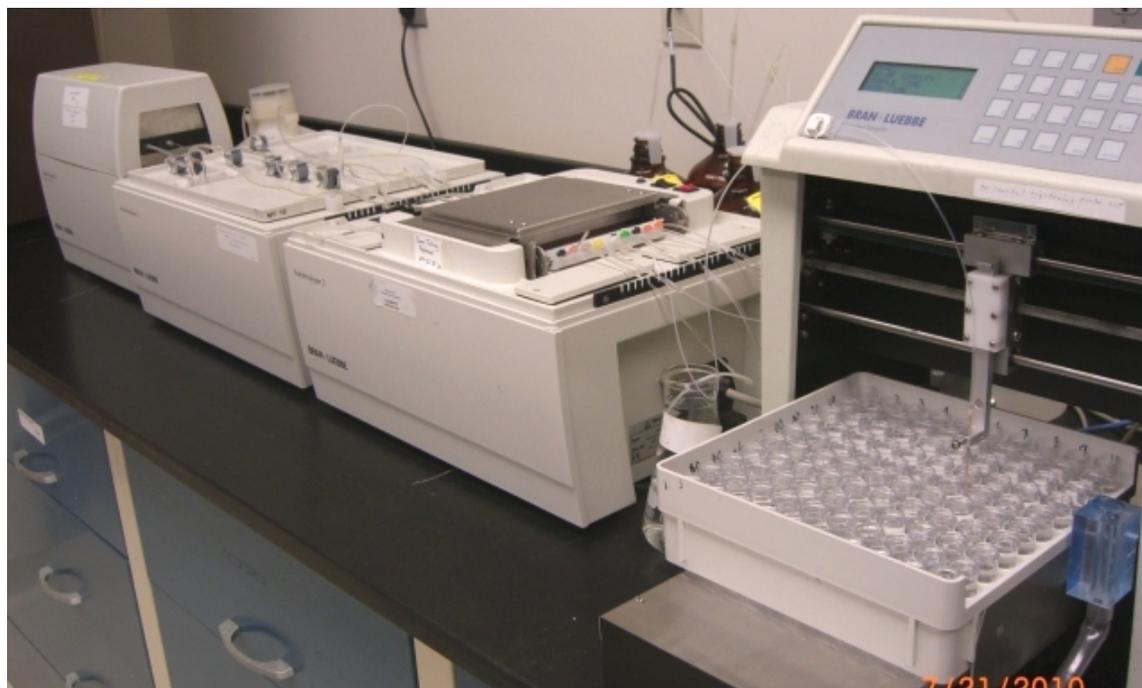


## 4.0 MATERIALS

### 4.1 Apparatus

- Analytical balance, sensitive to 0.1 milligrams (mg).
- Glassware: Class A volumetric flasks and pipettes as required.
- Bran+Luebbe Auto Analyzer 3 (see Figure 2): automated continuous flow analysis equipment designed to deliver and react sample and reagents in the required order and ratios. With the following:
  - Compact Auto-sampler with random access, PC or manually controlled.
  - High precision pump with multi-speeds, PC or manually controlled.
  - Chemistry module manifold with leak detector.
  - High sensitivity digital colorimetric detector with 660 nanometer (nm) filter and 10.0 millimeter (mm) by 1.0 mm (inside diameter) flow cells
- Autosampler vials, 4 mL and 5 mL.

**Figure 2** Bran+Luebbe Auto Analyzer 3



### 4.2 Reagents

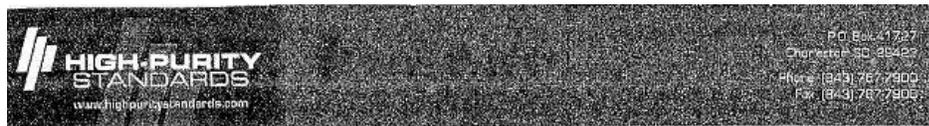
- Reagent Water: Deionized (DI) water of resistivity of 15 mega Ohms (M $\Omega$ ) or greater, derived from mixed bed ion exchangers, activated carbon filters and polishing exchangers. Water should contain particles no larger than 0.20 micrometers .

- Sodium hydroxide (NaOH), 10 Normal: Purchased commercially as a certified solution. NOTE: Highly caustic; use caution when handling. CAS# 1310-73-2
- Sodium Phenolate: Add 67 milliliters (mL) of 10 Normal NaOH solution (see note for NaOH) and 61.5 mL of liquefied phenol (commercially prepared reagent, approximately 89 percent phenol) to approximately 500 mL of reagent water. Allow the solution to cool, then dilute to 1000 mL (1 L) with reagent water. Store in an amber glass bottle. CAS# for Phenol is 108-95-2. Prepare every two weeks.
- Brij-35: Commercial surfactant solution available from VWR Scientific as CAS number 9002-92-0.
- EDTA: Add 1 mL 10 Normal NaOH to about 650 mL of DI water in a 1-liter (L) volumetric flask. While magnetically stirring, slowly add 33.6 grams (g) of EDTA. Add 2.0 mL of Brij-35. Mix well. Final volume 1-liter . Highest consumption reagent. Make as needed or every month.

*Note: EDTA is known and sold under several names. Always crosscheck the Chemical Abstracts Service (CAS) number for this reagent prior to ordering or using it. The CAS number is 6381-92-6.*

- Sodium Hypochlorite (NaOCl) solution: Mix 350 mL of a bleach solution containing 6.0 percent NaOCl (such as Clorox) to final volume of 1 L of reagent water. Store in an amber bottle, and prepare fresh every week. Since Clorox is a proprietary product, its formulation is subject to change. The analyst should verify that the bleach solution states that it contains 6 percent NaOCl. Purchasing a sodium hypochlorite solution from a laboratory reagent supply company is inadvisable due to the difficulty in obtaining recently prepared solutions. CAS#7681-52-9. Prepare fresh weekly.
- Sodium Nitroprusside: Dissolve 3.5 g of sodium nitroprusside in 500 mL of reagent water and dilute to 1 L. CAS# is 13755-38-9. Prepare monthly.
- Sampler Wash: Add fresh DI water daily.
- Reference Standard: WasteWtR Simple Nutrients Quality Control standard, catalog number 505, from Environmental Resource Associates (ERA). Certified NH<sub>3</sub>-N value in the parts-per-million (ppm) range. The reference standard (SRM) is analyzed to verify calibration.
- Control Standards are continuing calibration verification (CCV) standards. High Purity Standards, certified value of 1 ppm. Non-cataloged product - special AMEC mix. Manufacturer's lot is different from the 1-ppm calibration curve standard. To ensure calibration accuracy throughout each analytical run, a CCV is used for continuing calibration verification.
- Calibration standards are purchased as National Institute of Standards and Technology (NIST)-traceable solutions from High Purity Standards as a non-cataloged product - special AMEC mix. A certificate of analysis and expiration date is supplied with each solution (see Figure 3). They require no dilution and have the following concentrations in µg/mL: 0.00, 0.02, 0.05, 0.10, 0.25, 0.50, 1.00, and 2.00.

Figure 3 Certificate of Analysis



# Certificate of Analysis

SM-405-015  
 (NH<sub>3</sub>-N Curve Set #1)  
 Lot # 1014608

| Source            | Source Purity | Matrix                              | Standard Concentration                       |
|-------------------|---------------|-------------------------------------|--|
| Ammonium Chloride | 99.999%       | H <sub>2</sub> O<br>+ Tr Chloroform | 2.0 mg/L ± 0.02 mg/L<br>NH <sub>3</sub> as N |

This spectrometric standard solution has been prepared from high-purity reference materials. The matrix is 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography (IC).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by IC against a second source.

This standard is valid for three months from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

\*Chloroform is added to the master solution, but due to the limited solubility in water, it is not measurably present in this sample.

Exp Date: SEP 01 2010  
 MSDS ATTACHED

*Theodore C. Rains*  
 Theodore C. Rains, Ph.D.  
 President

Rec.  
 06-03-10  
 DL

## 5.0 SAFETY

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

## 6.0 PROCEDURE

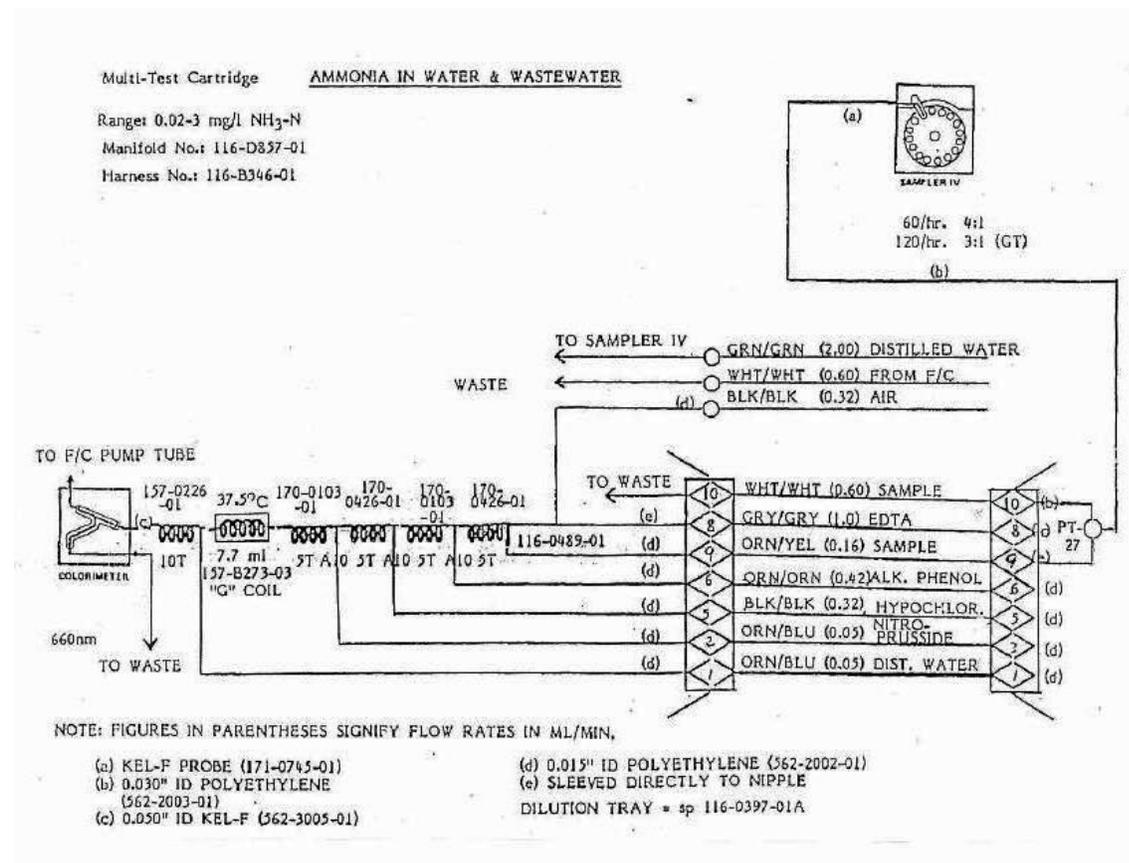
### 6.1 Sample Extraction

Samples are prepared and/or extracted according to specific project protocols.

### 6.2 Sample Analysis

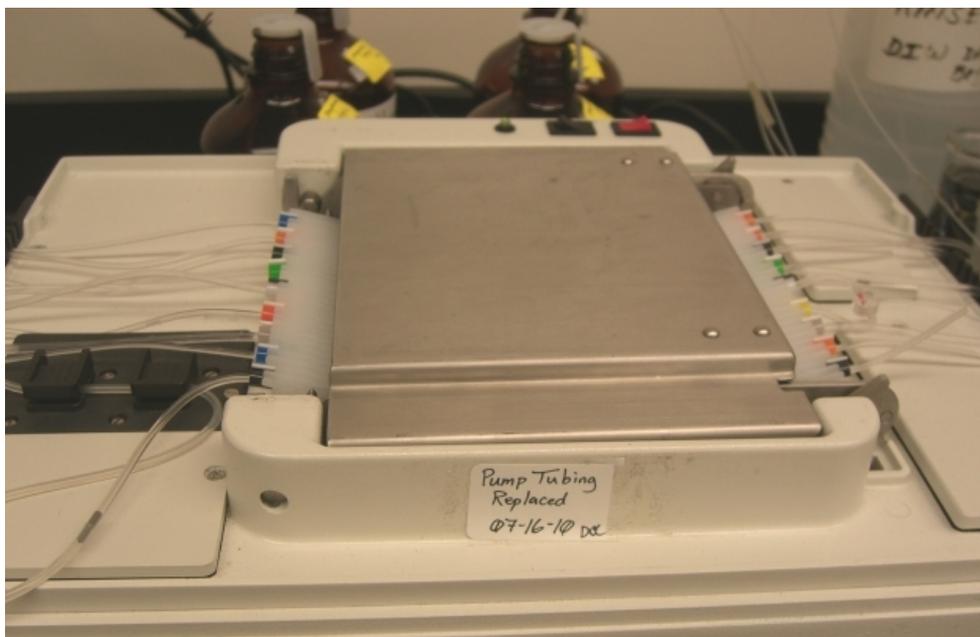
6.2.1 Connect tubing as shown in Figure 4.

**Figure 4** Pump Tubing and Coils for Auto Analyzer



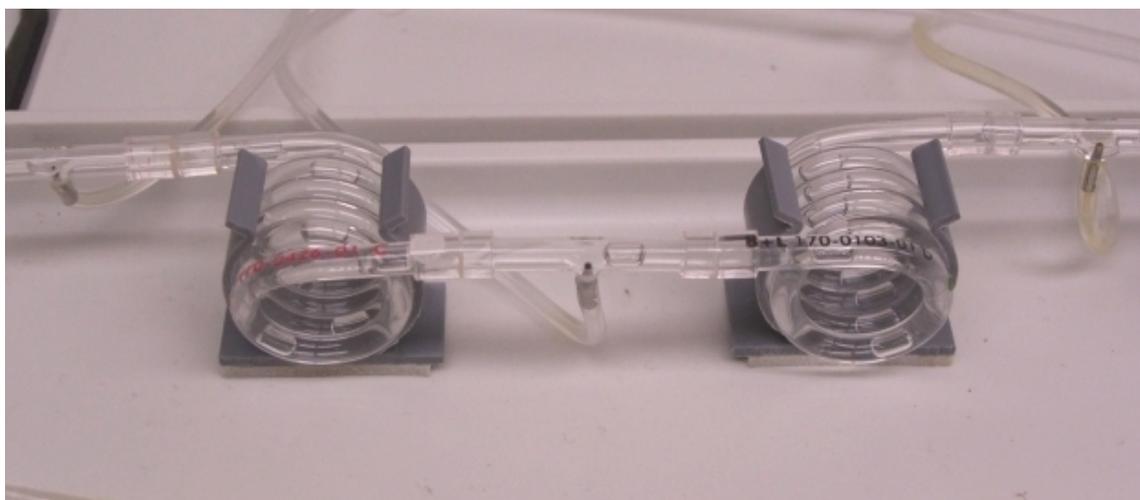
6.2.2 Place the platen on the pump (see Figure 5) and start the instrument. Pump reagent water through the system while it is warming up.

**Figure 5** Platen



6.2.3 Once a reproducible diagonal bubble pattern in the mixing coil has been established (see Figure 6), prepare the flow system for analysis by feeding reagents through the appropriate lines while feeding wash water through the sample line.

**Figure 6** Bubble Pattern

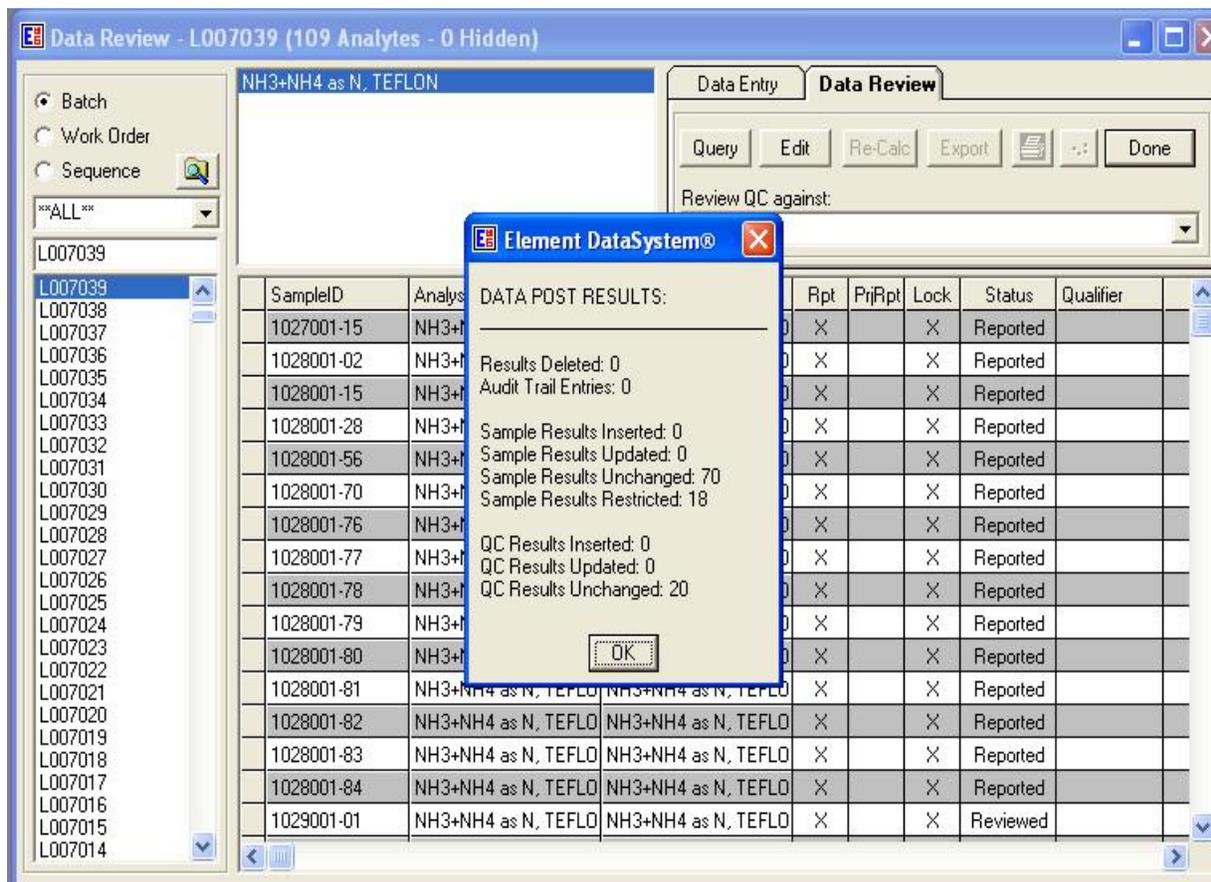


- 6.2.4 After the reagents have been pumped through the instrument for at least 20 minutes, check and adjust the baseline to 5 percent, if necessary. To adjust the baseline (while pumping reagents only), right click on the screen then click "Set Base". Note that large changes in the baseline reading may be an indication of contamination in the system or the reagents. The system can be cleaned by pumping a 1:2 Clorox solution through sample line for 10 minutes followed by 20 minute DI water rinse. Remake the reagents if necessary.
- 6.2.5 Check and adjust the full-scale response, if necessary, by analyzing the high standard and adjusting the gain such that its response is approximately 95 percent of the full scale of the instrument.
- 6.2.6 Place standards in the sampler in order of decreasing concentration. The high standard is analyzed twice, first as a primer then as a calibrant. Complete filling the sample tray with unknown samples. Note: Use the 5 mL autosampler vials for the high standard, blank calibrant and CCVs. The 4 mL vials are used for the remaining standards and samples. *Note: Verify that the run-log printout auto-sampler positions are correct; initial when complete.*
- 6.2.7 Begin analysis. See Bran+Luebbe AACE software operation manual for specific instructions.
- 6.2.8 Verify the reasonableness of blank carryover immediately following analysis of the standard curve. If carryover is determined to be unreasonable, investigate and correct the problem before continuing.
- 6.2.9 Rerun any sample analyzed after a high sample for verification that there was not carryover.

### **6.3 Data Analysis**

- 6.3.1 Export the data to Element (see Figure 6) using the batch processing feature outlined in SOP GLO3180-35 Element Batch Preparation (section 3.1.1.4).
- 6.3.2 The analyst should note any analytical anomalies in a separate batch narrative.
- 6.3.3 Assemble the data batch folder, including a hard copy of the Element data batch, copies of all run logs, certificates of analyses and processing methods, and any other necessary documentation.
- 6.3.4 Complete the batch folder checklists (Attachment B and C), and submit it for review and finalization.

**Figure 6** Element Batch



## 6.4 Quality Control

- 6.4.1 Method blanks (BLK) must be analyzed at a frequency of one per daily extraction. BLK concentrations should not exceed twice the reporting limit for the method. The reporting limit is 0.02 µg/mL.
- 6.4.2 Blank Spike (BS) will be analyzed at a frequency of one for every analytical run. Spikes are prepared as outlined in SOP GLM3180-001. The target concentration in the extract is 0.2 µg/mL.
- 6.4.3 A sample replicate (DUP) is analyzed at a frequency of 5 percent or every 20 samples. The criterion is ± 20 percent if the sample concentration is at least 5 times the detection limit (≥ 0.1 µg/mL); or ± the detection limit if the sample concentration is less than 5 times the detection limit (< 0.1 µg/mL).
- 6.4.4 The reference sample (SRM) must be run immediately following the daily calibration curve and at the end of the analyses. All values must be within 10 percent of their

respective true values or the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified prior to sample analyses.

- 6.4.5 The CCV must be run after every 10 samples and at the end of the sample run to verify that the standard curve is still valid. The CCV is normally a 1.0 µg/mL standard and must be within  $\pm 10$  percent of expected value. If the CCV is not within the specified range, reanalyze the CCV. If the recovery is still outside acceptable limits, the analysis must be terminated and the instrument recalibrated. All samples following the last acceptable CCV must be reanalyzed.
- 6.4.6 If data are uploaded to the Clean Air Status and Trends Network database, the BLK, SRM, and DUP prefixes will be changed to MB, RF, and RP respectively upon ingestion.

## **6.5 Calculations**

- 6.5.1 Calculations are performed with data reduction algorithms that reside in the Bran+Luebbe AACE instrument software Version 5.45.
- 6.5.2 Sample concentrations are calculated using the quadratic equation for the curve. The final concentration is uploaded into Element via DataTool.
- 6.5.3 Each Element analyses matrix includes the extraction volume conversion in a static data table. Final results are corrected for any sample dilutions in the Data Entry/Review section of Element™.

## **6.6 Corrective actions**

Attachment A shows the corrective actions taken when the QC samples are not within acceptance criteria.

## **6.7 Data review**

The checklists used by the analyst, peer reviewer, and supervisor in reviewing the analytical data are shown in Attachments B and C.

## 7.0 REFERENCES

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan*, Revision 8.0. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

Bran & Luebbe. 2006. Bran & Luebbe AACE Software 6.03 Rev. 2 No. MT7-60EN-03, September 2006.

Bran & Luebbe. 1983. Technicon Auto Analyzer II Method No. 696-82W, Ammonia in Water and Waste-Water, Technicon Industrial Systems, April 1983.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

U.S. Environmental Protection Agency (EPA). 1993. Determination of Ammonia Nitrogen by Semi-Automated Colorimetry (EPA 350.1) EPA-600/4-79-020, Revision 2.0, August 1993.

U.S. Environmental Protection Agency (EPA). 1983. *Methods for Chemical Analysis of Water and Wastes* (EPA 150.1), EPA 600/4-79-020, revised March 1983.

## **8.0 ATTACHMENTS**

Attachment A – Corrective Actions

Attachment B – Batch Folder Checklist

Attachment C – Batch Peer Review Checklist

Attachment D – Revisions Table

**ATTACHMENT A**  
**Summary of Corrective Action Procedures for NH<sub>3</sub> + NH<sub>4</sub><sup>+</sup> as N**

| Quality Control  | Acceptance Criteria                                   | Corrective Action  |
|--|---|--|
| Calibration curve correlation coefficient                                | ≥ 0.995   | Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument, or document why data are acceptable. |
| Calibration curve responses  | Brackets all samples                                  | Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable.  |
| Reference sample (SRM)   | ±10% of the certified true value                      | Rerun SRM. If still out of control, recalibrate instrument and reanalyze samples, or document why data are acceptable.                                       |
| Control standard (CCV)   | ±10% of the certified true value                      | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV, or document why data are acceptable.    |
| Method Blank (BLK)   | ≤2 times the DL                                       | Determine the cause of blank problem. Reanalyze samples, if necessary, or document why data are acceptable.  |
| Blank Spike (BS)   | ± 20%   | No corrective action established yet.  |
| Sample Replicate (DUP)   | ±20% RPD if the sample is greater than 5 times the DL | Determine the cause of the problem. Reanalyze the sample, if necessary, or document why data are acceptable.   |
| <b>Note:</b> DL = Detection limit.<br>RPD = Relative percent difference. |   |  |

**Source:** AMEC Engineering and Consulting, Inc



## ATTACHMENT C Batch Peer Review Checklist



### Batch Folder and ELEMENT Batch Review Checklist

Batch L007039

**Section 1.**

This section is to be completed by the analyst before submitting the batch and counter-signed during peer review. Peer reviewer also checks each statement for accuracy (indicated by a check preceding the item). Any discrepancies must be corrected before the batch can be accepted. Any "No" response must have a comment.

|   | Yes                                 | No                                  | Comments                                       |
|---|-------------------------------------|-------------------------------------|--|
| <input checked="" type="checkbox"/> 1. All samples uploaded correctly.              | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 2. All duplicate sources entered correctly.     | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 3. All GC results within established criteria:  |                                     |                                     |  |
| <input checked="" type="checkbox"/> MB  | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> BS  | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> CCV   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> SRM   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> DUP   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 4. All curve coefficients $\geq 0.995$ .        | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 5. All responses within calibration range.      | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 6. All dilution factors entered correctly.      | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 7. Other electronic flags in the ELEMENT batch? | <input type="checkbox"/>            | <input checked="" type="checkbox"/> | If Yes, describe in Additional Comments below. |

Analyst: Amil C. Lucan

Date: 07-28-10

Reviewed By: K. Baum

Date: 7/28/10

**Section 2.**

This section is to be completed by the reviewer. Initial each item. All items must be found to be true and be initialed before the batch can be accepted.

1. Instrument run log matches ELEMENT batch. (Exceptions allowed with sufficient documentation) KWB
2. All appropriate documentation present. (Includes, but is not limited to, standard prep logs, COA, code sheets, method print-outs, schedule print-outs, and run log copies.) KWB
3. Raw data (chromatograms, spectra intensities, etc.) is acceptable. (Additional documentation may be required.) KWB

Reviewed By: K. Baum

Date: 7/28/10

Additional Comments:

## ATTACHMENT D Revisions Table

Table - Revisions

| Document Revision No. | Section      | Revision  |
|-----------------------|--------------|---|
| 11.0                  | Attachment D | Addition of this table.   |
|                       | 6.2.6        | Inserted – <i>“Note: Verify that the run-log printout auto-sampler positions are correct; initial when complete.”</i> |

**TITLE: INDUCTIVELY COUPLED ARGON PLASMA-ATOMIC  
EMISSION SPECTROMETRY: PERKIN-ELMER OPTIMA 7300  
DV (MODIFIED EPA METHOD 6010-B)**

Effective

Date: April 2, 2015

Prepared by: Kathy W. Barry  
Laboratory Chemist

Kathy W Barry

Reviewed by: Garry L. Price  
Laboratory Operations Manager

Garry L Price

Reviewed by: Marcus O. Stewart  
QA Manager

Marcus O. Stewart

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |        |       |            |
|---------------|--------|-------|------------|
| Reviewed by:  | Title: | Date: | Signature: |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |

# **INDUCTIVELY COUPLED ARGON PLASMA-ATOMIC EMISSION SPECTROMETRY: PERKIN-ELMER OPTIMA 7300 DV (MODIFIED EPA METHOD 6010-B)**

## **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the determination of inorganic cations in air filters, water, and annular denuders by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES).

## **2.0 SCOPE**

This method is applicable to the determination of the cations in rainwater, cloud water, lake water, snow, and in Teflon filter extract media as dry deposition. The cations in solution that are routinely analyzed by this method and their reporting limits are as listed in Table 1.

**Table 1** Cations and their Reporting Limits

| <b>Cations</b>                | <b>Reporting Limit</b> |
|-------------------------------|------------------------|
| Calcium (Ca <sup>2+</sup> )   | 0.006 ppm              |
| Magnesium (Mg <sup>2+</sup> ) | 0.003 ppm              |
| Potassium (K <sup>+</sup> )   | 0.006 ppm              |
| Sodium (Na <sup>+</sup> )     | 0.005 ppm              |

**Note:** ppm = parts per million.

## **3.0 SUMMARY OF METHOD**

This method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced via radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the lines are measured simultaneously by segmented-array charge-coupled-device detector (SCD). This instrument can be operated in the traditional mode of viewing the torch radially or by viewing the torch axially to achieve much lower detection limits.

### **3.1 Method Interferences**

Atomic spectrometry can experience both positive and negative analytical biases from a number of sources. Root causes for bias are generally due to spectral overlap and variation between the sample and standard matrix.

Spectral overlap, the incomplete resolution of closely spaced photo-peaks, can be addressed in more than one way. Different (fully resolved) peaks can be chosen for quantitation. In many cases it is impractical or impossible to completely decouple analytical peaks and the analyst may wish to generate inter-element correction factors (IEC). IECs are determined by analyzing coupled elements in single element solutions to determine the relative bias on the determination of the other elements. IECs

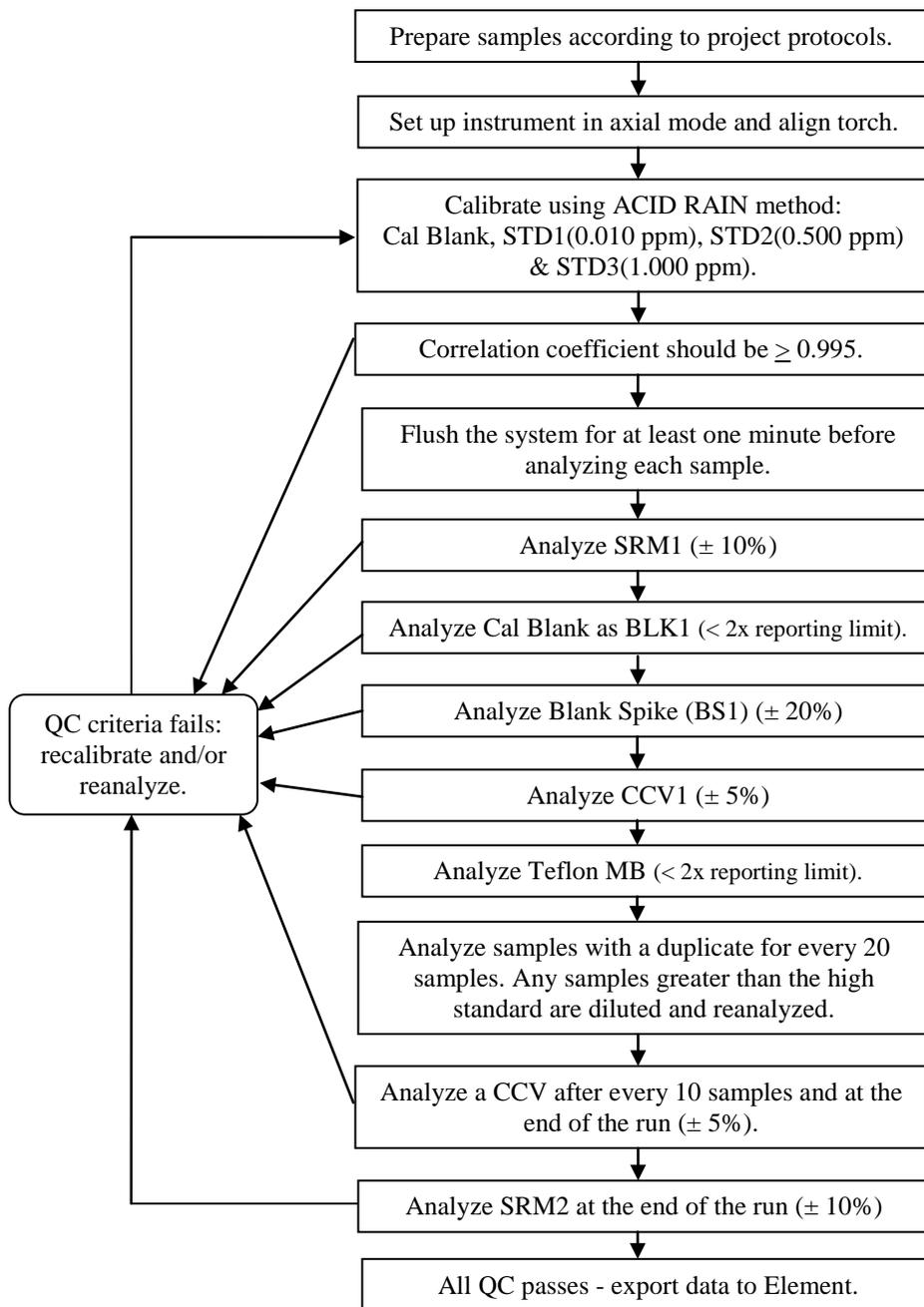
must be determined on a regular basis and verified prior to each analytical run. Data acquisition and reduction software then generates a sample result by solving the systems of equations generated by the analytical signal and the IEC.

Variation between the sample and standard matrix can also produce analytical bias. The most accepted solution to the problem is to closely match the calibration and reference matrices to the sample matrix. This approach can prove impractical or impossible when the sample matrix is unknown or when a number of disparate sample matrices are encountered. One possible solution for this case is the standard addition method of determining sample analyte concentration.

### **3.2 Deviations from the Method**

A general overview of the steps described in this SOP is depicted in Figure 1. Deviations from the analytical method described in this SOP are not permitted.

**Figure 1** Overview – Laboratory Operations Procedures for ICP-AES



## 4.0 MATERIALS

### 4.1 Apparatus

- Perkin-Elmer Optima 7300 DV ICP-AES Optical Emission Spectrometer-Plasma viewing axially (see Figure 2).
- S10 Autosampler
- NESLAB CFT-33m refrigerated recirculator or equivalent.
- Research Grade Argon.
- Class A volumetric flasks [200 and 100 milliliter (mL)].
- Adjustable Eppendorf micropipettes or equivalent.

**Figure 2** Perkin-Elmer Optima 7300 DV ICP-AES



### 4.2 Reagents

- Reagent Water: Deionized (DI) water of electrical resistivity of 15 mega Ohms ( $M\Omega$ ) or greater, derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 micrometers.
- High Purity Standards/ESE Special Mix #1 and the low standard ESE Simulated Rainwater (SR) Mix (see Table 2). Certificates of Analysis are required for all stock solutions purchased and are kept on file (see Figure 3). These are National Institute of Standards & Technology (NIST)-traceable Solutions obtained from High Purity Standards, Inc. of Charleston, SC.
- Mixed calibration standards made according to the outline in Table 2.

**Table 2** Acid Rain Standards

|  |  |
|--|--|
| Acid rain standards are made using the High Purity "SM-405-012 ESE Special Mix #1"<br>100 micrograms per milliliter ( $\mu\text{g}/\text{mL}$ ) $\text{Ca}^{2+}$ , $\text{Mg}^{2+}$ , $\text{Na}^+$ , $\text{K}^+$ stock solution.<br>Lot: XXXXXX      Expiration: XX/XX |  |
| 1.0 ppm  | 1.0 mL of stock solution is brought to a final volume of 100 mL with DI water.   |
| 0.5 ppm  | 1.0 mL of stock solution is brought to a final volume of 200 mL with DI water.   |
| 0.010 ppm  | This standard is not made daily – pre-made SM-405-005 ESE SR Mix from High Purity at 10-microgram per liter ( $\mu\text{g}/\text{L}$ ) concentration. Expiration is six months from shipping date. |
| Solutions diluted date XX/XX/XX expiration XX/XX/XX<br>All standards are dated upon receipt and preparation.   |  |

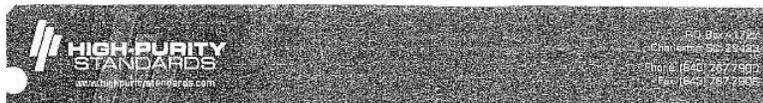
- Simulated Rainwater SR-2 Reference Sample (SRM) (also obtained from High Purity Standards, Inc.). The SRM is available in 250 mL, packaged as 5 x 50-mL bottles. The concentrations shown in Table 3 are the targeted values for each component. Each lot from High Purity comes with a certificate of analysis indicating the certified values. The SRM is used to verify the instrument calibration.
- Control Standards are continuing calibration verification (CCV) standards. The 0.5 ppm calibration standard in Table 2 is used to ensure calibration accuracy throughout each analytical run.

**Table 3** Simulated Rainwater

| Components                     | SR-2 Level ( $\mu\text{g}/\text{mL}$ ) |
|--------------------------------|--|
| Sodium ( $\text{Na}^+$ )       | 0.40                                   |
| Potassium ( $\text{K}^+$ )     | 0.10                                   |
| Calcium ( $\text{Ca}^{2+}$ )   | 0.05                                   |
| Magnesium ( $\text{Mg}^{2+}$ ) | 0.05                                   |

**Note:**  $\mu\text{g}/\text{mL}$  = micrograms per milliliter

Figure 3 Certificate of Analysis



## Certificate of Analysis

SM-405-012  
(ESE SP MX #1)  
Lot # 1004215

| Source                              | Source Purity | Matrix                | Standard Concentration  |
|-------------------------------------|---------------|-----------------------|---|
| High Purity Metals, Salts or Oxides | 99.98+%       | HNO <sub>3</sub> , 1% | 100 µg/mL ± 0.5%<br>Calcium<br>Magnesium<br>Potassium<br>Sodium |

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Exp Date: FEB 12 2011  
MSDS ATTACHED

*Theodore C. Rains*  
Theodore C. Rains, Ph.D.  
President

Rec.  
02-18-10  
DCL

### 5.0 SAFETY

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

Liquid argon, a cryogenic gas, is used in this procedure to generate a plasma environment for atomic spectra emission. The Chemical Abstracts System (CAS) number for argon is 7440-37-1. Argon is a simple asphyxiant, it constitutes a danger by reducing the oxygen level in confined spaces to below the concentration required to sustain life. This effect is particularly evident in the spills of liquid state

argon. Each liter of liquid argon vaporizes to fill 0.78 normal cubic meters of volume. Skin and eye contact with liquid argon is characterized by severe frostbite. Argon boils at (-) 185 degrees centigrade. Care must be taken not to obstruct the overpressure vent on any cryogenic gas dewar. Obstructed vents can lead to violent rupture of the container, rapid vaporization of the liquid, and direct exposure to the cryogen.

## 6.0 PROCEDURE

### 6.1 Sample Extraction

See SOP GLM3180-001, Section 6.1 for the extraction procedure.

### 6.2 Sample Analysis

- 6.2.1 Set up the instrument according to the manufacturer's recommendations and optimize the operating parameters. Select axial mode. Align the torch by using Tools, Spectrometer, then click "Hg Realign". If the torch was removed prior to the analysis, perform the "Hg Align" and "Align View" by aspirating a 1 ppm manganese solution.
- 6.2.2 Calibrate with the standards listed in Table 2, using the method ACID RAIN in the instrument software. *Note: Verify that the run-log printout auto-sampler positions are correct; initial when complete.*
- 6.2.3 Flush the system with DI water for at least 1 minute before analyzing each sample.
- 6.2.4 NIST-traceable reference samples (SRM1 and SRM2) must be analyzed after calibration, before any samples are analyzed, and at the end of the sample analysis. All values must be within 10 percent of their respective certified values or the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified prior to sample analyses.
- 6.2.5 Control standards (CCV1, CCV2... CCV9, CCVA) must be analyzed after every 10 samples and at the end of the run. The measured concentrations of the elements for which calibration was performed must be within 5 percent of their respective true values. If the CCV does not pass these criteria, re-analyze the CCV. If the CCV still fails criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified prior to continuing sample analyses. All samples run after the last acceptable CCV must be reanalyzed.
- 6.2.6 The method blank (BLK1) is analyzed prior to the analysis of samples. The method blank fails criteria if the results are greater than two times the reporting limit. If the method blank fails criteria, re-calibrate with fresh DI water. The calibration blank (DI water) is analyzed as the method blank. An extraction blank is also analyzed as a sample called a TMBxx (Teflon Method Blank) xx representing the year.
- 6.2.7 Analyze the samples with two replicate readings per sample, making dilutions for any elements that exceed the linear range. If the relative percent difference (RPD) between the replicate

readings is greater than 20 percent for an element whose concentration is 10 times the instrument reporting limit, repeat the analysis once. There is no criterion for samples whose concentration is less than 10 times the reporting limit.

6.2.8 A replicate sample (DUP) is analyzed at a frequency of 5 percent (1 per 20). The acceptance criteria is  $\pm 20$  percent for samples greater than five times the reporting limit and is  $\pm$  the reporting limit if the sample concentration is less than five times the reporting limit.

6.2.9 Aqueous samples are reported in final concentration by the instrument. If a dilution was required, the concentration in the diluted sample and the dilution factor are reported in the data batch.

6.2.10 A blank spike (BS1) equivalent to a Laboratory Control Sample (LCS) is created during the extraction process as outlined in SOP GLM3180-001. The target concentration in the extract for all four analytes is 0.1  $\mu\text{g}/\text{mL}$ .

### 6.3 Data Analysis

6.3.1 Prepare the Element upload file using the export feature of the Winlab software, and generate an upload file in final concentration as described in SOP GLO3180-35, Element Batch Preparation.

6.3.2 Generate data batch in Element and DataTool using the uploaded file (Figure 4). The analyst can note any analytical anomalies in a separate batch narrative. Print a hard copy of the data batch to be included in the data batch folder.

**Figure 4** Element Data Transfer Completion

The screenshot shows the 'Data Review' window in Winlab software. The window title is 'Data Review - L007041 (437 Analytes - 0 Hidden)'. The table below represents the data shown in the window. A 'Data Post Results' dialog box is overlaid on the table, displaying the following information:

- Results Deleted: 0
- Audit Trail Entries: 0
- Sample Results Inserted: 0
- Sample Results Updated: 0
- Sample Results Unchanged: 0
- Sample Results Restricted: 352
- QC Results Inserted: 0
- QC Results Updated: 0
- QC Results Unchanged: 64

| SampleID   | Analysis          | Analyte           | Rpt | PrPct | Lock | Status   | Qualifier  | RfResult | CalResult | Units                   | Diln | FfResult | Type | FUnits                | IMDL  | FMDL    | ILOD |
|------------|-------------------|-------------------|-----|-------|------|----------|------------|----------|-----------|-------------------------|------|----------|------|-----------------------|-------|---------|------|
| L007001-15 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 957118E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.4893   | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L007001-15 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 700509E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.9251   | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L007001-15 | SODIUM, TEFLON    | SODIUM, TEFLON    | X   | X     | X    | Reported | 954003E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.4385   | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-02 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 574534E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.6436   | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-02 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 398326E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.5397   | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-02 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 141601E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.035    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-02 | SODIUM, TEFLON    | SODIUM, TEFLON    | X   | X     | X    | Reported | 0.143495   |          |           | $\mu\text{g}/\text{mL}$ | 1    | 3.737    | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-15 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 603469E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 2.151    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-15 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 848539E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.7121   | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-15 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 174803E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.164    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-20 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 106009     |          |           | $\mu\text{g}/\text{mL}$ | 1    | 2.665    | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-20 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 622E-03    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.1855   | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-20 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 409E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.0479   | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-20 | SODIUM, TEFLON    | SODIUM, TEFLON    | X   | X     | X    | Reported | 639E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.5340   | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-56 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 338991E    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 8.499    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-96 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 737E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.6934   | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-56 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 689E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 2.148    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-56 | SODIUM, TEFLON    | SODIUM, TEFLON    | X   | X     | X    | Reported | 559E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.4645   | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-70 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 127281E    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 3.182    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-70 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 115E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.5003   | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-70 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 178E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.296    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-70 | SODIUM, TEFLON    | SODIUM, TEFLON    | X   | X     | X    | Reported | 839E-02    |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.6889   | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-76 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 208991     |          |           | $\mu\text{g}/\text{mL}$ | 1    | 5.238    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-76 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 376969E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.094    | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-76 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 971984E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.243    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-76 | SODIUM, TEFLON    | SODIUM, TEFLON    | X   | X     | X    | Reported | 131449E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 0.7829   | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-77 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 0.36       |          |           | $\mu\text{g}/\text{mL}$ | 3    | 27       | PR   | $\mu\text{g (total)}$ | 0.15  | 0.45    |      |
| L028001-77 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 531415E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.483    | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-77 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 138696E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.535    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-77 | SODIUM, TEFLON    | SODIUM, TEFLON    | X   | X     | X    | Reported | 072017E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.518    | PR   | $\mu\text{g (total)}$ | 0.125 | 0.12500 |      |
| L028001-78 | CALCIUM, TEFLON   | CALCIUM, TEFLON   | X   | X     | X    | Reported | 0.256003   |          |           | $\mu\text{g}/\text{mL}$ | 1    | 6.400    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |
| L028001-78 | MAGNESIUM, TEFLON | MAGNESIUM, TEFLON | X   | X     | X    | Reported | 981168E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.748    | PR   | $\mu\text{g (total)}$ | 0.075 | 0.07500 |      |
| L028001-78 | POTASSIUM, TEFLON | POTASSIUM, TEFLON | X   | X     | X    | Reported | 049749E-02 |          |           | $\mu\text{g}/\text{mL}$ | 1    | 1.262    | PR   | $\mu\text{g (total)}$ | 0.15  | 0.15000 |      |

- 6.3.3 Assemble the data batch folder, including the Element data batch, copies of all run logs, certificates of analyses and processing methods, hard copies of raw data, and any other necessary documentation.
- 6.3.4 Complete the batch folder check lists (Attachment B and C), and submit for review and finalization.
- 6.3.5 Archive all electronic files generated from data collection and processing in condensed form on the AMEC network.
- 6.3.6 If data are uploaded to the Clean Air Status and Trends Network database, the BLK, SRM, and DUP prefixes will be changed to MB, RF, and RP respectively upon ingestion.

#### **6.4 Quality Control**

- 6.4.1 One MB is analyzed with each extraction unless specified differently by a project. The MB is deionized water for wet deposition samples. The MB for extracted samples is the applicable volume of extraction solution followed by the appropriate extraction procedure. The MB results must be less than or equal to two times the reporting limit for the analytes of concern.
- 6.4.2 A CCV is analyzed at a frequency of 10 percent for every analytical batch, unless specified differently by a project, as well as at the beginning and end of the run. The measured value of the CCV must be within  $\pm 5$  percent of the certified value.
- 6.4.3 A SRM is used for an initial and a final calibration verification. The measured value of the reference sample must be within  $\pm 10$  percent of the certified value.
- 6.4.4 Sample replicates (DUP) are analyzed at a frequency of  $\pm 5$  percent. For samples greater than five times the reporting limit, the relative percent difference (RPD) of the replicate samples must be with  $\pm 20$  percent. For samples with concentrations less than or equal to five times the reporting limit, the absolute difference between sample and replicate must be less than the reporting limit.
- 6.4.5 All curves must have a correlation coefficient greater than or equal to 0.955.

#### **6.5 Calculations**

- 6.5.1 All calculations are performed with data reduction algorithms that reside in the instrument's operating software (WINLAB).
- 6.5.2 Analytical signals from the calibration sources are used to generate a separate polynomial curve for each cation of interest. These calculations include the slope, intercept, uncertainty, and correlation coefficient of the curve. The calibration data is used to relate the sample's analytical signal to a mass per volume concentration. The final concentration is uploaded into Element via DataTool.
- 6.5.3 Each Element analyses matrix includes the extraction volume conversion in a static data table. Any dilution factors are noted on the instrument analysis schedule and manually entered in the data entry section of Element.

## 6.6 Corrective actions

Attachment A shows the corrective actions taken when the QC samples are not within acceptance criteria. Most corrective actions within the scope of this SOP are responsive to failed QC parameters. Generally, optimizing the instrument analytical parameters and regenerating the analytical standards will alleviate these problems. Otherwise, a service technician should be consulted and the problem remedied.

## 6.7 Data review

The checklists used by the analyst, peer reviewer, and supervisor in reviewing the analytical data are attached as Attachments B and C.

## 7.0 REFERENCES

AMEC E&I, Inc. (AMEC). 2011. Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

Perkin Elmer. 2000. Perkin Elmer Winlab 32, 0993-6335, January 2000.

Perkin Elmer. 2008. Perkin Elmer Optima 7300 Hardware Guide, 0993-6878, June.

U.S. Environmental Protection Agency (EPA). 1995. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/R-96/027, November.

U.S. Environmental Protection Agency (EPA). 1996. Method 6010B, 2<sup>nd</sup> Revision - Test Methods for Evaluating Solid Wastes, EPA SW 846 3rd Edition, December.

U.S. Environmental Protection Agency (EPA). 1997. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA 540/R-94/013, February.

## **8.0 ATTACHMENTS**

Attachment A – Corrective Actions

Attachment B – Batch Folder Checklist

Attachment C – Batch Peer Review Checklist

Attachment D – Revisions Table

## Attachment A

### Summary of Corrective Action Procedures for ICP

| Quality Control  | Acceptance Criteria   | Corrective Action  |
|--|---|--|
| Calibration curve correlation coefficient                                | $\geq 0.995$  | Rerun calibration standards. If still out of control, prepare new calibration standards and recalibrate the instrument, or document why data are acceptable. |
| Calibration curve responses  | Brackets all samples  | Dilute and reanalyze samples within the calibration curve range, or document why data are acceptable.  |
| Reference standard (SRM)   | $\pm 10\%$ of the certified true value                      | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples, or document why data are acceptable.                                  |
| Control standard (CCV)   | $\pm 5\%$ of the certified true value                       | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV, or document why data are acceptable.    |
| Method Blank (BLK)   | $\leq 2$ times the DL                                       | Determine the cause of blank problem. Reanalyze the sample, if necessary, or document why data are acceptable.   |
| Blank Spike (BS)   | $\pm 20\%$ of target  | Not established  |
| Sample Replicate (DUP)   | $\pm 20\%$ RPD if the sample is greater than 5 times the DL | Determine the cause of the problem. Reanalyze the sample, if necessary, or document why data are acceptable.   |
| <b>Note:</b> DL = Detection limit.<br>RPD = Relative percent difference. |   |  |

Source: AMEC



## ATTACHMENT C

### Batch Peer Review Checklist



#### Batch Folder and ELEMENT Batch Review Checklist

Batch L007041

**Section 1:**

This section is to be completed by the analyst before submitting the batch and counter signed during peer review. Peer reviewer also checks each statement for accuracy (indicated by a check preceding the item). Any discrepancies must be corrected before the batch can be accepted. Any "No" response must have a comment.

|   | Yes                                 | No                                  | Comments                                       |
|---|-------------------------------------|-------------------------------------|--|
| <input checked="" type="checkbox"/> 1. All samples uploaded correctly.              | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 2. All duplicate sources entered correctly.     | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| 3. All QC results within established criteria:                                      |                                     |                                     |  |
| <input checked="" type="checkbox"/> MB  | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> BS  | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> CCV   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> SRM   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> DUP   | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 4. All curve coefficients $\geq 0.995$ .        | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 5. All responses within calibration range.      | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 6. All dilution factors entered correctly.      | <input checked="" type="checkbox"/> | <input type="checkbox"/>            |  |
| <input checked="" type="checkbox"/> 7. Other electronic flags in the ELEMENT batch? | <input type="checkbox"/>            | <input checked="" type="checkbox"/> | If Yes, describe in Additional Comments below. |

Analyst: Samuel C. Lucas

Date: 07-28-10

Reviewed By: Heidi Brakefield

Date: 7/29/10

**Section 2:**

This section is to be completed by the reviewer. Initial each item. All items must be found to be true and be initialed before the batch can be accepted.

1. Instrument run log matches ELEMENT batch. (Exceptions allowed with sufficient documentation) KS
2. All appropriate documentation present. (Includes, but is not limited to, standard prep logs, COA, code sheets, method print-outs, schedule print-outs, and run log copies.) KS
3. Raw data (chromatograms, spectra intensities, etc.) is acceptable. (Additional documentation may be required.) KS

Reviewed By: Heidi Brakefield

Date: 7/29/10

Additional Comments:

## ATTACHMENT D

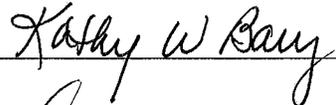
### Revisions Table

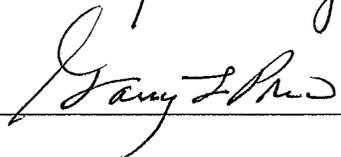
Table - Revisions

| Document Revision No. | Section      | Revision  |
|-----------------------|--------------|---|
| 9.0                   | Attachment D | Addition of this table.   |
|                       | 6.2.2        | Inserted – <i>“Note: Verify that the run-log printout auto-sampler positions are correct; initial when complete.”</i> |

**TITLE: DETERMINATION OF HYDROGEN ION (pH) IN WATER  
 (EPA METHOD 150.1)**

Effective Date: April 6<sup>th</sup> 2015

Prepared by: Kathy W. Barry  
 Laboratory Chemist 

Reviewed by: Garry L. Price  
 Laboratory Operations Manager 

Reviewed by: Marcus O. Stewart  
 QA Manager 

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |        |       |            |
|---------------|--------|-------|------------|
| Reviewed by:  | Title: | Date: | Signature: |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |

## **DETERMINATION OF HYDROGEN ION (pH) IN WATER (EPA METHOD 150.1)**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the determination of hydrogen ion (pH) in water.

### **2.0 SCOPE**

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes, and acid rain (atmospheric deposition).

### **3.0 SUMMARY OF METHOD**

The pH of a sample is determined potentiometrically using a combination electrode.

#### **3.1 Method Interferences**

Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10.0, the measured pH may be incorrectly low. Using a low-sodium error electrode can minimize this error. Strongly acidic solutions with a true pH <1.0 may give incorrectly high pH measurements. Strongly acidic or basic solutions are unlikely in atmospheric deposition samples.

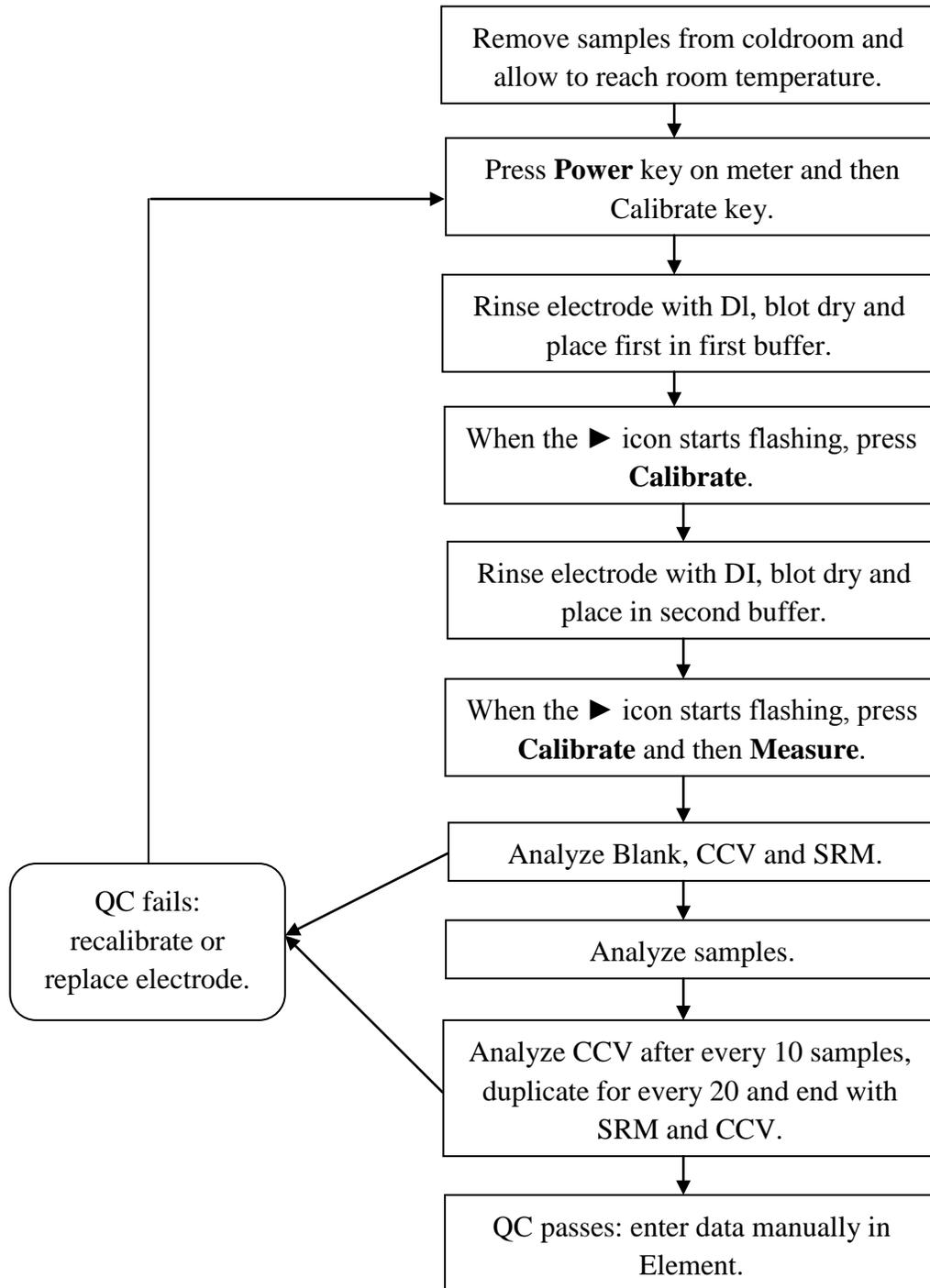
Temperature fluctuations will cause measurement errors including mechanical effects caused by changes in the electrode properties as well as chemical effects caused by equilibrium changes. Errors of this nature can be most effectively minimized by allowing all solutions to equilibrate to the same temperature before beginning analysis. Always record the temperature at which pH is measured.

Errors will occur when the electrode becomes coated with any type of adhering substance (i.e., oil or particulate matter). If this occurs, follow the steps outlined in the User's Guide to clean the electrode.

#### **3.2 Deviations from the Method**

A general overview of the steps described in this SOP is depicted Figure 1. Deviations from the analytical method described in this SOP are not permitted.

**Figure 1** Overview – Laboratory Operations Procedures for pH Measurement

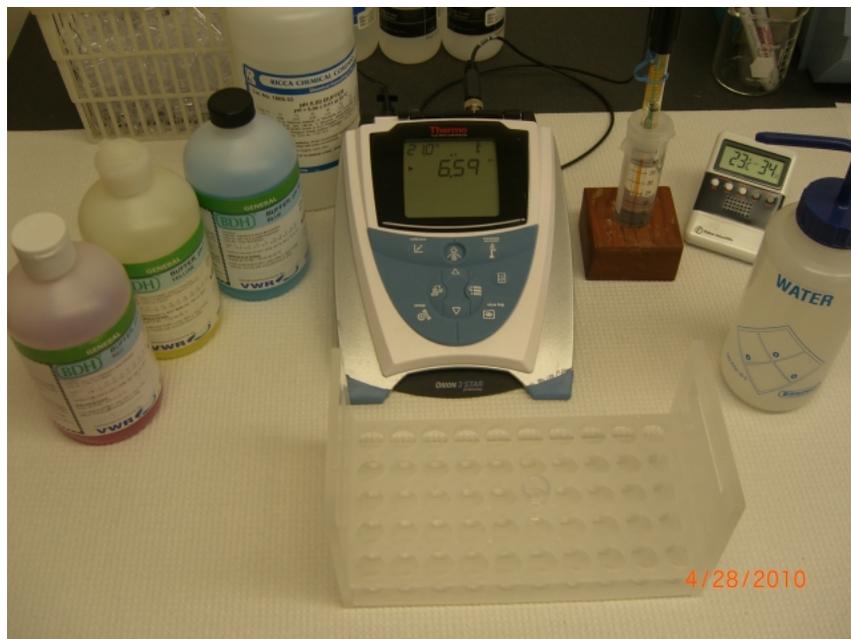


## 4.0 MATERIALS

### 4.1 Apparatus

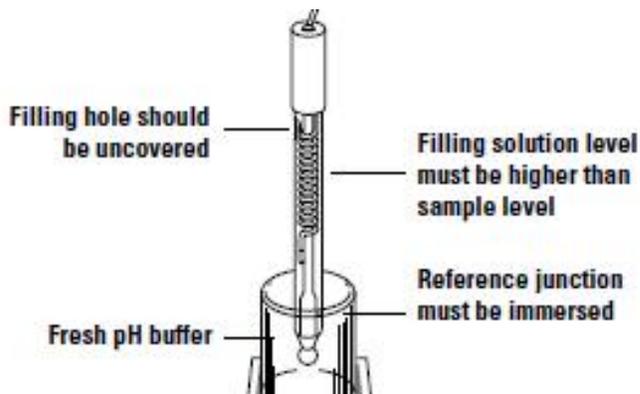
- pH meter with automatic temperature compensation (ATC) capabilities, Thermo Orion 3-Star pH bench-top meter, PN: 1112000, or equivalent (see Figure 2).

**Figure 2** pH Meter



- Glass combination semi-micro electrode, Orion ROSS Ultra PN: 8103BNUWP, or equivalent.
- The electrode should be every six (6) months.
- Analytical vessels capable of containing sufficient sample to cover the reference junction of the probe (see Figure 3). Use the smallest vessel capable of allowing sufficient submersion of the probe to obtain a reading.
- Kimwipes.
- Wash bottle filled with DI water for rinsing the probe between samples.
- Thermometer, for ambient room temperature readings.
- Digital scanning thermometer, for surface temperature readings of the solutions.

**Figure 3** pH Electrode



## 4.2 Reagents

- Reagent water: DI water with resistivity of 15 mega ohms ( $M\Omega$ ) or greater, derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 micrometers ( $\mu m$ ).
- Standard buffer solutions of 4, 7, and 10 pH units, which are available from a commercial vendor. Alternatively, standard buffer solutions of 3, 5, and 8 pH units (also available commercially) may be used. A Certificate of Analysis and an expiration date is provided with each lot (see Figure 4).

**Note:** *pH 10 buffer solution is subject to contamination from microbial growth. Store out of the light and away from heat to minimize the risk of contamination. Always have at least one extra unopened bottle of this buffer on hand.*

- Electrode internal filling solution: 3 molar (M) potassium chloride (KCl) solution, or alternate solution as appropriate to the sample type, available from the electrode manufacturer or other vendor. See the probe User's Guide for instructions regarding the selection of an alternative filling solution.
- Electrode storage solution: Available from the manufacturer or other vendor. Alternatively, use a solution of 200 mL pH 7 buffer to which 1 g KCl has been added.
- Standard Reference Material (SRM) is purchased as a ready-to-use NIST-traceable standard solution (e.g., Simulated Rainwater #1 from High Purity Standards or equivalent) with a Certificate of Analysis and an expiration date. Note that the SRM is used to verify the calibration and must be from a source other than that of the calibration standards.
- Continuing calibration verification (CCV) is purchased from commercial vendor. A Certificate of Analysis and an expiration date is provided with each lot. The CCV is used to ensure calibration accuracy and check for drift during each analysis.

**Figure 4** Certificate of Analysis



1310 Goshen Parkway  
 West Chester, PA 19380  
 phone: 1-800-932-5000

**Certificate of Analysis**

**Buffer, Reference Standard, pH 7.00 ± 0.01 at 25°C (Color Coded Yellow)**

Lot Number: 2912258      Product Number: BDH5046      Expiration Date: NOV 2011      Manufacture Date: 12/9/2009

The certified value for this product is confirmed in independent testing by a second qualified chemist.

**Contains:**

| Name  | CAS#        | Grade                              |
|---|-------------|------------------------------------|
| Inert Dye   | Proprietary | Commercial Grade                   |
| Potassium Phosphate, Monobasic                      | 7778-77-0   | ACS                                |
| Preservative (No Mercury compounds or Formaldehyde) | Proprietary | Commercial Grade                   |
| Sodium Phosphate, Dibasic                           | 7558-79-4   | ACS                                |
| Water, Deionized                                    | 7732-18-5   | ACS, ASTM D 1193 (Type I), EP, USP |

| Test Name                                     | Assay Method         | Specification             | Result             |
|---|----------------------|---------------------------|--------------------|
| Appearance                                    | Clarity, Color, Odor | Clear, yellow, odorless   | Passed Test        |
| pH at 25 °C (traceable to NIST SRM 186 & 191) | pH determination     | 7.00 ± 0.01 pH at 25.0 °C | 7.01 pH at 25.0 °C |

Volumetric glassware complies with Class A tolerance requirements of ASTM E 288 and NIST Circular 434; it is calibrated before first use and recalibrated regularly in accordance with ASTM E 542 and NIST Procedure NBSIR 74-461. Balances are calibrated regularly with weights certified traceable to the NIST national mass standard. Thermometers and temperature probes are calibrated before first use and recalibrated regularly with a thermometer traceable to NIST standards. All products are prepared according to master documents that assure manufacture according to validated methods. Batch records document raw material traceability and production and testing history for each lot manufactured.

Shelf Life (unopened container):

| Part Number | Shelf Life |
|-------------|------------|
| BDH5046-16  | 24 months  |

Recommended Storage: 15°C - 30°C (59°F - 86°F)

*LaNelle Ohlhausen*  
 LaNelle Ohlhausen  
 Quality Assurance



**VWR.COM**  
 1.800.932.5000

This Certificate of Analysis is designed to comply with ISO Guide 31 "Reference Materials – Contents of Certificates and Labels."

*Rec'd 2/3/10  
 /kwrb*

Version: 0

## 5.0 SAFETY

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

## 6.0 PROCEDURE

### 6.1 Electrode Preparation

- 6.1.1 Remove the electrode from the storage solution and rinse any salt deposits from the exterior with DI water.
- 6.1.2 Check the level of the filling solution. The filling solution should be at least at the end of the coil and no higher than to the bottom of the filling hole.
- 6.1.3 If the probe has been unused for longer than a week, drain the solution using a syringe fitted with a plastic pipette tip and refill the chamber with fresh solution.
- 6.1.4 If KCl deposits are noted on the interior of the probe, rinse with several successive portions of filling solution, or in extreme cases, DI water followed by filling solution, until no deposits are observed floating in the filled reservoir.

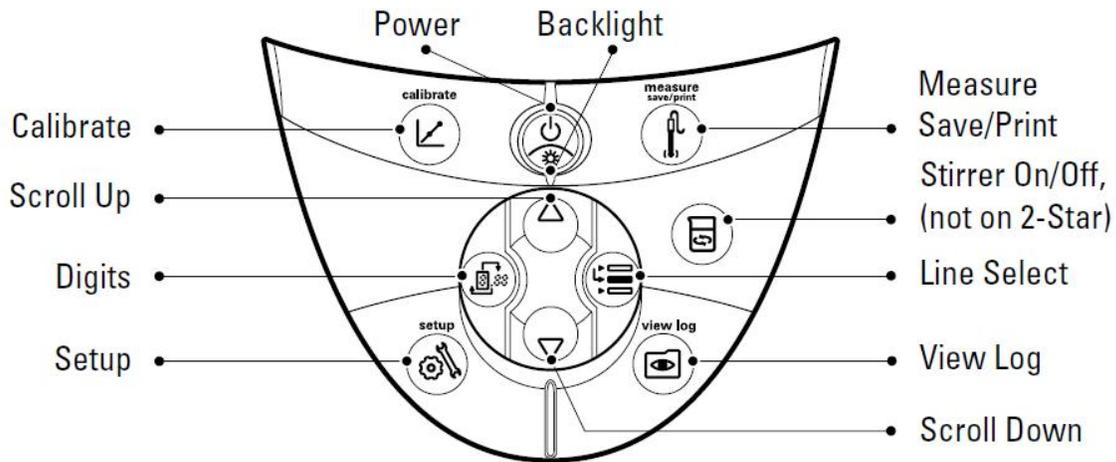
*Note: The deposits on the interior of the probe do not interfere with analysis, but if crystals break free of the inner walls and become suspended in the reference solution, changes in calibration can result requiring recalibration and subsequent reanalysis of affected samples. These deposits appear to be linked to prolonged periods of idleness. The probe should therefore be checked at least once a week to maintain optimum performance. See the User's Guide for directions on cleaning the probe.*

- 6.1.5 Keep the filling hole uncovered during analysis.

### 6.2 Set displayed meter temperature to match room temperature

*See Figure 5 for a diagram of the keypad for the Orion 3-Star Bench-top pH Meter.*

**Figure 5** Diagram of Orion 3-Star Bench-top Meter Keypad



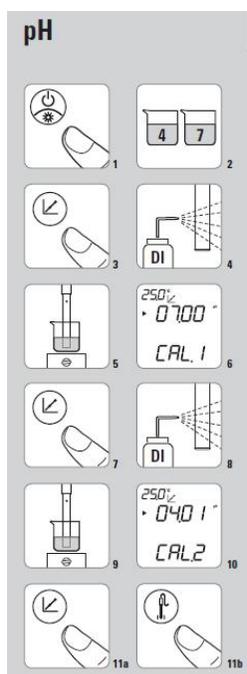
| Key | Description   | Key | Description   |
|-----|---|-----|---|
|     | Turns the meter on, if the meter is off.  |     | Changes the measurement units of the selected line in the measurement mode.   |
|     | Toggles the backlight on and off, if the meter is on (3-Star, 4-Star and 5-Star meters only).<br>If the meter is on, hold down the key for about three seconds to turn off the meter.   |     | Changes the value on the selected line in the setup, methods and log view modes.<br>Edits the value of the flashing digit for setup, password entry and calibration modes.  |
|     | Scrolls the arrow icon on left of screen among the three display lines, so the selected line can be edited or calibrated.   |     | Selects the next digit to edit and moves the decimal point for setup, password entry and calibration modes.   |
|     | Starts the calibration for the selected line in the measurement mode.<br>If the arrow icon points to the top line and the displayed units are pH, pressing the key will start a pH calibration.<br>Each time the key is pressed in the calibration mode, the meter will accept the calibration point and move to the next point until the maximum number of calibration points are reached. |     | Prints and logs a measurement in the continuous or timed measurement modes.<br>Prints, logs and freezes the display when the reading becomes stable in the AUTO-READ measurement mode.<br>Exits the setup menu and returns to measurement mode.<br>Accepts the calibration and returns to measurement mode. |
|     | Enters the setup menu, starting with selected line in the measurement mode.<br>If the arrow icon points to the top line and the displayed units are ISE, pressing the key will enter the ISE setup screen.  |     | Enters the log view and download menu.  |
|     |   |     | Turns the stirrer on and off.   |

- 6.2.1 Turn on meter.
- 6.2.2 Determine the room temperature by reading the thermometer next to the meter. Record this temperature in the logbook next to the pH 7 buffer lot number.
- 6.2.3 Use the **Setup** key to access the Setup menus.
- 6.2.4 Select the General Instrument Setup Menu screen by using the [▼] four times. “gen” will be displayed on the top line of the screen; “degC” will be displayed on the line below. The current temperature set-point will be displayed on the third line.
- 6.2.5 Access the temperature by pressing the **Line Select** key twice to highlight the set temperature. The first digit (a zero) will be blinking.
- 6.2.6 Use the **Digits** key to scroll across to the digit of interest, then use [▲] or [▼] to increase or decrease the digit in increments of one as needed to match the room temperature.
- 6.2.7 Press the **Line Select** key again to return to the Manual Temperature Menu.
- 6.2.8 Press the **Measure Save/Print** key to save the change. The temperature displayed in the upper left corner of the meter display should now correctly match the room temperature.

### 6.3 Calibration

*Note: This calibration method is for a two-point calibration utilizing the AutoBuffer recognition feature of the Orion 3-Star Bench-top pH meter (see Figure 6 for an overview of the calibration steps). Other calibration methods are listed in the manual for this meter and may be used in cases as approved by the Laboratory Manager and/or QA Manager.*

**Figure 6** pH Calibration Overview



- 6.3.1 Stir or gently agitate all buffers and standards prior to aliquoting and making measurements. Record the lot numbers of all buffers and standards used.
- 6.3.2 Fill two vessels with a portion of the pH 7 buffer (or alternately, the middle value buffer if points other than 4, 7, and 10 are being used). See Figure 6-2.
- 6.3.3 Press the **Calibrate** key to enter Calibration mode (see Figure 6-3). The “Calibrate” symbol will appear in the upper left corner of the display; “pH” and “CAL” will be displayed on two lines in the center of the screen for several seconds before displaying the measured pH.
- 6.3.4 Rinse the electrode with DI water and gently blot the probe free of drops with a Kimwipe (see Figure 6-4). Use care to not rub or wipe the surface of the probe when drying as static electric build-up could interfere with subsequent measurements.
- 6.3.5 Wait for the display to change to measure and immerse the probe in the first vessel (see Figure 6-5). Note: The pH of DI water is in the range of 5.6 – 5.8 pH units. The best sample results are obtained when the measured pH value displayed prior to immersing the probe in a new sample aliquot falls in this range. Avoid beginning a new measurement if the probe shows signs of drifting.
- 6.3.6 Swirl the probe gently to rinse before moving to the second aliquot. Stop swirling the probe and allow the electrode to equilibrate in the second aliquot.
- 6.3.7 When the meter recognizes the buffer the display will change to show the recognized value, and the first digit (a zero) will blink (see Figure 6-6).
- 6.3.8 Use the Digits key followed by [▼] or [▲] as needed to adjust the value to match the true value of the buffer. The true value of the buffer at various ambient temperatures should be listed on the side of the buffer bottle.
- 6.3.9 Press **Calibrate** (see Figure 6-7) to accept the value and move on to the second calibration point. “Cal 2” should now be displayed at the bottom of the screen.
- 6.3.10 Repeat steps 6.3.2 through 6.3.8 for the second buffer, usually pH 4 (see Figure 6-8 thru 6-11a).
- 6.3.11 Press **Measure Save/Print** (see Figure 6-11b) to accept the value and end the calibration procedure. The meter will automatically calculate the slope before proceeding to Measurement mode. Enter the slope in the logbook next to the lot number for the pH 4 buffer.

## 6.4 Verify Calibration

- 6.4.1 Fill two vessels with a portion of the pH 10 buffer.
- 6.4.2 Rinse the electrode with DI water and gently blot the probe free of drops with a Kimwipe. Wait for the display to change to measure and immerse the probe in the first vessel.

- 6.4.3 Swirl the probe gently to rinse before moving to the next aliquot. Stop swirling the probe and allow the electrode to equilibrate in the second aliquot.
- 6.4.4 A small “pH” will be blinking to the right of the measured value on the meter display when the electrode is equilibrating. This icon will stop blinking when a stable reading has been reached. Record the value of the stable reading.
- 6.4.5 A reading of  $10.00 \pm 0.05$  pH units indicates that the electrode has been properly calibrated, and sample analysis can begin (6.6). If the reading is outside of the accepted range, proceed to 6.5.

## 6.5 Calibration Verification Failure

- 6.5.1 Repeat step 6.4.1 through 6.4.4 using a new bottle of pH 10 buffer. If the measurement of the new buffer falls within the accepted range, proceed with sample analysis (6.6).
- 6.5.2 If the measurement of the new buffer does not fall within the accepted range repeat Calibration (6.3) with fresh aliquots of buffer solutions. Use a scanning thermometer to verify the temperatures of the solutions if temperature differences are suspected. Solutions should be  $\pm 1$  °C of the recorded temperature.
- 6.5.3 If after recalibrating the value still falls outside of the accepted range, empty and refill the filling solution chamber before calibrating and verifying the calibration again.
- 6.5.4 If steps 6.5.1 through 6.5.3 do not correct the problem, see Users Guide and follow the directions for cleaning the pH probe.
- 6.5.5 If steps 6.5.1 through 6.5.4 do not correct the problem, check with the Laboratory Manager.
- 6.5.6 Re-equilibrate the electrode by placing the probe back into the pH 4 buffer until the reading reaches the value recorded in the logbook.

## 6.6 Sample Analysis

*Note: Acid rain samples should not be stirred. Instead, swirl the samples gently for a few seconds after the introduction of the electrode. Allow the electrode to equilibrate. The air-water interface should not be disturbed while measurements are being made.*

- 6.6.1 Fill two vessels with a portion of the sample.
- 6.6.2 Rinse the electrode with DI water and gently blot the probe free of drops with a Kimwipe and immerse the probe in the first vessel.
- 6.6.3 Swirl the probe gently to rinse before moving to the next aliquot. Stop swirling the probe and allow the electrode to equilibrate in the second aliquot.
- 6.6.4 Record the value in the logbook once the reading has stabilized. Ignore any drifting of the displayed values unless the “pH” icon begins to blink again, in which case wait until stability returns before recording the value.

## 6.7 Data Analysis

- 6.7.1 Enter the data in Element (see Figure 7) using the manual batch processing feature outlined in SOP GLO3180-35 Element Batch Preparation (section 3.3).
- 6.7.2 The analyst should note any analytical anomalies in a separate batch narrative.
- 6.7.3 Assemble the data batch folder, including a hard copy of the Element data batch, copies of all run logs, certificates of analyses and processing methods, and any other necessary documentation.
- 6.7.4 Complete the batch folder checklists (see Attachments B and C), and submit it for review and finalization.

**Figure 7** Element Manual Batch

| SampleID | Analysis     | Analyte | Rpt | P/F/Pl | Lock | Status   | Qualifier | IResult | FResult    | Type | MDL   | LOD | MRL   | Units     | DIn | Recovery     |
|----------|--------------|---------|-----|--------|------|----------|-----------|---------|------------|------|-------|-----|-------|-----------|-----|--------------|
| L001024  | 0940009-01   | pH_LAB  | X   | X      | X    | Reported |           | 4.6     | 4.600      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001023  | 0940009-02   | pH_LAB  | X   | X      | X    | Reported |           | 4.6     | 4.600      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001022  | 0940009-03   | pH_LAB  | X   | X      | X    | Reported |           | 5.23    | 5.230      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001021  | 0940009-04   | pH_LAB  | X   | X      | X    | Reported |           | 4.63    | 4.630      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001019  | 0945008-01   | pH_LAB  | X   | X      | X    | Reported |           | 5.5     | 5.500      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001018  | 0945008-02   | pH_LAB  | X   | X      | X    | Reported |           | 5.59    | 5.590      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001017  | 0945008-03   | pH_LAB  | X   | X      | X    | Reported |           | 5.04    | 5.040      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001016  | 0945008-04   | pH_LAB  | X   | X      | X    | Reported |           | 5.02    | 5.020      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001015  | 0949010-01   | pH_LAB  | X   | X      | X    | Reported |           | 5.3     | 5.300      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001014  | 0949010-02   | pH_LAB  | X   | X      | X    | Reported |           | 5.03    | 5.030      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001013  | 0949010-03   | pH_LAB  | X   | X      | X    | Reported |           | 4.58    | 4.580      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001012  | 0949010-04   | pH_LAB  | X   | X      | X    | Reported |           | 5.64    | 5.640      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001011  | 0950010-01   | pH_LAB  | X   | X      | X    | Reported |           | 4.94    | 4.940      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001010  | 0950010-02   | pH_LAB  | X   | X      | X    | Reported |           | 5.79    | 5.790      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001009  | 0950010-03   | pH_LAB  | X   | X      | X    | Reported |           | 4.59    | 4.590      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001008  | 0950010-04   | pH_LAB  | X   | X      | X    | Reported |           | 6.96    | 6.960      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001007  | 0950010-05   | pH_LAB  | X   | X      | X    | Reported |           | 7.07    | 7.070      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001006  | 0950010-06   | pH_LAB  | X   | X      | X    | Reported |           | 5.39    | 5.390      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001005  | 0950010-07   | pH_LAB  | X   | X      | X    | Reported |           | 6.79    | 6.790      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001004  | 0950010-08   | pH_LAB  | X   | X      | X    | Reported |           | 6.78    | 6.780      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001003  | 0950010-09   | pH_LAB  | X   | X      | X    | Reported |           | 4.94    | 4.940      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001002  | 0950010-10   | pH_LAB  | X   | X      | X    | Reported |           | 6.92    | 6.920      | PR   | 0.000 |     | 0.000 | std units | 1   |              |
| L001001  | L001025-BLK1 | pH_LAB  | X   | X      | X    | QC       |           | 0       | ND [0.000] | NA   | 0.000 |     | 0.000 | std units | 1   |              |
| L912044  | L001025-CCV1 | pH_LAB  | X   | X      | X    | QC       |           | 4.96    | 4.960      | NA   |       |     |       | std units | 1   | 99 (95-105)  |
| L912043  | L001025-CCV2 | pH_LAB  | X   | X      | X    | QC       |           | 5.01    | 5.010      | NA   |       |     |       | std units | 1   | 100 (95-105) |
| L912042  | L001025-CCV3 | pH_LAB  | X   | X      | X    | QC       |           | 4.99    | 4.990      | NA   |       |     |       | std units | 1   | 100 (95-105) |
| L912041  | L001025-CCV4 | pH_LAB  | X   | X      | X    | QC       |           | 4.98    | 4.980      | NA   |       |     |       | std units | 1   | 100 (95-105) |
| L912040  | L001025-DJF1 | pH_LAB  | X   | X      | X    | QC       |           | 5.57    | 5.570      | NA   | 0.000 |     | 0.000 | std units | 1   |              |
| L912039  | L001025-DJF2 | pH_LAB  | X   | X      | X    | QC       |           | 6.93    | 6.930      | NA   | 0.000 |     | 0.000 | std units | 1   |              |
| L912038  | L001025-SRM1 | pH_LAB  | X   | X      | X    | QC       |           | 3.75    | 3.750      | NA   |       |     |       | std units | 1   | 99 (99-101)  |
| L912037  | L001025-SRM2 | pH_LAB  | X   | X      | X    | QC       |           | 3.84    | 3.840      | NA   |       |     |       | std units | 1   | 101 (99-101) |
| L912036  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912035  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912034  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912033  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912032  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912031  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912030  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912029  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912028  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912027  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912026  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912025  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912024  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912023  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |
| L912022  |              |         |     |        |      |          |           |         |            |      |       |     |       |           |     |              |

## 6.8 Quality Control

- 6.8.1 Method blanks (BLK), consisting of DI water, must be analyzed at the beginning of each day's analysis unless specified differently by a project.
- 6.8.2 Reference samples (SRM) must be analyzed at the beginning and at the end of each run. Acceptance criteria are  $\pm 0.05$  pH units of the certified value.

- 6.8.3 Control standards (CCV) must be analyzed at the beginning and end of each run, as well as at a frequency of 10 percent. Acceptance criteria are  $\pm 0.05$  pH units of the known value.
- 6.8.4 Duplicate samples (DUP) must be performed at a frequency of 20 percent. Reading should be  $\pm 0.05$  pH units of the original value.
- 6.8.5 If data are uploaded to the Clean Air Status and Trends Network database, the BLK, SRM, and DUP prefixes will be changed to MB, RF, and RP respectively upon ingestion.

## **6.9 Calculations**

All calculations are performed internally by the pH meter including the determination of the calibration curve from the potentiometric values (in millivolts – mV) obtained during the two-step calibration procedure outlined in section 6.3. Sample results are displayed directly as pH values by the meter. No external calculations are performed.

## **6.10 Corrective actions**

Attachment A shows the corrective actions taken when the QC samples are not within acceptance criteria.

## **6.11 Data review**

The checklists used by the analyst, peer reviewer, and supervisor in reviewing the analytical data are shown in Attachments B and C.

## 7.0 REFERENCES

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan*, Revision 8.0. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

Clesceri, L.S., Greenberg, A.E., Trussel, R.R. (eds) 1989. *Standard Methods for the Examination of Water and Wastewater*, 17th edition. APHA-AWWA-WPCF, Washington D.C.

*Thermo Scientific. 2008. Orion Star Meter Users Guide. DN 257250-001 Revision B.*

*Thermo Scientific Orion Electrode User's Guide.*

U.S. Environmental Protection Agency (EPA). 1995. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/R-96/027, November.

U.S. Environmental Protection Agency (EPA). 1983. *Methods for Chemical Analysis of Water and Wastes* (EPA 150.1), EPA 600/4-79-020, revised March.

## **8.0 ATTACHMENTS**

Attachment A – Corrective Actions

Attachment B – Batch Folder Checklist

Attachment C – Batch Peer Review Checklist

**ATTACHMENT A**  
**Corrective Actions**

| <b>Quality Control</b>   | <b>Acceptance Criteria</b>             | <b>Corrective Action</b>   |
|--------------------------|--|--|
| pH 10 buffer             | $\pm 0.05$ (9.95-10.05)                | Repeat the analysis with a new pH 10 buffer. If the new buffer is not within the acceptance criteria, verify the buffer solutions are within $\pm 1^{\circ}\text{C}$ of room temperature and recalibrate. If still outside acceptance criteria, change electrode filling solution. Additional failure will require cleaning (see electrode User's Guide) or replacing the electrode. |
| Reference standard (SRM) | $\pm 0.05$ of the certified true value | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples, or document why data are acceptable.  |
| Control standard (CCV)   | $\pm 0.05$ of the certified true value | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV, or document why data are acceptable.  |
| Sample Replicate (DUP)   | $\pm 0.05$ of the certified true value | Determine the cause of the problem. Reanalyze the sample, if necessary, or document why data are acceptable.   |
| Source: AMEC             |  |  |

## ATTACHMENT B Batch Folder Checklist

|               |                                 |
|---------------|---------------------------------|
| Batch #       | <u>L001025</u>                  |
| Initial or NA |                                 |
| <u>KWB</u>    | ELEMENT batch printout          |
| <u>KWB</u>    | Method reference                |
| <u>KWB</u>    | Instrument logbook copies       |
| <u>KWB</u>    | Reagent/Standard prep logsheets |
| <u>NA</u>     | Original raw data               |

|                          |                 |
|--------------------------|-----------------|
| <u>K. Barry</u>          | <u>1/20/10</u>  |
| Analyst                  | Date            |
| <u>Paul C. Lucor</u>     | <u>01-20-10</u> |
| Batch Folder Reviewed by | Date            |

Notes: ✓

|                     |                |
|---------------------|----------------|
| <u>[Signature]</u>  | <u>1/20/10</u> |
| ELEMENT Reviewed by | Date           |

## ATTACHMENT C

### Batch Peer Review Checklist



#### Batch Folder and ELEMENT Batch Review Checklist

Batch L001025

**Section 1.**

This section is to be completed by the analyst before submitting the batch and counter-signed during peer review. Peer reviewer also checks each statement for accuracy (indicated by a check preceding the item). Any discrepancies must be corrected before the batch can be accepted. Any "No" response must have a comment.

|   | Yes | No  | Comments                                       |
|---|-----|-----|--|
| ✓ 1. All samples uploaded correctly.              | ✓   | --- | ---  |
| ✓ 2. All duplicate sources entered correctly.     | ✓   | --- | ---  |
| 3. All QC results within established criteria:    |     |     |  |
| ✓ MB  | ✓   | --- | ---  |
| ✓ BS  | NA  | --- | ---  |
| ✓ CCV   | ✓   | --- | ---  |
| ✓ SRM   | ✓   | --- | ---  |
| ✓ DUP   | ✓   | --- | ---  |
| ✓ 4. All curve coefficients $\geq 0.995$ .        | NA  | --- | ---  |
| ✓ 5. All responses within calibration range.      | ✓   | --- | ---  |
| ✓ 6. All dilution factors entered correctly.      | ✓   | --- | ---  |
| ✓ 7. Other electronic flags in the ELEMENT batch? | ✓   | --- | If Yes, describe in Additional Comments below. |

Analyst: H. Barry

Date: 1/20/10

Reviewed By: Samuel C. Lucas

Date: 01-20-10

**Section 2.**

This section is to be completed by the reviewer. Initial each item. All items must be found to be true and be initialed before the batch can be accepted.

1. Instrument run log matches ELEMENT batch. (Exceptions allowed with sufficient documentation) DCL
2. All appropriate documentation present. (Includes, but is not limited to, standard prep logs, COA, code sheets, method print-outs, schedule print-outs, and run log copies.) DCL
3. Raw data (chromatograms, spectra intensities, etc.) is acceptable. (Additional documentation may be required.) DCL

Reviewed By: Samuel C. Lucas

Date: 01-20-10

Additional Comments:

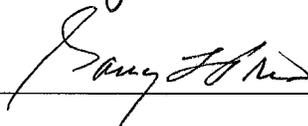
All samples : Sampled to analysis dates.

OK  
 JCB 1/20/10

**TITLE: DETERMINATION OF SPECIFIC CONDUCTANCE FOR WET DEPOSITION SAMPLES (EPA METHOD 120.1)**

Effective Date: April 6<sup>th</sup> 2015

Prepared by: Kathy W. Barry  
 Laboratory Chemist 

Reviewed by: Garry L. Price  
 Laboratory Operations Manager 

Reviewed by: Marcus O. Stewart  
 QA Manager 

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |        |       |            |
|---------------|--------|-------|------------|
| Reviewed by:  | Title: | Date: | Signature: |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |

## **DETERMINATION OF SPECIFIC CONDUCTANCE FOR WET DEPOSITION SAMPLES (EPA METHOD 120.1)**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the determination of the conductivity of water samples.

### **2.0 SCOPE**

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes, and acid rain (atmospheric wet deposition).

### **3.0 SUMMARY OF METHOD**

The specific conductance of a sample is measured by the use of a self-contained conductivity meter, Wheatstone bridge-type, or equivalent.

Samples are preferably analyzed at 25 degrees Celsius (°C). Automatic Temperature Compensation (ATC) is used by the instrument to make temperature corrections to report the results at 25 °C.

#### **3.1 Method Interferences**

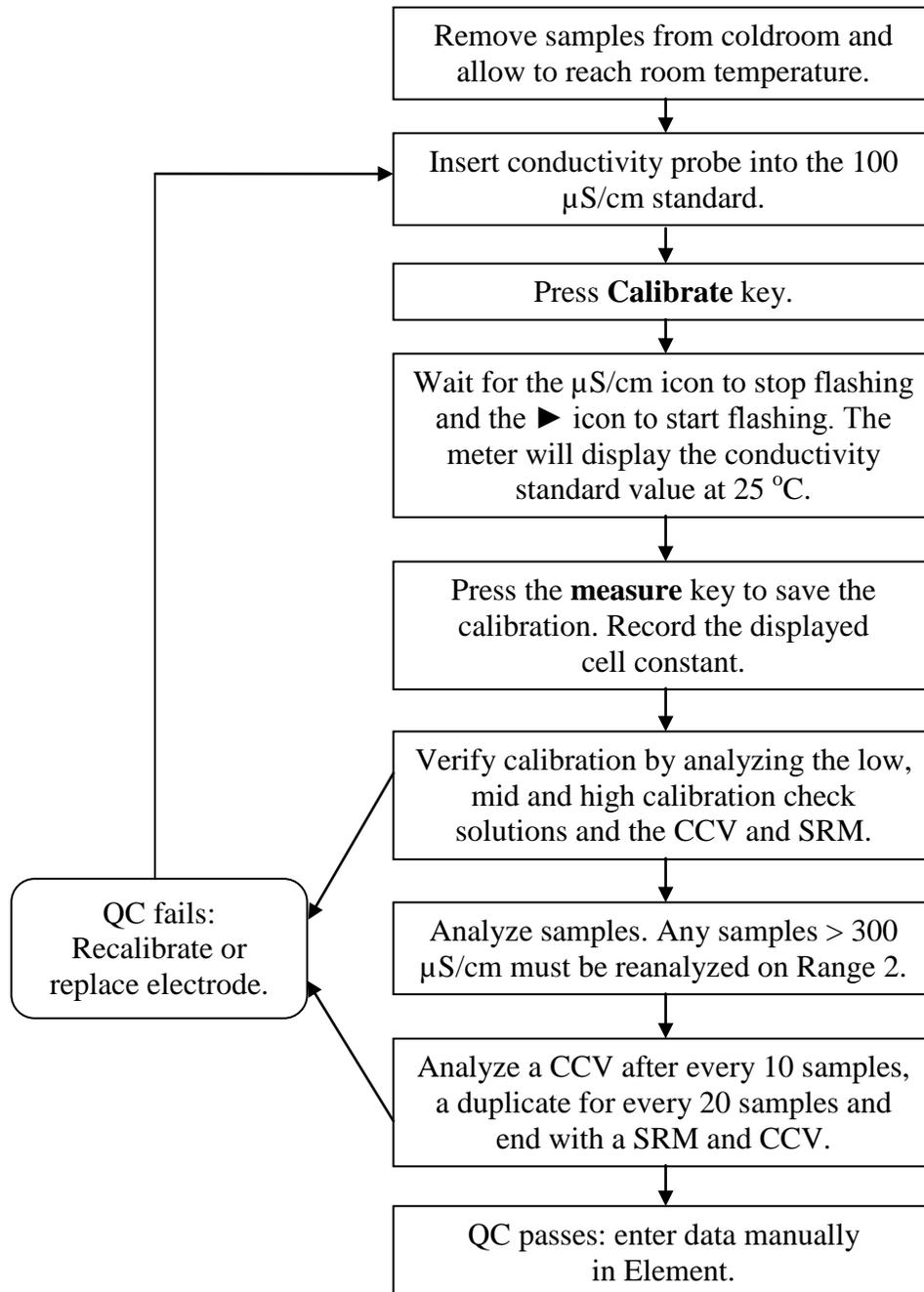
Electrolytic conductivity increases with temperature approximately 2.1 percent per °C. Significant differences exist between the temperature coefficients of conductivity when comparing different ions. Therefore, temperature variations and corrections represent the largest source of potential error. Dirty conductivity cells can cause erroneous readings.

Dissolved carbon dioxide (CO<sub>2</sub>) and ammonia (NH<sub>3</sub>) from the atmosphere increase the conductivity of samples. Protect all solutions and samples from the atmosphere as much as possible.

#### **3.2 Deviations from the Method**

A general overview of the steps described in this SOP is depicted Figure 1. Deviations from the analytical method described in this SOP are not permitted.

**Figure 1** Overview – Laboratory Operations Procedures for Specific Conductance of Range 1 Samples (0-300  $\mu\text{S}/\text{cm}$ )



## 4.0 MATERIALS

### 4.1 Apparatus

- Conductivity meter, with a range of 0 microSeimens per centimeter ( $\mu\text{S}/\text{cm}$ ) to 200 milliSeimens per centimeter ( $\text{mS}/\text{cm}$ ) with automatic temperature correction capabilities. The Orion 3 Star meter (see Figure 2) is currently in use in the laboratory, and instructions are based on the corresponding instrument manual.

**Figure 2** Conductivity Meter



- Conductivity cell probe: Orion #013016MD – two-electrode steel conductivity probe, with a nominal cell constant:  $K=0.1 \text{ cm}^{-1}$ . This probe is suitable for sample conductivity levels from  $0.01 \mu\text{S}/\text{cm}$  to  $300 \mu\text{S}/\text{cm}$  (Range 1) and is the current preferred probe. Follow manufacturer's directions for storage, cleaning, and maintenance of probe.
- Orion #013005D – four-electrode ceramic and graphite conductivity probe, with a nominal cell constant:  $K=0.475 \text{ cm}^{-1}$ . It exhibits stable readings above  $20 \mu\text{S}/\text{cm}$  and is used for Range 2. Follow manufacturer's directions for storage, cleaning, and maintenance of probe.
- Class A glass pipettes – 2, 3, 5, and 7 milliliter (mL)
- Class A volumetric flasks – 200, 250, 500, and 1000 mL
- Analytical balance sensitive to 0.0001 gram (g)
- Kimwipes
- Nalgene bottles – 30 mL, cleaned per SOP GLO3180-001 or other appropriately sized vessels for use when sample volume is limited
- Wash bottle filled with reagent water for cleaning the probe
- Thermometer for ambient room temperature readings
- Digital scanning thermometer for surface temperature readings of the solutions

## 4.2 Reagents

- Reagent Water: Deionized (DI) water of resistivity of 15 mega ohms ( $M\Omega$ ) or greater, derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 micrometers.
- Laboratory-prepared Calibration Standards: If laboratory-prepared calibration standards are used, a commercially available control standard should be used for verification. Substituting a laboratory-prepared standard for a commercially unavailable conductivity level is acceptable. See Attachment A for conductivity target calculation procedure. Class A volumetric flasks and pipettes must be used for preparation of all solutions.
- Potassium Chloride (KCl) Stock Solutions
  - KCl Stock Solution I (0.050 Molar [M]) KCl: Dissolve 0.932 (g) of pre-dried (minimum of two hours at 105 °C) KCl in 250 mL of DI water. Conductivity of this solution should be 6668  $\mu\text{S}/\text{cm}$ . Expiration is one year from preparation date.
  - KCl Stock Solution II (0.500 M KCl): Dissolve 9.32 (g) of pre-dried (minimum of two hours at 105 °C) KCl in 250 mL of DI water. Conductivity of this solution should be 58.64  $\text{mS}/\text{cm}$ . Expiration is one year from preparation date.
  - KCl Stock Solution III (0.100 M KCl): Dissolve 1.864 (g) of pre-dried (minimum of two hours at 105 °C) KCl in 250 mL of DI water. Conductivity of this solution should be 12.90  $\text{mS}/\text{cm}$ . Expiration is one year from preparation date.
- Calibration Standards, Range 1 (0-300  $\mu\text{S}/\text{cm}$ ):
  - Calibration Standard I – (0.500 millimolar mM KCl): Dilute 5.0 mL KCl Stock Solution I (0.050 M KCl) to 500 mL with DI water. Conductivity of the solution is 73.90  $\mu\text{S}/\text{cm}$ . This standard may also be used as a Mid-level Calibration Check Standard for this range. Expiration is six months from preparation date.
  - High Calibration Check Standard I – (2.0 mM KCl): Dilute 2.0 mL Stock Solution II (0.500 M KCl) to 500 mL with DI water. Conductivity of the solution should be 289.7  $\mu\text{S}/\text{cm}$ . This solution is also used as the Mid-level Calibration Check Standard for Range 2. Expiration is six months from preparation date.
- Calibration Standards, Range 2 (> 100  $\mu\text{S}/\text{cm}$ ):
  - Calibration Standard II – Range 2 (5.00 mM KCl): Dilute 2.0 mL KCl Stock Solution II (0.500 M KCl) to 200 mL with DI water. Conductivity of the solution should be 717.8  $\mu\text{S}/\text{cm}$ . Expiration is six months from preparation date.
  - Low Calibration Check Standard – Range 2 (1.5 mM KCl): Dilute 3.0 mL of Stock Solution III (0.100 M KCl) to 200 mL with DI water. Conductivity of the solution should be 218.4  $\mu\text{S}/\text{cm}$ . Expiration is six months from preparation date.
  - High Calibration Check Standard II– Range 2 (14.0 mM KCl): Dilute 7.0 mL Stock Solution II (0.500 M KCl) to 250 mL with DI water. Conductivity of the solution should be 1955  $\mu\text{S}/\text{cm}$ . Expiration is six months from preparation date.

- **Control Standard Solutions:**
  - Control Standard Solution II – Range 2 (7.5 mM KCl): Dilute 3.0 mL of Stock Solution II (0.500 M KCl) to 200 mL DI water. Conductivity of the solution should be 1065  $\mu\text{S}/\text{cm}$ . Expiration is six months from preparation date.
- Commercial Solutions - commercial National Institute of Standards and Technology (NIST) traceable calibration, reference and calibration standards from different manufacturers are all acceptable for usage. A Certificate of Analysis and expiration date must be supplied with all commercial standards and reference solutions (see Figure 3).
  - Commercial Calibration Standard I – Range 1: Thermo Scientific 100  $\mu\text{S}/\text{cm}$  standard conductivity solution, or equivalent.
  - Commercial Calibration Standard II – Range 2: Thermo Scientific 1413  $\mu\text{S}/\text{cm}$  standard conductivity solution, or equivalent.
  - Commercial Low Calibration Check Standard I – Range 1: Commercial NIST traceable standard conductivity solution of approximately 10  $\mu\text{S}/\text{cm}$ .
  - Commercial Low Calibration Check Standard II – Range 1: Commercial NIST traceable standard conductivity solution of approximately 5  $\mu\text{S}/\text{cm}$ .
  - Commercial Reference Solution I – Range 1: High-Purity Standards Simulated Rainwater 1, or equivalent. This standard should be in the range of 20-30  $\mu\text{S}/\text{cm}$ .
  - Commercial Reference Solution II – Range 2: Commercial mid-range NIST traceable standard conductivity solution in the range of 200-500  $\mu\text{S}/\text{cm}$ .
  - Commercial Control Solution – Range 1: Commercial NIST traceable standard solution of approximately 15  $\mu\text{S}/\text{cm}$  concentration.

Figure 3 Certificate of Analysis



DF17001

## Certificate of Analysis

**Product Description:**

Name: Simulated Rainwater Level I  
Part number: SR-1  
Lot number: 0935107  
Material Purity: 99.99-99.999%  
Matrix: H<sub>2</sub>O

*Rec'd 6/17/10 RWB*  
*Exp 6/15/11*

**Certified Values:**

| Component | Certified Value (mg/L) | NIST SRM ID | NIST SRM Lot # |
|-----------|------------------------|-------------|----------------|
| Chloride  | 0.216 ± 0.003          | 3182        | 060925         |
| Fluoride  | 0.053 ± 0.003          | 3183        | 050721         |
| Nitrate   | 0.502 ± 0.018          | 3185        | 050517         |
| Sulfate   | 2.56 ± 0.03            | 3181        | 080603         |
| Sodium    | 0.20 ± 0.01            | 3152a       | 010728         |
| Potassium | 0.049 ± 0.002          | 3141a       | 051220         |
| Calcium   | 0.011 ± 0.001          | 3109a       | 050825         |
| Magnesium | 0.020 ± 0.001          | 3131a       | 050302         |

| Parameter            | Certified Value           | NIST SRM ID | NIST SRM Lot # |
|----------------------|---------------------------|-------------|----------------|
| pH                   | 4.28 ± 0.10, at 20.1°C    | *           | *              |
| Specific Conductance | 23.6 ± 3 µS/cm, at 25.0°C | 3191        | 050403         |

The Certified values are based on gravimetric and volumetric preparations and verified against SRM 3100 Series developed by the National Institute of Standards and Technology (NIST), via inductively coupled plasma optical emission spectrometry (ICP-OES) and Ion Chromatography using an internally developed laboratory method. The uncertainty in the certified value is calculated for a 95% confidence interval.

\* Refer to Traceability Information, Section C.

**Reference Values:**

| Component | Value (mg/L) | NIST SRM ID | NIST SRM Lot # |
|-----------|--------------|-------------|----------------|
| Ammonium  | 0.10         | *           | *              |

**Preparation Information:**

The highest purity source materials were purchased from qualified vendors per ISO 9001:2008 guidelines and assayed by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography (IC) for conformity prior to use. This standard was prepared using methods developed at the National Institute of Standards and Technology for the preparation of SRM Spectrometric Standard Solutions. The matrix is 18 megaohm deionized water.

Lot No.: 0935107  
Rev. No.: 1.0.0  
Page 1 of 2

## 5.0 SAFETY

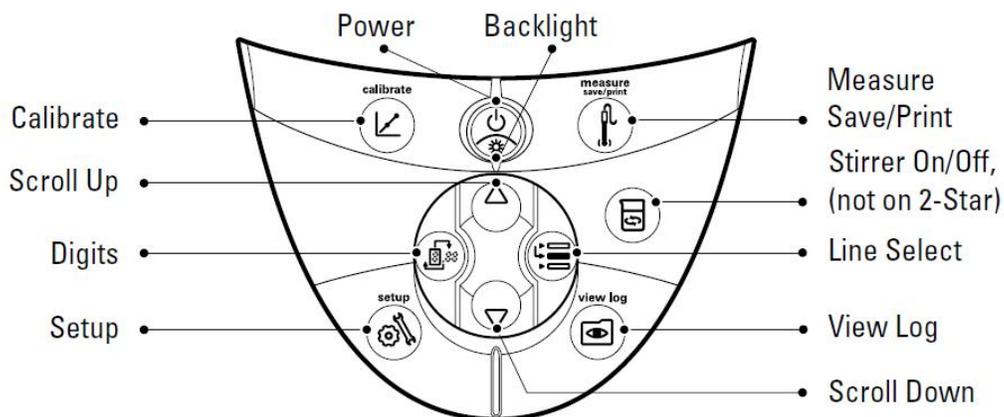
The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the

AMEC laboratory or at the Web sites <http://www.msdsearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

## 6.0 PROCEDURE

### 6.1 Calibration

**Figure 4** Diagram of Orion 3-Star Conductivity Bench-top Meter Keypad

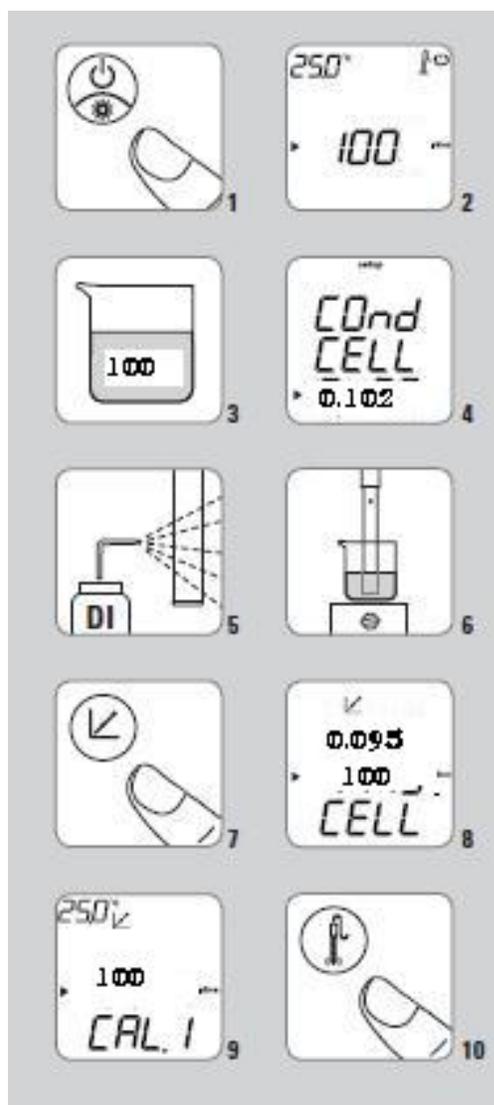


| Key | Description   | Key | Description   |
|-----|---|-----|---|
|     | Turns the meter on, if the meter is off.<br>Toggles the backlight on and off, if the meter is on (3-Star, 4-Star and 5-Star meters only).<br>If the meter is on, hold down the key for about three seconds to turn off the meter.   |     | Changes the measurement units of the selected line in the measurement mode.   |
|     | Scrolls the arrow icon on left of screen among the three display lines, so the selected line can be edited or calibrated.   |     | Changes the value on the selected line in the setup, methods and log view modes.<br>Edits the value of the flashing digit for setup, password entry and calibration modes.  |
|     | Starts the calibration for the selected line in the measurement mode.<br>If the arrow icon points to the top line and the displayed units are pH, pressing the key will start a pH calibration.<br>Each time the key is pressed in the calibration mode, the meter will accept the calibration point and move to the next point until the maximum number of calibration points are reached. |     | Selects the next digit to edit and moves the decimal point for setup, password entry and calibration modes.   |
|     | Enters the setup menu, starting with selected line in the measurement mode.<br>If the arrow icon points to the top line and the displayed units are ISE, pressing the key will enter the ISE setup screen.  |     | Prints and logs a measurement in the continuous or timed measurement modes.<br>Prints, logs and freezes the display when the reading becomes stable in the AUTO-READ measurement mode.<br>Exits the setup menu and returns to measurement mode.<br>Accepts the calibration and returns to measurement mode. |
|     |   |     | Enters the log view and download menu.  |
|     |   |     | Turns the stirrer on and off.   |

**Note:** This calibration method is for Range 1- a one-point calibration utilizing the AutoBuffer recognition feature of the Orion 3-Star Bench-top conductivity meter (see Figure 4 for a diagram of the keypad and Figure 5 for an overview of the calibration steps). Other calibration methods are listed in the manual for this meter and may be used in cases as approved by the Laboratory Manager and/or QA Manager.

**Figure 5** Conductivity Calibration Overview

- 6.1.1 Turn the on the meter (see Fig 5-1). The display will show the room temperature (see Fig 5-2).
- 6.1.2 Select the Thermo Scientific Orion conductivity standard for Range 1: 100  $\mu\text{S}/\text{cm}$  (see Fig 5-3).
- 6.1.3 In the **Setup** Mode enter the nominal cell constant value for the conductivity probe (0.102) (see Fig 5-4).
- 6.1.4 Rinse the conductivity probe with DI and blot dry with a Kimwipe<sup>®</sup> (see Fig 5-5) and insert into the 100  $\mu\text{S}/\text{cm}$  standard (see Fig 5-6). Probe should be inserted in the liquid far enough to cover the ports (see Figure 6).
- 6.1.5 Press the **Calibrate** key (see Fig 5-7). The meter will show the manual calibration display for about five seconds. Do not press any keys. The meter will then proceed to the autocalibration display (see Fig 5-8).
- 6.1.6 Wait for the  $\mu\text{S}/\text{cm}$  icon to stop flashing and the **►** icon to start flashing. The meter will display the conductivity standard value at 25  $^{\circ}\text{C}$  (see Fig 5-9). The displayed value should be within  $\pm 5\%$  of the certified value. If the displayed value is not within acceptable limits, use a fresh standard and recalibrate.
- 6.1.7 Press the **Measure** key (see Fig 5-10) to save and end the calibration. The calculated cell constant will be displayed and then the meter will proceed to the measurement mode. Record the cell constant.



**Figure 6** Immersion Depth for Probe



## 6.2 Verify Calibration

- 6.2.1 The meter is typically in AUTO-READ mode. Press the **Measure** key to take a measurement. Once the reading stabilizes, the **AR** icon will stop flashing and the display will freeze. Press the **Measure** key again to take a new measurement.
- 6.2.2 Rinse the probe with DI water and blot excess water from the outside surfaces with a Kimwipe prior to immersing the probe in the next sample. Store the probe in DI water between samples.
- 6.2.3 Check the calibration using the low, mid, and high calibration check solutions. The acceptance criterion is  $\pm 10$  percent of the certified or calculated value. If the calibration check standards do not fall within range, re-calibrate the meter. If the readings are within the acceptance range, record the values and temperature on the Conductivity Calibration logbook page (see Attachment B).
- 6.2.4 Analyze a control and reference sample. The values must be within  $\pm 15$  percent of the certified or calculated value or the meter must be recalibrated.
- 6.2.5 If re-calibrating the meter does not give conductivity readings within the acceptable criteria, clean the conductivity cell following the manufacturer's instructions.
- 6.2.6 If cleaning the conductivity cell does not give conductivity readings within the acceptable criteria, consult the Analytical Laboratory Manager. The conductivity cell may need to be replaced or replatinized.

### **6.3 Sample Analysis**

*Note: Acid rain samples should not be stirred. Instead, swirl the samples gently for a few seconds after the introduction of the probe.*

- 6.3.1 Allow the unpreserved samples to come to room temperature, prior to analyses.
- 6.3.2 Fill a vessel with the sample. The 30 mL Nalgene bottles require a minimum of 20 mL of sample to properly immerse the probe. If there is insufficient sample available, 7 mL scintillation vials may be used (the minimum sample volume for the vial is 3 mL). If the smaller vial is used a note should be made in the instrument logbook (see Attachment C).
- 6.3.3 Follow the analysis procedure detailed in Verify Calibration (section 6.2).
- 6.3.4 Record the results, temperature, and any pertinent comments in the appropriate place in the Conductivity Logbook.
- 6.3.5 If any samples are found to be outside of Range 1, recalibrate the meter using the alternate probe and Range 2 standards. Reanalyze the high sample. Include the data from any reanalysis and the QC with the initial analysis.
- 6.3.6 For overnight or long-term storage, the conductivity cell should be thoroughly rinsed and stored dry.

### **6.4 Data Analysis**

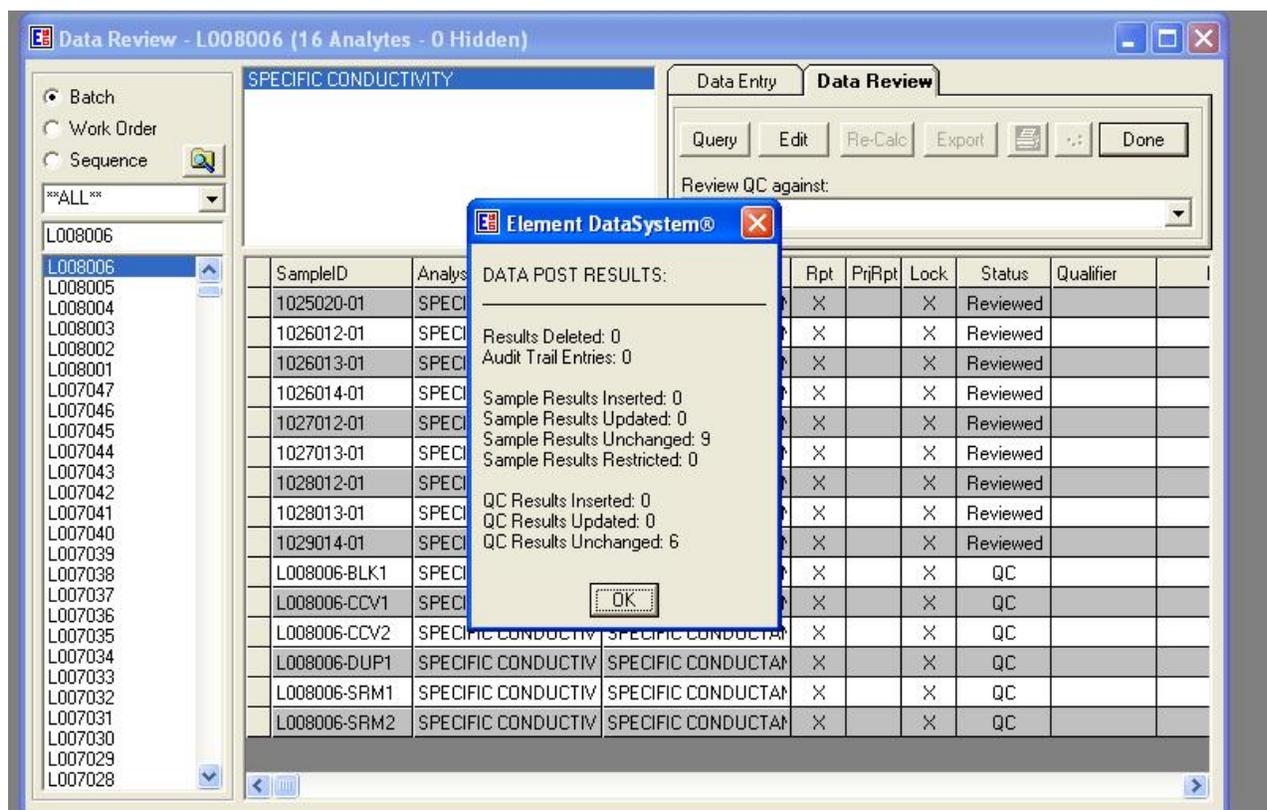
- 6.4.1 Enter the data in Element (see Figure 7) using the manual batch processing feature outlined in SOP GLO3180-35 Element Batch Preparation (section 3.3).
- 6.4.2 The analyst should note any analytical anomalies in a separate batch narrative.
- 6.4.3 Assemble the data batch folder, including a hard copy of the Element data batch, copies of all logbooks, certificates of analyses and processing methods, and any other necessary documentation.
- 6.4.4 Complete the batch folder checklists (Attachment E and F), and submit it for review and finalization.

### **6.5 Quality Control**

- 6.5.1 A method blank (BLK) must be analyzed at the beginning of each run. BLK consists of DI water. A method blank should also be analyzed with any samples reanalyzed at a different calibration range.
- 6.5.2 Replicate samples (DUP) must be analyzed at a frequency of 5 percent for every analytical batch unless specified differently by a project. Value must be within  $\pm 15$  percent.

- 6.5.3 Reference samples (SRM) are analyzed at the beginning and end of each run. Value must be within  $\pm 15$  percent of the certified value. Additional reference samples are analyzed with any samples reanalyzed at a different calibration range.
- 6.5.4 Control standards are continuing calibration verification (CCV) standards that are analyzed every 10 samples. Values must be within  $\pm 15$  percent of the certified value. Additional control standards are analyzed with any samples reanalyzed at a different calibration range.
- 6.5.5 If data are uploaded to the Clean Air Status and Trends Network database, the BLK, SRM, and DUP prefixes will be changed to MB, RF, and RP respectively upon ingestion.

**Figure 7** Element Manual Batch



## 6.6 Calculations

Calculations are performed internally by the conductivity meter from the calibration obtained during the autocalibration procedure outlined in section 6.1 and include temperature correction based on the stored temperature coefficient (2.1 percent). Sample results are displayed directly as specific conductance,  $\mu\text{S}/\text{cm}$  at 25 °C by the meter. No external calculations are performed.

## **6.7 Corrective actions**

Attachment D shows the corrective actions taken when the QC Samples are not within acceptance criteria.

## **6.8 Data review**

The checklists used by the analyst, peer reviewer, and supervisor in reviewing the analytical data are shown in Attachments E and F.

## 7.0 REFERENCES

AMEC E&I, Inc. (AMEC). 2011. Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0. Prepared for U.S. Environmental Protection Agency (EPA), Washington, D.C. Contract No. EP-W-09-028. Gainesville, FL.

Clesceri, L.S., Greenberg, A.E., Trussel, R.R. (eds). 1989. *Standard Methods for the Examination of Water and Wastewater*, 17<sup>th</sup> edition APHA-AWWA-WPCF, Washington DC.

Thermo Scientific. Orion Star Meter Users Guide. DN 257250-001 Revision B 10-08.

Thermo Scientific. 2007. Orion Electrode User's Guide. DN 25478.001 Revision A.

U.S. Environmental Protection Agency (EPA). 1995. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/R-96/027, November.

U.S. Environmental Protection Agency (EPA). 1983. *Methods for Chemical Analysis of Water and Wastes* (EPA 120.1), EPA 600/4-79-020, revised March 1983.

## **8.0 ATTACHMENTS**

Attachment A – Calculating Conductivity Targets

Attachment B – Conductivity Calibration Logbook page

Attachment C – Conductivity Logbook page

Attachment D – Corrective Actions

Attachment E – Batch Folder Checklist

Attachment F – Batch Peer Review Checklist

**ATTACHMENT A**  
**Calculating Conductivity Targets**  
**Conductivity of KCl Solutions at 25 °C**

| <b>8.1</b> | <b>Concentration</b><br><i>M</i> | <b>Equivalent Conductivity</b><br><i>mho/cm/equiv.</i> | <b>Conductivity</b><br><i>μS/cm</i> |
|------------|----------------------------------|--|-------------------------------------|
|            | 0                                | 149.85   |                                     |
|            | 0.0001                           | 149.43   | 14.94                               |
|            | 0.0005                           | 147.81   | 73.90                               |
|            | 0.001                            | 146.95   | 147.0                               |
|            | 0.005                            | 143.55   | 717.8                               |
|            | 0.01                             | 141.27   | 1413                                |
|            | 0.02                             | 138.34   | 2767                                |
|            | 0.05                             | 133.37   | 6668                                |
|            | 0.1                              | 128.96   | 12900                               |
|            | 0.2                              | 124.08   | 24820                               |
|            | 0.5                              | 117.27   | 58640                               |
|            | 1                                | 111.87   | 111900                              |

Standard Methods for the Examination of Water and Wastewater, 17<sup>th</sup> edition, 1989.

Edited by Clesceri, L.S., Greenberg, A.E., Trussel, R.R. APHA-AWWA-WPCF, Washington D.C.

p. 2-60

To calculate conductivity for a solution:

$$k = c\lambda 10^3$$

where k is measured conductivity in μS/cm  
λ is equivalent conductivity in mho/cm/equiv.  
and c is equivalent concentration (molar concentration for KCl)

Use closest equivalent conductivity for calculation.

## ATTACHMENT B

### Sample Conductivity Calibration Logbook Page

*Note: The Conductivity Calibration Logbook is a loose-leaf binder-type logbook. This sheet reflects only one combination of electrode and sample range. Please select the correct sheet for the electrode and range in use. Target values are for example only. Actual values will vary and will be reflected on the sheets used for the logbook.*

#### Conductivity Calibration

Range 1 (0 – 300 µmhos/cm)

Date 8/4/10  
 Analyst K. Barry

Single Point Cal:

Target 100 Temp. (°C) 22.9 cell constant 0.094 Response 100.1

Cal. Check:

|           | Target       | Response     | Temp. (°C)  |
|-----------|--------------|--------------|-------------|
| Low STD 1 | <u>10.28</u> | <u>10.41</u> | <u>23.1</u> |
| Low STD 2 | <u>5.00</u>  | <u>5.16</u>  | <u>23.1</u> |
| Mid STD   | <u>73.90</u> | <u>74.2</u>  | <u>23.0</u> |
| High STD  | <u>289.7</u> | <u>295.8</u> | <u>23.1</u> |

Low STD 1 VWR Lot: 101705436 Exp: 5/17/11 Target: 10.28

Low STD 2 Hamilton Lot: 1324464 Exp: 12/18/10 Target: 5.0

Mid STD 180739VFD Prep: 6/11/10 Exp: 12/18/10 Target: 73.90

By: KWB

High STD 180739VLF Prep: 6/11/10 Exp: 12/18/10 Target: 289.7

By: KWB



**ATTACHMENT D**  
**Summary of Corrective Action Procedures for Conductivity Measurements**

| <b>Quality Control</b>                         | <b>Acceptance Criteria</b>                           | <b>Corrective Action</b>  |
|--|--|---|
| Autocalibration standard                       | $\pm 5\%$ of the certified or calculated true value  | Repeat the calibration with a new standard; clean the probe; replatinize or replace the probe.  |
| Low, Mid, and High Calibration check standards | $\pm 10\%$ of the certified or calculated true value | Rerun check standard. If still out of control, recalibrate instrument clean the probe, replatinize, or replace the probe.                                 |
| Reference standard (SRM)                       | $\pm 15\%$ of the certified or calculated true value | Rerun reference. If still out of control, recalibrate instrument and reanalyze samples, or document why data are acceptable.                              |
| Control standard (CCV)                         | $\pm 15\%$ of the certified or calculated true value | Rerun standard. If still out of control, recalibrate instrument and reanalyze samples run since last acceptable CCV, or document why data are acceptable. |
| Sample Replicate (DUP)                         | $\pm 15$ of the original value.                      | Determine the cause of the problem. Reanalyze the sample, if necessary, or document why data are acceptable.  |

Source: AMEC

## ATTACHMENT E Batch Folder Checklist

|               |                                 |
|---------------|---------------------------------|
| Batch #       | <u>L008006</u>                  |
| Initial or NA |                                 |
| <u>KWB</u>    | ELEMENT batch printout          |
| <u>KWB</u>    | Method reference                |
| <u>KWB</u>    | Instrument logbook copies       |
| <u>KWB</u>    | Reagent/Standard prep logsheets |
| <u>NA</u>     | Original raw data               |

|                          |                 |
|--------------------------|-----------------|
| <u>K. Barry</u>          | <u>8/5/10</u>   |
| Analyst                  | Date            |
| <u>Amil C. Lucas</u>     | <u>08/05/10</u> |
| Batch Folder Reviewed by | Date            |

Notes: ✓

|                     |               |
|---------------------|---------------|
| <u>[Signature]</u>  | <u>8/5/10</u> |
| ELEMENT Reviewed by | Date          |

## ATTACHMENT F Batch Peer Review Checklist



### Batch Folder and ELEMENT Batch Review Checklist

Batch L008006

**Section 1.**

This section is to be completed by the analyst before submitting the batch and counter-signed during peer review. Peer reviewer also checks each statement for accuracy (indicated by a check preceding the item). Any discrepancies must be corrected before the batch can be accepted. Any "No" response must have a comment.

|   | Yes | No | Comments                                       |
|---|-----|----|--|
| ✓ 1. All samples uploaded correctly.              | ✓   |    |  |
| ✓ 2. All duplicate sources entered correctly.     | ✓   |    |  |
| 3. All QC results within established criteria:    |     |    |  |
| ✓ MB  | ✓   |    |  |
| ✓ BS  | NA  |    |  |
| ✓ CCV   |     |    |  |
| ✓ SRM   | ✓   |    |  |
| ✓ DUP   | ✓   |    |  |
| ✓ 4. All curve coefficients $\geq 0.995$ .        | NA  |    |  |
| ✓ 5. All responses within calibration range.      | ✓   |    |  |
| ✓ 6. All dilution factors entered correctly.      | ✓   |    |  |
| ✓ 7. Other electronic flags in the ELEMENT batch? |     | ✓  | If Yes, describe in Additional Comments below. |

Analyst: K Barry

Date: 8/5/10

Reviewed By: Samuel C. Lucas

Date: 08-05-10

**Section 2.**

This section is to be completed by the reviewer. Initial each item. All items must be found to be true and be initialed before the batch can be accepted.

- Instrument run log matches ELEMENT batch. (Exceptions allowed with sufficient documentation) DCL
- All appropriate documentation present. (Includes, but is not limited to, standard prep logs, COA, code sheets, method print-outs, schedule print-outs, and run log copies.) DCL
- Raw data (chromatograms, spectra intensities, etc.) is acceptable. (Additional documentation may be required.) DCL

Reviewed By: Samuel C. Lucas

Date: 08-05-10

Additional Comments:

**TITLE: SAMPLE CONTAINER PREPARATION FOR DRY AND WET DEPOSITION**

Effective  
Date: 11-10-11

Prepared by: Kathy W. Barry Kathy W Barry  
Laboratory Chemist

Reviewed by: Garry L. Price Garry L Price  
Laboratory Operations Manager

Reviewed by: Marcus O. Stewart Marcus O. Stewart  
QA Manager

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Materials
- 4.0 Procedure
- 5.0 References

| Annual Review |        |          |                   |
|---------------|--------|----------|-------------------|
| Reviewed by:  | Title: | Date:    | Signature:        |
| MS            | QA Mgr | 10/26/12 | Marcus O. Stewart |
| MS            | QA Mgr | 10/31/14 | Marcus O. Stewart |
| MS            | QA Mgr | 10/16/15 | Marcus O. Stewart |
|               |        |          |                   |
|               |        |          |                   |

## **SAMPLE CONTAINER PREPARATION FOR DRY AND WET DEPOSITION**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for the cleaning of bottles, buckets, and bucket lids used to store dry and wet deposition samples.

### **2.0 SCOPE**

This SOP applies to all extraction bottles, wet deposition filtration bottles, cloud bottles, one liter (L) field sample bottles, and wet deposition buckets and bucket lids that are used to collect, store and extract Clean Air Status and Trends Network (CASTNET) samples.

### **3.0 MATERIALS**

Powder-free gloves.

Resealable plastic bags.

Bottles - high density polyethylene (HDPE), various sizes.

Wet deposition bottle labels.

Sponge.

Large plastic bags.

Plastic-backed absorbent paper.

Deionized (DI) water of resistivity of 15 M $\Omega$  or greater, derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 microns.

Large plastic tubs.

Aluminum foil.

Drying oven.

**Figure 1.** Sample Containers



#### **4.0 PROCEDURE**

*Note: Powder-free gloves are to be worn throughout all steps of this procedure.*

##### **4.1 4.1 Cleaning of bottles**

- 4.1.1 Line a large plastic tub with aluminum foil.
- 4.1.2 Use only new polyethylene bottles and discard any bottles that appear to have defects (cracks, bad threading, abraded interior surfaces).
- 4.1.3 Rinse each bottle and cap a minimum of three times with DI water.
- 4.1.4 Shake out excess water with each rinse. Once the 8-ounce filtration sample bottle and cap are shaken dry, they are ready to use for sample filtration.
- 4.1.5 Place the extraction bottles, cloud bottles and caps in the foil lined trays and place in the designated oven at 70 degrees Celsius (°C) for approximately 2 hours.
- 4.1.6 When the bottles/caps are dry, remove from oven.
- 4.1.7 Cover the bottles with foil and allow to cool.
- 4.1.8 Once the bottles are cool, attach caps to the bottles and store in the designated box.
- 4.1.9 For cloud bottles, attach one cloud bottle label (see Figure 2) to each bottle and store in the large plastic bags.

**Figure 2.** Cloud Bottle Label

|               |               |            |
|---------------|---------------|------------|
|               | <b>CLOUDS</b> |            |
| <b>SITE :</b> | <b>CLD303</b> |            |
|               | <b>ON</b>     | <b>OFF</b> |
| <b>DATE</b>   | _____         | _____      |
| <b>TIME</b>   | _____         | _____      |

**4.2 Cleaning of 1-L wet deposition field sample bottles.**

- 4.2.1 4.2.1 Prior to cleaning, remove any sample labels attached to the outside of the bottle. Any bottles that are showing sign of wear (cracks, bad threading, abraded interior surfaces) should be discarded.
- 4.2.2 4.2.2 Line the counter top with the plastic-backed absorbent paper or line the bottom of the large plastic tub.
- 4.2.3 Rinse each bottle and cap a minimum of three times with DI water.
- 4.2.4 Shake out excess water each time.
- 4.2.5 Place bottle/cap upside down but slightly tilted to dry on the absorbent paper.
- 4.2.6 The next day, when the items are dry, place caps back on bottles.
- 4.2.7 Attach one wet deposition bottle label (see Figure 3) to each bottle.
- 4.2.8 Store cleaned field bottles in resealable plastic bags in designated area.

**Figure 3.** Wet Deposition Bottle Label

|  |       |                       |
|--|-------|-----------------------|
| <b>Castnet Wet Deposition Bottle Label</b> |       |                       |
| <b>SITE #</b>                              | _____ |                       |
| <b>ON DATE</b>                             | _____ | <b>OFF DATE</b> _____ |

### 4.3 **Cleaning of wet deposition buckets and bucket lids.**

- 4.3.1 Line the drying area with the plastic-backed absorbent paper.
- 4.3.2 Rinse the sponge thoroughly with DI water. Wash the bucket/lid very well both inside and out using the sponge. Rinse the inside of the bucket/lid two or three times by swirling DI water.
- 4.3.3 Hit the bucket on the absorbent paper to knock off the excess water and place upside down to dry. The lids only need a good shake to get the excess water off and are placed upside down to dry.
- 4.3.4 The next day, when the items are dry, wrap each bucket and lid in a new plastic bag (with the open end of the bucket facing downward), tie the bag closed, and store in the designated area. If the bucket is identified as a Dry Fall Bucket, attach one "Dry Fall Bucket" label (see Figure 4) to the bucket prior to wrapping in a plastic bag.

**Figure 4.** Dry Fall Bucket Label

|   |
|---|
| <b>SITE</b> _____ <b>DRY FALL BUCKET</b>    |
| <b>ON DATE</b> _____ <b>ON TIME</b> _____   |
| <b>OFF DATE</b> _____ <b>OFF TIME</b> _____ |

## 5.0 REFERENCES

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

**TITLE: WASHING OF FILTER PACK EQUIPMENT AND DENUDERS**

Effective  
Date: 11-4-11

Prepared by: Kathy W. Barry Kathy W Barry  
Laboratory Chemist

Reviewed by: Garry L. Price Garry Price  
Laboratory Operations Manager

Reviewed by: Marcus O. Stewart Marcus O. Stewart  
QA Manager

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Materials
- 4.0 Safety
- 5.0 Procedure
- 6.0 References
- 7.0 Attachments

| Annual Review |        |          |                   |
|---------------|--------|----------|-------------------|
| Reviewed by:  | Title: | Date:    | Signature:        |
| MS            | QA Mgr | 10/26/11 | Marcus O. Stewart |
| MS            | QA Mgr | 10/31/11 | Marcus O. Stewart |
| MS            | QA Mgr | 10/16/11 | Marcus O. Stewart |
|               |        |          |                   |
|               |        |          |                   |

## **WASHING OF FILTER PACK EQUIPMENT AND DENUDEERS**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for the cleaning of filter pack equipment and denuders to minimize any contamination.

### **2.0 SCOPE**

This SOP applies to all types of filter packs and denuders (Savillex, National Oceanic and Atmospheric Administration, and University Research Glassware-URG) used for Clean Air Status and Trends Network (CASTNET) and other projects.

### **3.0 MATERIALS**

Powder-free gloves

Various sizes of plastic tubs

Brushes dedicated to this procedure

Resealable plastic bags

Resealable plastic containers

Squeeze bottle.

Aluminum foil

Drying oven

Deionized (DI) water of resistivity of 15 mega ohms (M $\Omega$ ) or greater, derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 micrometers.

Methanol (MeOH), ACS reagent grade or better, CAS No. 67-56-1.

### **4.0 SAFETY**

The analyst must be aware of the hazards associated with the chemicals used in this method.

Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

## 5.0 PROCEDURE

*Note: Powder-free gloves are to be worn throughout all steps of this procedure. These procedures are performed in the washroom where all required components are available.*

### 5.1 Filter Pack Equipment

- 5.1.1 Take the filter pack apart. Any parts which appear worn, damaged or broken are to be replaced.
- 5.1.2 Place the filter support rings, grids, end caps and forceps in separate precleaned tubs filled with DI water. Agitate the items by hand for a few minutes and then allow to soak as needed.
- 5.1.3 Line large plastic tubs with aluminum foil as necessary.
- 5.1.4 Wash the filter collar and bottom support under a continuous flow of DI water. Scrub these parts clean using brushes. Make sure all grooves and places where particulate matter may collect are thoroughly cleaned.
- 5.1.5 Rinse the filter support rings, grids, end caps and forceps a minimum of three times with DI water. Shake as much excess water from these parts as possible before placing them in a foil-lined tub (see Figure 1).

**Figure 1** Foil lined tubs with parts for drying.



- 5.1.6 Place the tubs in the project drying oven at 70 degrees Celsius (°C) for approximately two hours or until dry.
- 5.1.7 Once dry, remove the tubs and immediately cover them with aluminum foil. Filter packs can be stored this way for up to three days.
- 5.1.8 Store the forceps in resealable plastic bags until used.
- 5.1.9 For long-term storage, the filter packs or various parts are placed in resealable plastic bags and then stored in large resealable plastic containers.

## 5.2 Denuders

- 5.2.1 Remove end caps from the denuder
- 5.2.2 Rinse each cap with DI water, filling, swishing, and dumping three times.
- 5.2.3 Using a properly labeled squeeze bottle, rinse the denuder with MeOH over the labeled denuder waste container.
- 5.2.4 Rinse the denuder, running DI water through the channels for approximately 10 seconds on each side. Rinse the outside threads of both ends.
- 5.2.5 Put the denuder into a large wash tub and fill with DI water.
- 5.2.6 Soak for approximately 24 hours.
- 5.2.7 Shake water out of the denuder and caps and place in an aluminum foil lined heat resistant tray (see Figure 2). Cover with aluminum foil and dry in the oven for about two hours at 60° C.

**Figure 2** Foil lined tubs with denuders for drying.



- 5.2.8 Once cool, cap the denuders and store in a foil lined and covered tub.

## **6.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0. Prepared for U.S. Environmental Protection Agency (EPA), Washington, D.C. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/R-96/027, November.

## **7.0 ATTACHMENTS**

This SOP does not contain attachments.

**TITLE: CENTRAL SAMPLE STORAGE ORGANIZATION FOR DRY AND WET DEPOSITION SAMPLES**

Effective  
Date: 11-4-11

Prepared by: Kathy W. Barry Kathy W Barry  
Laboratory Chemist

Reviewed by: Garry L. Price Garry L Price  
Laboratory Operations Manager

Reviewed by: Marcus O. Stewart Marcus O. Stewart  
QA Manager

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Materials
- 4.0 Safety
- 5.0 Procedure
- 6.0 References
- 7.0 Attachments

| Annual Review |        |          |                   |
|---------------|--------|----------|-------------------|
| Reviewed by:  | Title: | Date:    | Signature:        |
| MS            | QA Mgr | 10/26/12 | Marcus O. Stewart |
| MS            | QA Mgr | 10/31/14 | Marcus O. Stewart |
| MS            | QA Mgr | 10/16/15 | Marcus O. Stewart |
|               |        |          |                   |
|               |        |          |                   |

## **CENTRAL SAMPLE STORAGE ORGANIZATION FOR DRY AND WET DEPOSITION SAMPLES**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for the storage of samples at the Gainesville Laboratory.

### **2.0 SCOPE**

This SOP applies to all dry deposition filters, denuders, extracts, and wet deposition for Clean Air Status and Trends Network (CASTNET) and other project samples.

### **3.0 MATERIALS**

Samples, pre- and post-extraction  
Standard refrigerators  
Walk-in Coldrooms  
Traceable/ISO Calibrated Thermometers  
Temperature log books

### **4.0 SAFETY**

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

### **5.0 PROCEDURE**

#### **5.1 Short-term Storage Areas**

5.1.1 Extracted samples are stored overnight, prior to analysis, in refrigerators in the analytical area of the laboratory (see Figure 1). The boxes of extracted samples are labeled with field groups, extraction and analysis dates, and the appropriate initials. After analysis, the samples are transferred to the main coldroom (see Figure 2).

**Figure 1** Laboratory Refrigerator



**Figure 2** Laboratory Walk-in Coldroom



- 5.1.2 The main laboratory walk-in coldroom, located in Room 1912, is used for storage of analyzed extracts and all other samples. The shelves are organized as follows (see Figure 3):
- 5.1.2.1 Filters to be extracted are stored in the appropriate box (Teflon, nylon, or cellulose) immediately inside the coldroom on the right-hand side of the door.
  - 5.1.2.2 Citric Acid filters, denuder samples, and gray-label nylon samples are stored on the right side of the coldroom.
  - 5.1.2.3 Wet deposition samples are placed in large plastic tubs on the floor or shelves immediately inside the coldroom on the left-hand side of the door.
  - 5.1.2.4 Performance Evaluation Environment Canada (ECAN) and US Geological Survey (USGS) samples are placed on the shelves immediately inside the coldroom on the left-hand side of the door.
  - 5.1.2.5 Analyzed Teflon and nylon samples are stored on the left-hand side, with Teflon samples closest to the door.
  - 5.1.2.6 Analyzed cellulose samples are stored along the back of the coldroom.

**Figure 3** Sample Organization in Walk-in Coldroom



## **5.2 Long-term Storage**

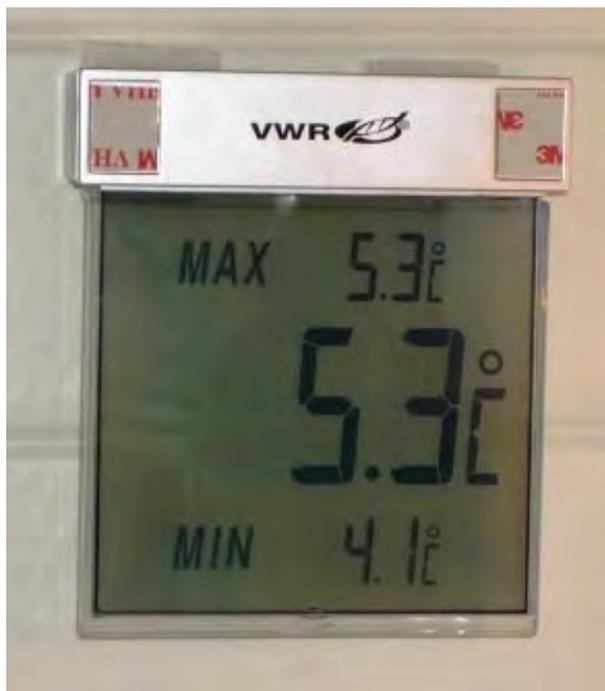
5.2.1 Samples are transferred from the main laboratory coldroom to the outside coldroom on an approximately quarterly basis and are stored for one year past the end of the sampling year.

5.2.2 After one year in the outside coldroom, samples are transferred to an on-site storage trailer for an additional year. This storage is not under climate control.

## **5.3 Temperature Monitoring**

5.3.1 Monitoring of all sample storage refrigerators and the inside walk-in coldroom is done on a daily basis, excluding weekends and holidays. The outside walk-in coldroom is checked weekly. The temperature displayed on the thermometer (see Figure 4) is recorded in the appropriate logbook.

**Figure 4** Thermometer



5.3.2 The temperature of all sample storage refrigerators and walk-in coldrooms shall be maintained at 4 degrees Celsius ( $^{\circ}\text{C}$ )  $\pm 2$   $^{\circ}\text{C}$  [2-6  $^{\circ}\text{C}$ ]. If the temperature deviates from this range, the coldroom should be checked to ensure the refrigeration system is operating and then the temperature checked again within 2 hours. If the temperature is still out of range, the Laboratory Operations Manager will be notified immediately and a service call will be placed. Actions taken for out of range conditions will be noted in the logbook.

#### **5.4 Corrective Actions**

When necessary, samples may be reanalyzed to determine if there was any adverse effect from being stored in a location that did not meet the temperature requirements.

## 6.0 REFERENCES

- AMEC E&I, Inc. (AMEC). 2011. Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.
- Clesceri, L.S., Greenberg, A.E., Trussel, R.R. (eds). 1989. *Standard Methods for the Examination of Water and Wastewater*, 17<sup>th</sup> edition. APHA-AWWA-WPCF, Washington, DC.
- U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.
- U.S. Environmental Protection Agency (EPA). 1983. *Methods for Chemical Analysis of Water and Wastes* (EPA 305.2), EPA-600/4-79-020, Revised March 1983.

## **7.0 ATTACHMENTS**

Attachment A – Temperature Log

## ATTACHMENT A Temperature Log

|                             |    |       |
|-----------------------------|----|-------|
| Corrected Temperature Range |    |       |
| 2°                          | to | 6° °C |

  
**ENVIRONMENTAL GAINESVILLE LAB**  
**TEMPERATURE LOG**

| Checked By                                | Date    | Temp °C | Thermometer Number | Comments |
|---|---------|---------|--------------------|----------|
| KWB                                       | 6/29/10 | 4.0     | 4569               |          |
| RW  | 6/30/10 | 4.0     | ↓                  |          |
| RW  | 7/1/10  | 4.5     | ↓                  |          |
| KWB                                       | 7/2/10  | 4.0     | ↓                  |          |
| RW  | 7/6/10  | 6.0     | ↓                  |          |
| KWB                                       | 7/7/10  | 5.0     | ↓                  |          |
| RW  | 7/8/10  | 4.5     | ↓                  |          |
| RW  | 7/9/10  | 4.5     | ↓                  |          |
| RW  | 7/12/10 | 4.0     | ↓                  |          |
| RW  | 7/13/10 | 4.0     | ↓                  |          |
| RW  | 7/14/10 | 4.5     | ↓                  |          |
| RW  | 7/15/10 | 4.0     | ↓                  |          |
| RW  | 7/16/10 | 4.0     | ↓                  |          |
| RW  | 7/19/10 | 4.5     | ↓                  |          |
| KWB                                       | 7/20/10 | 5.0     | ↓                  |          |
| RW  | 7/21/10 | 4.5     | ↓                  |          |
| KWB                                       | 7/22/10 | 4.5     | ↓                  |          |
| RW  | 7/23/10 | 4.5     | ↓                  |          |
| RW  | 7/26/10 | 4.0     | ↓                  |          |
| KWB                                       | 7/27/10 | 4.5     | ↓                  |          |
| RW  | 7/28/10 | 4.0     | ↓                  |          |
| KWB                                       | 7/29/10 | 4.0     | ↓                  |          |
| RW  | 7/30/10 | 4.0     | ↓                  |          |
| RW  | 8/2/10  | 5.0     | ↓                  |          |
| RW  | 8/3/10  | 4.0     | ↓                  |          |
| RW  | 8/4/10  | 4.0     | ↓                  |          |
| RW  | 8/5/10  | 4.0     | ↓                  |          |
| ACTIONS TAKEN FOR OUT OF RANGE CONDITONS: |         |         |                    |          |

Book # B-1865

**TITLE: WHATMAN CELLULOSE FILTER PREPARATION WITH POTASSIUM CARBONATE**

Effective Date: 11-4-11

Prepared by: Kathy W. Barry *Kathy W Barry*  
 Laboratory Chemist

Reviewed by: Garry L. Price *Garry L Price*  
 Laboratory Operations Manager

Reviewed by: Marcus O. Stewart *Marcus O. Stewart*  
 QA Manager

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |               |                 |                         |
|---------------|---------------|-----------------|-------------------------|
| Reviewed by:  | Title:        | Date:           | Signature:              |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/26/12</i> | <i>Marcus O Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>6/01/11</i>  | <i>Marcus O Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/16/15</i> | <i>Marcus O Stewart</i> |
|               |               |                 |                         |
|               |               |                 |                         |

## **WHATMAN CELLULOSE FILTER PREPARATION WITH POTASSIUM CARBONATE**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the preparation of Whatman #41 cellulose filters for Clean Air Status and Trends Network (CASTNET) filter packs.

### **2.0 SCOPE**

This method is applicable to all prepared Whatman cellulose filters used to capture sulfur dioxide (SO<sub>2</sub>) in the atmosphere.

### **3.0 SUMMARY**

Whatman #41 cellulose filters are rinsed to remove potential sulfate and nitrate contamination and then impregnated with a potassium carbonate solution.

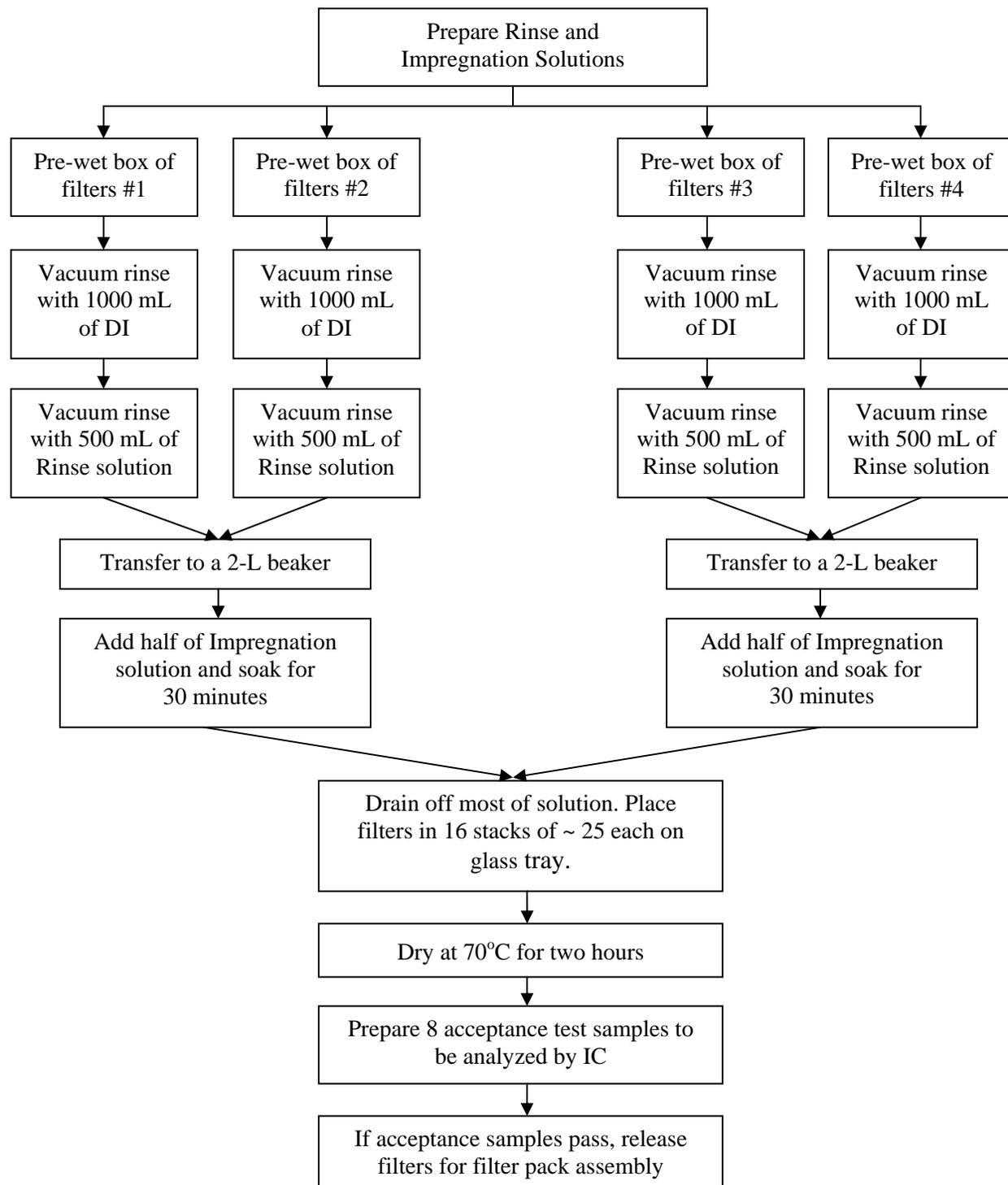
#### **3.1 Method Interferences**

Reagents from new vendors and those without a Certificate of Analysis listing the sulfate concentration should be tested for the presence of sulfate prior to use in the impregnation solution.

#### **3.2 Deviations from the Method**

A general overview of the steps described in this SOP is depicted in Figure 1. Deviations from the analytical method described in this SOP are not permitted.

**Figure 1** Overview – Laboratory Operations Procedures for Whatman Filter Preparation



## **4.0 MATERIALS (SUFFICIENT FOR ONE IMPREGNATION GROUP)**

### **4.1 Apparatus (see Figure 2)**

Two 2-liter (L) Class A volumetric flasks

Glass trays

Two 2-L glass beakers

100-milliliter (mL) graduated cylinder

Large weighing boats

Powder-free gloves

Plastic forceps

Drying oven

Parafilm paraffin/polyethelyene material

Buchner funnel

Timer

Three high-density polyethylene (HDPE) wash bottles

Whatman Grade No. 41, 47-millimeter (mm) -diameter ashless cellulose filters

Resealable plastic bags and appropriate labels

Plastic-backed absorbent paper

KimWipes, large and small sizes

Vacuum apparatus, pump, side-arm vacuum flask, and appropriate tubing

Filter Impregnation Logbook

60-mL Nalgene bottle

### **4.2 Reagents**

Reagent water: DI water with resistivity of 15 mega ohms ( $M\Omega$ ) or greater, derived from mixed bed ion exchangers, activated carbon filters, and polishing exchangers. Water should contain particles no larger than 0.20 micrometer ( $\mu\text{m}$ ).

Anhydrous Potassium Carbonate ( $\text{K}_2\text{CO}_3$ ) granular CAS#584-08-7

Glycerol, American Chemical Society (ACS) reagent or better grade CAS# 56-81-5 with Certificate of Analysis (see Figure 3)

**Figure 2** Whatman Cellulose Filter Impregnation Apparatus



**Figure 3** Glycerol Certificate of Analysis



**Certificate of Analysis**

Product: BDH Glycerol  
Cat: BDH1172-1LP  
Lot: 021610G  
Date of Manufacture: 02/17/10  
Use Before: 08/18/11

| <u>Test</u>                  | <u>Specification</u> |             | <u>Result</u> | <u>Units</u> |
|------------------------------|----------------------|-------------|---------------|--------------|
|                              | <u>Min.</u>          | <u>Max.</u> |               |              |
| Appearance                   | Clear                |             | Clear         |              |
| Identification               | Passes test          |             | Passes test   |              |
| Water                        |                      | 0.3         | 0.048         | %            |
| Residue after Ignition       |                      | 0.01        | <0.01         | %            |
| Assay                        | 99.7                 |             | 99.9          | %            |
| Color (APHA)                 |                      | 20          | <20           |              |
| Specific Gravity (25°C/25°C) | 1.2613               |             | 1.2616        |              |
| Chlorides                    |                      | 10          | <10           | ppm          |
| Sulfates                     |                      | 20          | <20           | ppm          |
| Heavy Metals (as Pb)         |                      | 5           | <5            | ppm          |
| Chlorinated Compounds        |                      | 30          | <30           | ppm          |
| Fatty Acids and Esters       | Passes test          |             | Passes test   |              |

Quality Control Approval

Distributed Exclusively by



## 5.0 SAFETY

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

## 6.0 PROCEDURE

### 6.1 Filter Impregnation Solution

- 6.1.1 Add 300 – 500 mL DI water to a 2-L volumetric flask.
- 6.1.2 Weigh 250 grams (g) of  $K_2CO_3$  into a weighing boat. Quantitatively transfer the  $K_2CO_3$  to the volumetric flask and swirl to dissolve. Vent the stopper on the flask frequently since some effervescence will occur.
- 6.1.3 Repeat step 6.1.2 (for a total of 500 g of  $K_2CO_3$  in the flask).
- 6.1.4 Measure 100 mL of glycerol and then carefully pour the glycerol into the flask. Cap and shake the solution carefully to ensure thorough mixing, being sure to vent the flask often during this process.
- 6.1.5 Bring up to volume with DI water. Cap securely and invert at least 10 times to ensure thorough mixing, being sure to vent the flask periodically.

### 6.2 Filter Rinse Solution

- 6.2.1 Weigh 28 g of  $K_2CO_3$  into a weighing boat.
- 6.2.2 Add 300-500 mL DI water to a 2-L volumetric flask. Quantitatively transfer the  $K_2CO_3$  to the volumetric flask and swirl to dissolve. Vent the stopper on the flask periodically since some effervescence will occur.
- 6.2.3 Cap and shake the solution carefully to ensure thorough mixing, being sure to vent the flask often during this process.
- 6.2.4 Bring up to volume with DI water. Cap securely and invert at least 10 times to ensure thorough mixing. Vent periodically.

### 6.3 Impregnation Procedure

- 6.3.1 Pre-wet one box of Whatman cellulose filters (approximately 100 filters) under a stream of DI water. Use plastic forceps (see Figure 4).

**Figure 4** Rinsing Filters



6.3.2 Wearing powder-free plastic gloves, place one box of Whatman filters (approximately 100 filters) in a small Buchner funnel inserted into the vacuum flask. Using vacuum aspiration, rinse the filters with approximately 1000 mL of DI water (see Figure 5).

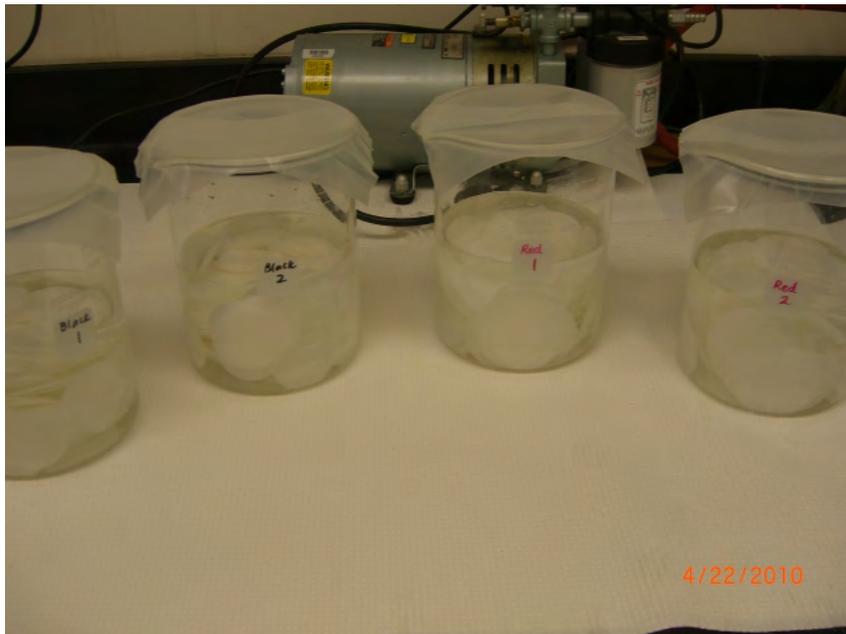
*Note: A wash bottle works well (contains 500 mL) and allows the analyst to direct the water stream around and between the filters. Proper washing is required to remove potential sulfate and nitrate contamination.*

**Figure 5** Buchner Funnel Rinse



- 6.3.3 Aspirate filters to near dryness.
- 6.3.4 Rinse the filters with approximately 500 mL of the Filter Rinse solution. Aspirate to near dryness.
- 6.3.5 Carefully transfer filters into a 2-L beaker. Cover with Parafilm.
- 6.3.6 Repeat this procedure with another set of 100 filters until a total of 200 filters are in the 2-L beaker. Use another 2-L beaker for the remaining 200 filters.
- 6.3.7 Fill a 60-mL Nalgene bottle with approximately 50 mL of the Impregnation Solution before proceeding.
- 6.3.8 Carefully divide the Impregnation Solution between the two large 2-L beakers (see Figure 6). Swirl the filters gently in the solution and the cover the beaker with Parafilm.

**Figure 6** Filters Soaking in Beakers



- 6.3.9 Allow the filters to soak in the solution for at least for 30 minutes, swirling the filters in the solution at least two more times.
- 6.3.10 Rinse the glass drying trays with copious amounts of DI water using gloved hands to rub the surfaces clean. Sit the trays out to dry on the absorbent paper. Blot excess water from trays using KimWipes.
- 6.3.11 Preheat the drying oven to 70 degrees Celsius ( $^{\circ}\text{C}$ ).
- 6.3.12 Fill out information required in the Impregnation notebook (see Attachment A).
- 6.3.13 After the filters have soaked for the appropriate amount of time, drain off most of the Impregnation Solution into the appropriately marked waste receptacle. With clean plastic forceps, carefully remove the filters a few at a time and place in 16 small stacks per clean glass tray (approximately 25 per stack). Work quickly to prevent contamination (see Figure 7).

**Figure 7** Filters Stacked and Drying



6.3.14 **Place** the trays in the oven for two hours to dry, checking on the filters periodically.

6.3.15 **Remove** the trays from the oven. Continue working quickly to prevent contamination.

6.3.16 **Eight** acceptance test samples are prepared for each impregnation group. Each sample consists of two filters. The necessary labels are kept in the extraction area.

- Label the appropriate number of 60-mL Nalgene bottles with preprinted sample labels: 9 bottles total per Impregnation group (8 bottles for filters and one empty bottle to test 600  $\mu$ L of the Impregnation Solution).
- Label a resealable plastic bag and the reserved bottle of impregnation fluid with impregnation labels (see Attachment B).
- Discard the top two filters of each stack. Place one filter from the middle of each stack into the bottles, two filters per bottle. The middle filters of two stacks comprise one sample. Place the remaining filters from each stack, discarding the two bottom filters, in the resealable plastic bag.
- After all filters are bagged and bottled, place the acceptance test samples, the bagged filters, and the reserved impregnation fluid in the extraction area to be tested.
- Submit the date of impregnation and the sample names of the acceptance tests to data services via e-mail for sample log-in.

6.3.17 Rinse all used equipment with DI water and return to designated storage area.

#### **6.4 Corrective actions**

Cellulose filters are acceptance tested after the impregnation procedure. Between 400 and 800 filters are prepared at one time. The filters are assigned to an impregnation group of 400 filters that is uniquely associated both with the impregnation date and a group of acceptance test samples. Each impregnation group is isolated throughout the entire impregnation procedure and is stored separately before use. Acceptance testing is performed on 4 percent of the filters contained in each impregnation group. The acceptance test samples are prepared with two cellulose filters per sample and are extracted and analyzed according to normal procedures. Acceptance test results for an impregnation group are considered satisfactory if no more than one sample in the group shows sulfate contamination above 3.83 micrograms ( $\mu\text{g}$ ) (equivalent to an approximate ambient concentration of 0.17  $\mu\text{g}$  per cubic meter [ $\mu\text{g}/\text{m}^3$ ] as  $\text{SO}_2$ ). All cellulose filter acceptance test results are stored in Element. The impregnation group used for each filter pack is referenced in the filter pack preparation logbook. If the analytical results fail the acceptance limits, the batch is discarded.

## **7.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, D.C. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

## **8.0 ATTACHMENTS**

Attachment A – Potassium Carbonate Filter Impregnation Log

Attachment B – Impregnation Label

## ATTACHMENT A Potassium Carbonate Filter Impregnation Log



### Potassium Carbonate Filter Impregnation Log

Date: 4/5/10      K<sub>2</sub>CO<sub>3</sub> CAS# 584-08-7      Group A Lot: G47151  
 Analyst: K. Grakey      Group B Lot: G47151  
 Glycerol CAS# 56-81-5 Lot: H21302

|           | Filter Lot#           | No. Filters |           | Filter Lot#           | No. Filters |
|-----------|-----------------------|-------------|-----------|-----------------------|-------------|
| Group A:  | <u>J11582573</u>      | <u>400</u>  | Group B:  | <u>J11582573</u>      | <u>400</u>  |
| WAT IDs:  | <u>102005-01 → 0%</u> |             | WAT IDs:  | <u>102005-09 → 16</u> |             |
| WSOL IDs: | <u>102005-17</u>      |             | WSOL IDs: | <u>102005-18</u>      |             |

Soaking      Drying      Oven Temp (°C): 70  
 Start Time: 10:45      End time: 11:15      Start Time: 11:40      End time: 13:40

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Date: 4/23/10      K<sub>2</sub>CO<sub>3</sub> CAS# 584-08-7      Group A Lot: G47151  
 Analyst: K. Barry      Group B Lot: G47151  
 Glycerol CAS# 56-81-5 Lot: 011810C (804)

|           | Filter Lot#          | No. Filters |           | Filter Lot#           | No. Filters |
|-----------|----------------------|-------------|-----------|-----------------------|-------------|
| Group A:  | <u>X11588562</u>     | <u>400</u>  | Group B:  | <u>J11582573</u>      | <u>400</u>  |
| WAT IDs:  | <u>1025007 (1-8)</u> |             | WAT IDs:  | <u>1025007 (9-16)</u> |             |
| WSOL IDs: | <u>1025007-17</u>    |             | WSOL IDs: | <u>1025007-18</u>     |             |

Soaking      Drying      Oven Temp (°C): 70  
 Start Time: 10:20      End time: 10:50      Start Time: 11:20      End time: 1:35

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

## ATTACHMENT B Impregnation Label

|          |                          |
|----------|--------------------------|
| TYPE:    | <u>Wintman - A</u>       |
| DATE:    | <u>4/23/10</u>           |
| NUMBER:  | <u>1025007 (1-8, 17)</u> |
| PREP BY: | <u>KWB</u>               |

This is a copy of a recent actual label. Red and black marks are used to differentiate between the impregnation groups when more than one group is prepared on a given day. (This label is for the black group for that day.)

1025007 (1-8) Refers to the range of Element identification (ID) numbers of the filter acceptance test samples for these filters.

1025007-17 Refers to the Element IDs of the impregnation fluid acceptance test sample for these filters. This sample is prepared in the same manner as the Method Blank for cellulose filters (GLM3180-001).

**TITLE: COATING AND EXTRACTION OF AMBIENT AIR  
DENUDER TUBES**

Effective  
Date: 11-4-11

Prepared by: Kathy W. Barry  
Laboratory Chemist

Kathy W. Barry

Reviewed by: Garry L. Price  
Laboratory Operations Manager

Garry L. Price

Reviewed by: Marcus O. Stewart  
QA Manager

Marcus O. Stewart

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |        |          |                   |
|---------------|--------|----------|-------------------|
| Reviewed by:  | Title: | Date:    | Signature:        |
| MS            | QA Mgr | 10/26/11 | Marcus O. Stewart |
| MS            | QA Mgr | 10/21/11 | Marcus O. Stewart |
| MS            | QA Mgr | 10/11/11 | Marcus O. Stewart |
|               |        |          |                   |
|               |        |          |                   |

## COATING AND EXTRACTION OF AMBIENT AIR DENUDER TUBES

### 1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the coating and extraction of denuder tubes used in ambient air collection.

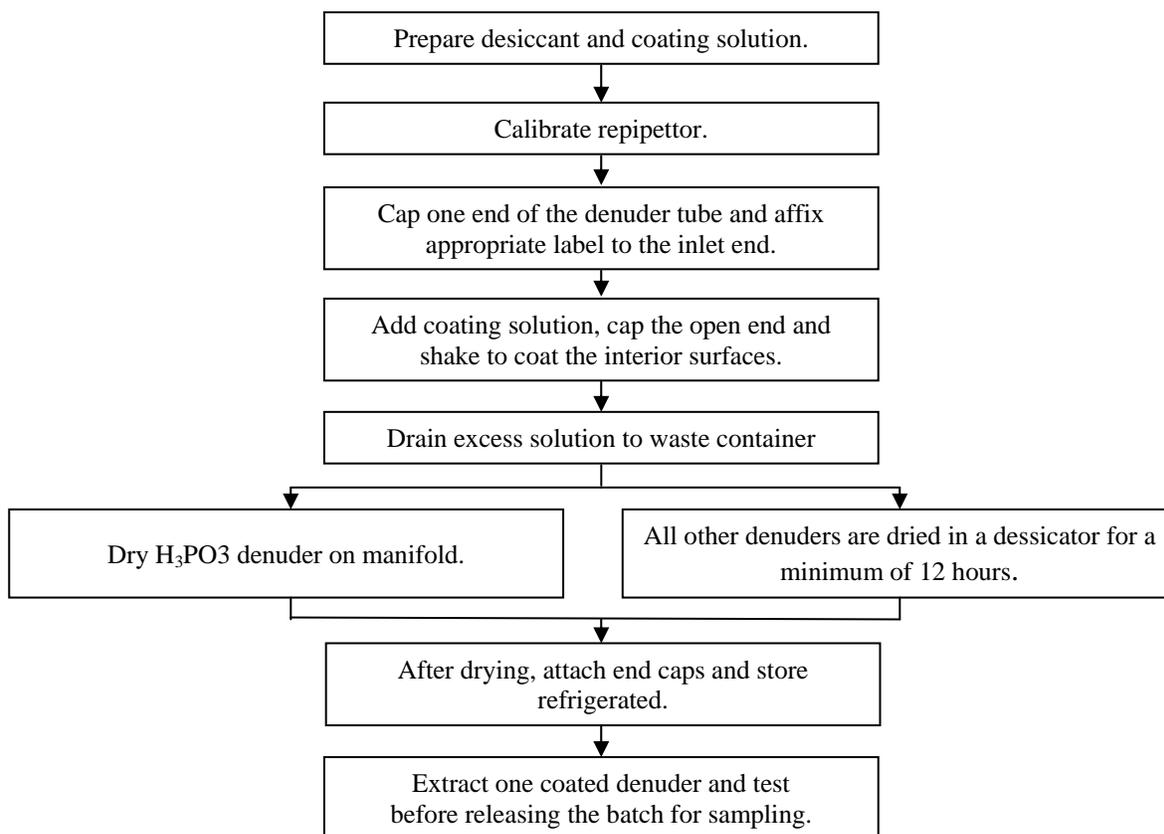
### 2.0 SCOPE

This SOP applies to all denuders that are prepared for the CASTNET and CASTNET-like ambient air projects.

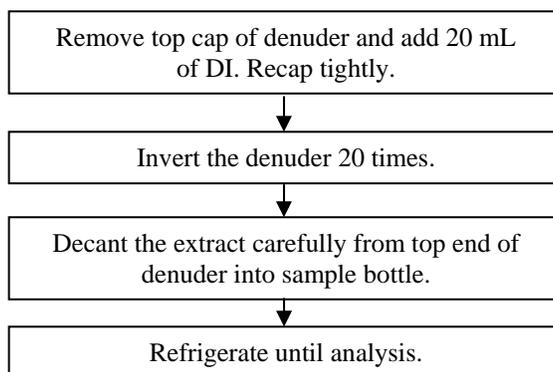
### 3.0 SUMMARY OF METHOD

A general overview of the steps described in this SOP for coating denuders is depicted Figure 1. Figure 2 depicts an overview of the extraction steps. Deviations from the analytical method described in this SOP are not permitted.

**Figure 1** Overview – Laboratory Operations Procedures for Coating Denuder Tubes



**Figure 2** Overview – Laboratory Operations Procedures for Extracting Exposed Denuder Tubes



## 4.0 MATERIALS

### 4.1 Apparatus

Desiccator

Coating labels for tubes

Repipettors

Powder-free gloves

Desiccant

Pre-cleaned denuders

Large metal trays

Drying oven

Class A volumetric flasks, 1000 mL

Nitrogen cylinder

Nalgene extract bottles, 30 mL

### 4.2 Reagents

- Reagent Water: Deionized (DI) water of resistivity of 15 mega Ohms (M $\Omega$ ) or greater, derived from mixed bed ion exchangers, activated carbon filters and polishing exchangers. Water should contain particles no larger than 0.20 micrometers.
- Methanol (MeOH), ACS reagent grade or better, CAS # 67-56-1.
- Glycerol, ACS reagent grade or better, CAS # 56-81-5.
- Citric Acid, ACS reagent grade or better, CAS # 5949-29-1.
- Sodium Carbonate(Na<sub>2</sub>CO<sub>3</sub>), ACS reagent grade or better, CAS # 497-19-8
- Potassium Chloride(KCl), ACS reagent grade or better, CAS # 7447-40-7
- Phosphorus Acid (H<sub>3</sub>PO<sub>3</sub>), ACS reagent grade or better, CAS #13598-36-2
- Citric Acid Coating Solution (1 percent): Dissolve 10 g of citric acid in 900 mL of MeOH in a 1000 mL volumetric flask. Dilute to volume with DI.

- Sodium Carbonate Coating Solution (1 percent): Dissolve 10 g of  $\text{Na}_2\text{CO}_3$  and 10 g of glycerol in 500 mL of DI in a 1000mL volumetric flask. Dilute to volume with MeOH.
- Potassium Chloride Coating Solution (1 percent): Dissolve 10 g of KCl in 500 ml of DI in a 1000 mL volumetric flask. Dilute to volume with MeOH.
- Phosphorus Acid Coating Solution (1 percent): Dissolve 10 g of  $\text{H}_3\text{PO}_3$  in 500 mL of MeOH in a 1000 mL volumetric flask. Add another 400 mL of MeOH and dilute to volume with DI.

## 5.0 SAFETY

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. If the analyst is not familiar with the hazards associated with the chemicals being used, the Materials Safety Data Sheets (MSDS) must be consulted. The MSDS by chemical and brand can be found in the AMEC laboratory or at the Web sites <http://www.msdssearch.com> or <http://www.hazard.com/msds/index.php> using the CAS number.

## 6.0 PROCEDURE

### 6.1 Denuder Coating

- 6.1.1 Prepare the desiccant by drying it 2-4 hours or until dry at 105 degrees Celsius ( $^{\circ}\text{C}$ ). Pour the dried desiccant into the metal trays and place in the desiccator.
- 6.1.2 Obtain the appropriate coating solution. Coating solutions are made in the laboratory and are given to the CASTNET technician responsible for coating the denuders.
- 6.1.3 Calibrate the repipettors (see Figure 3) prior to each day's use by determining the mass of deionized water delivered. Adjust the repipettor to deliver  $10.0 \pm 0.5$  g.

**Figure 3** Repipettors



6.1.4 Securely cap the bottom of the denuder tube. The quartz inner tube is recessed approximately one inch on the top end (inlet side) of the denuder. On the bottom (outlet) of the denuder, the inner quartz tube is recessed about  $\frac{1}{8}$  inch from the end of the aluminum tube (see Figure 4 and 5).

**Figure 4** Denuder top (inlet)



**Figure 5** Denuder bottom (outlet)



6.1.5 Affix a denuder coating identification label to the outside of the denuder tube at the inlet end (see Figure 6). Labels are color-coded to aid in identification: citric acid – yellow, sodium carbonate – green, potassium chloride – orange, phosphorous acid – red.

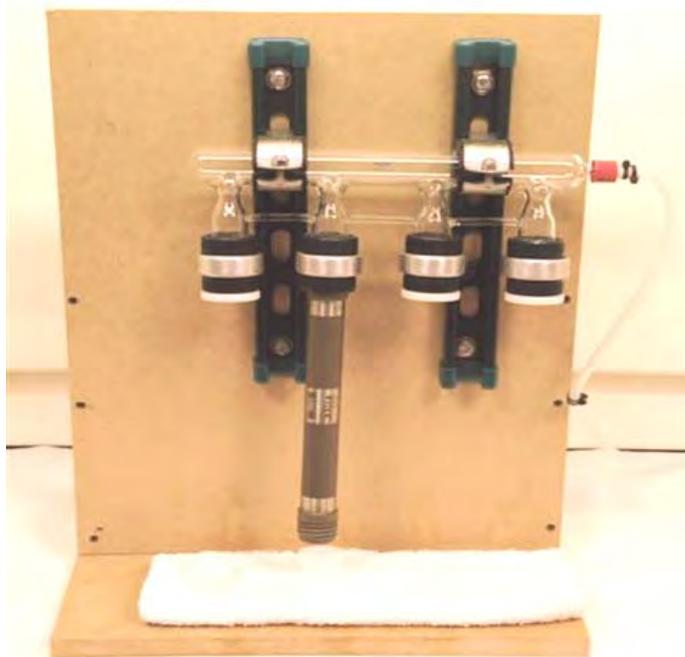
**Figure 6** Denuder tubes with labels



6.1.6 Using the repipettor, deliver 10 mL of the coating solution directly into the top of the denuder tube. Securely cap the open end of the denuder. Invert the denuder 20 times, alternately using a figure eight motion and vertical shaking to thoroughly coat the interior surfaces of the denuder.

- 6.1.7 Carefully open both ends of the denuder and drain the excess solution out into an appropriate waste container. **Note: never dispose of coating solutions (or any chemicals) in laboratory sinks.**
- 6.1.8 Phosphorous acid denuders are screwed securely into the URG drying manifold so that the direction of flow is downward (see Figure 7). Dry for approximately 20 minutes with a 5 liter per minute nitrogen purge. The “honeycomb” quartz inside the URG denuder will cloud when dry.

**Figure 7** URG Drying Manifold.



- 6.1.9 Citric acid, carbonate and potassium chloride denuders and end caps are placed inside the assigned desiccator to dry for a minimum of 12 hours (see Figure 8).

**Figure 8** Dessicator



6.1.10 Once dry, attach the end caps and refrigerate until shipment.

6.1.11 For each coating batch, one denuder will be extracted following the procedure outlined in section 6.2. The results must be less than or equal to two times the reporting limit for the batch to be used.

6.1.12 Prior to shipment, a color coded Lab sample identification label will be attached to the denuder.

## **6.2 Extracting Exposed Denuders.**

6.2.1 Ensure the bottom cap of the denuder is attached firmly and remove the top cap.

6.2.2 Using a repipettor, add 20 mL of DI water into the denuder and cap tightly. Invert the denuder 20 times.

6.2.3 Tap the top cap a couple of times. Unscrew the top cap and set aside.

6.2.4 Decant the extract carefully from top end of the denuder into the sample extract bottle.

6.2.5 Remove the color coded Lab sample identification label from the denuder and attach it to the sample extract bottle.

6.2.6 Refrigerate until analysis.

## **7.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

## **8.0 ATTACHMENTS**

This SOP does not contain attachments.

**TITLE: CLEAN AIR STATUS AND TRENDS NETWORK (CASTNET)  
 THREE-STAGE AMBIENT AIR FILTER PACK RECEIPT,  
 DISASSEMBLY AND LOGIN**

Effective  
 Date: 11-9-11

Prepared by: Kathy W. Barry Kathy W. Barry  
 Laboratory Chemist

Reviewed by: Garry L. Price Garry L. Price  
 Laboratory Operations Manager

Reviewed by: Marcus O. Stewart Marcus O. Stewart  
 QA Manager

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Procedure
- 6.0 References
- 7.0 Attachments

| Annual Review |              |                 |                          |
|---------------|--------------|-----------------|--------------------------|
| Reviewed by:  | Title:       | Date:           | Signature:               |
| <u>MS</u>     | <u>QAMgr</u> | <u>10/26/12</u> | <u>Marcus O. Stewart</u> |
| <u>MS</u>     | <u>QAMgr</u> | <u>10/31/14</u> | <u>Marcus O. Stewart</u> |
| <u>MS</u>     | <u>QAMgr</u> | <u>10/1/15</u>  | <u>Marcus O. Stewart</u> |
|               |              |                 |                          |
|               |              |                 |                          |

## **CLEAN AIR STATUS AND TRENDS NETWORK (CASTNET) THREE-STAGE AMBIENT AIR FILTER PACK RECEIPT, DISASSEMBLY AND LOGIN**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for receiving and unpacking the three-stage filter packs, and logging the samples into the Laboratory Information Management System (LIMS).

### **2.0 SCOPE**

This SOP applies to all filter packs that are prepared for the CASTNET and CASTNET-like ambient air projects.

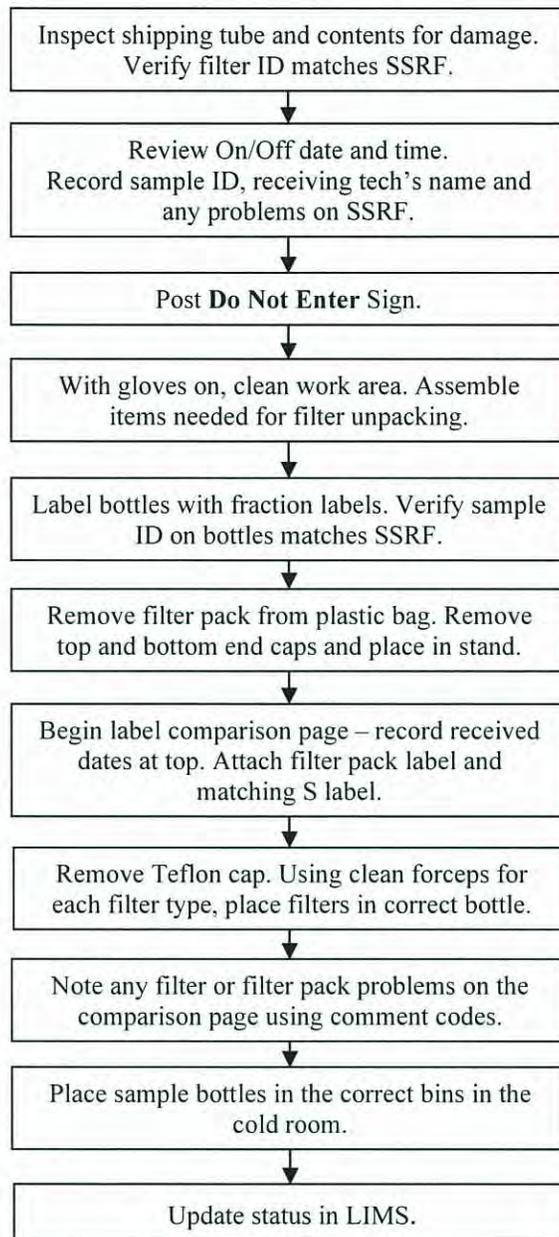
### **3.0 SUMMARY OF METHOD**

A general overview of the steps described in this SOP is depicted Figure 1. Deviations from the analytical method described in this SOP are not permitted.

### **4.0 MATERIALS**

- Powder-free gloves
- Clean forceps
- Clean high-density polyethylene (HDPE) bottles (30 mL, 60 mL)
- Large plastic trays
- Filter packing/unpacking stand
- Filter pack wrench
- Kimwipes
- Field group folder with fraction labels (see Attachment A):
  - S - Sample Log-in, white label
  - T - Teflon filter, white label
  - N - Nylon filter, orange label
  - W – Cellulose (K<sub>2</sub>CO<sub>3</sub> impregnated) filter, blue label
- Large tubs
- Filter packing notebook
- Site Status Report Form (SSRF)

**Figure 1** Overview – Laboratory Operations Procedures for Filter Pack Disassembly



## 5.0 PROCEDURE

### 5.1 Receipt, Initial Inspection, and Assignment of Sample Number to CASTNET Filter Packs

- 5.1.1 The filter pack shipping tubes are received at the designated AMEC shipping/receiving area. The tubes are normally shipped to AMEC by the U.S. Postal Service using Merchandise Return Labels. Other shipping providers are acceptable.
- 5.1.2 The filter pack shipping tubes are then delivered by the AMEC shipping/receiving technician directly to the CASTNET filter pack receiving area and are placed into the custody of the CASTNET filter pack technician.
- 5.1.3 Examine the shipping container for damage from shipping and note any variances in the filter-packing notebook.
- 5.1.4 Open the shipping tube and remove the filter pack and Site Status Report Form (SSRF) (see Figure 2). Verify that the filter identification (ID) number on the filter pack agrees with the filter ID number stated on the chain of custody label on the SSRF. The site number listed on the chain of custody label is also verified with the site number entered by the site operator in the top left corner of the form. If these are not in agreement or not completed, note it in the shaded Comments area of the form (see Figure 3).

**Figure 2** Shipping tubes and filter pack.



- 5.1.5 Initial and date the SSRF form in the shaded box labeled “Lab Use Only” (see Figure 3).

Figure 3 SSRF

| CASTNET  |  | SITE STATUS REPORT FORM (SSRF)  |  |  |  |  |  |
|--|--|---|--|--|--|--|--|
| DAS AND OZONE  |  | FILTER PACK INFO  |  | SITE OBSERVATIONS DURING FILTER INSTALLATION   |  |  |  |
| SITE NAME / NO<br><i>CND 125</i>   |  | ACTUAL SAMPLE RUN<br><i>1034001-21</i>  |  | CIRCLE IF PRESENT: (DEW) FROST SNOW FOG RAIN   |  |  |  |
| VISIT DATE / DAY<br><i>08-17-10 Tue</i>  |  | FILTER PACK ID#<br><i>1034001-21</i>  |  | WHAT % OF LEAVES: 0-25% 26-50% 51-75% 76-100%  |  |  |  |
| FIELD OPERATIONS COORDINATOR CALLED:<br>(name) <i>Trey Harrison</i>  |  | DATE<br><i>08/17/10 08:24/10</i>  |  | HAVE DROPPED <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>    |  |  |  |
| DAS PRIMARY BACKUP   |  | TIME<br><i>08:17:00 08:24:10</i>  |  | HAVE FALL COLOR <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> |  |  |  |
| JULIAN DATE <input checked="" type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>  |  | MFC (PUMP OFF) <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>            |  | ARE GREEN <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input checked="" type="checkbox"/>       |  |  |  |
| TIME (2000H) <input checked="" type="checkbox"/> <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> |  | MFC LEAK CHECK <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> |  | CURRENT 5-MINUTE AVERAGES  |  |  |  |
| DATA DOWNLOADED <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>                    |  | DAS FLOW (LPM) <i>7.50 7.50</i>   |  | REASONABLE YES NO  |  |  |  |
| FILES BACKED UP <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>                    |  | ROTAMETER (LPM) <i>1.50 1.50</i>  |  | EQUIPMENT  |  |  |  |
| FILE NAMES   |  | ELAPSED TIME (HRS) <i>08:00 08:00</i>   |  | RAINFALL (INCHES) <i>0.00</i>  |  |  |  |
| OZONE CHECKS   |  | RETURN SHIPMENT PREPARED BY <i>Hazel Perry</i> DATE <i>108-24-10</i>  |  | CLEAN & LEVEL <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>   |  |  |  |
| A B  |  | EXPECTED SHIPMENT DATE <i>08-24-10</i>  |  | 15-TIP CHECK <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>    |  |  |  |
| SAMPLE FREQUENCY   |  | LAB USE ONLY  |  | WIND DIRECTION (DEGREES) <i>070 070</i>  |  |  |  |
| WORK   |  | LAB SAMPLE NO. <i>1034001-21-CND125</i>   |  | WIND SPEED (Mph) <i>0.0 0.0</i>  |  |  |  |
| CELL PRESSURE (mm)   |  | LAB TECH RECEIPT <i>H. Reed</i> DATE <i>08-17-10</i>  |  | TEMPERATURE (°C) <i>0.0 0.0</i>  |  |  |  |
| CELL TEMPERATURE (°C)  |  | COMMENTS:   |  | TEMPERATURE (°F) <i>32.0 32.0</i>  |  |  |  |
| SAMPLE FLOW (LPM)  |  | CHAIN-OF-CUSTODY LABEL  |  | BLOWER WORKING <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>  |  |  |  |
| OUTSIDE FILTER CHANGED <input type="checkbox"/> <input type="checkbox"/>   |  | WEEK 33 2010  |  | RELATIVE HUMIDITY (%) <i>0.0 0.0</i>   |  |  |  |
| INSIDE FILTER CHANGED <input type="checkbox"/> <input checked="" type="checkbox"/>   |  | Filter Pack ID #: <i>1034001-21</i> Site: <i>CND125</i>   |  | SOLAR RADIATION (W/M <sup>2</sup> ) <i>0.0 0.0</i>   |  |  |  |
| WATER IN LINE/TRAP <input type="checkbox"/> <input checked="" type="checkbox"/>  |  | Scheduled On Date: <i>08/17/2010</i>  |  | CLEAN & LEVEL <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>   |  |  |  |
| HERMETIC MOTOR <input type="checkbox"/> <input type="checkbox"/>   |  | SHIPMENT OPENED BY: <i>Hazel Perry</i> DATE: <i>08-17-10</i>  |  | WETNESS SENSOR <input checked="" type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>  |  |  |  |
| FITTED WINDSHIELD <input type="checkbox"/> <input type="checkbox"/>  |  | PACKED BY: 3578   |  | SHIELDER TEMPERATURE (°F) MAX <input checked="" type="checkbox"/> <input type="checkbox"/>                                     |  |  |  |
| OFFSET OR BACKSCATTER <i>0.00</i>  |  |   |  | MIN <input checked="" type="checkbox"/> <input type="checkbox"/>   |  |  |  |
| SPAN DR. COEFFICIENT <i>1.041</i>  |  |   |  | THERMOMETER RESET YES <input checked="" type="checkbox"/> NO <input type="checkbox"/>  |  |  |  |
| AUTO CAL ZERO ppb 400 ppb 90 ppb   |  |   |  | NOTES:<br><i>Overcast skies<br/>Light, cool breeze</i>   |  |  |  |
| SPAN CAL <i>10 21667 D5F -2.48% -1.7%</i>  |  |   |  |  |  |  |  |
| DMC USE ONLY   |  |   |  |  |  |  |  |
| REVIEWED BY / ON   |  |   |  |  |  |  |  |

- 5.1.6 Review the filter pack On/Off Date and Time, for completeness and correctness. If these data are incomplete or inconsistencies exist, note it on the form and initial and date. If there are extensive problems with the SSRF, notify the Project Manager.
- 5.1.7 Assign the AMEC sample number according to the recorded filter On Date. Each Tuesday is assigned a CASTNET sample week number. The On Date on the SSRF corresponds to a sample week number on the CASTNET calendar. This sample week number is used to determine the correct field group folder to use. The field group setup follows this example:  
XXYY001-Z, Where: XX = year,  
YY = week number, and  
Z = assigned number for each site.  
The last digit "1" denotes the Element project. This number represents the CASTNET base program  
Clearly write the AMEC sample number in the shaded box labeled "Lab Use Only" on the SSRF.

## 5.2 Unpacking Procedure:

5.2.1 Prior to unpacking, post the Do Not Enter sign (see Figure 4).

**Figure 4** Do Not Enter Sign



- 5.2.2 Powder-free gloves must be worn when performing this procedure.
- 5.2.3 Clean the work area and packing/unpacking stand with Kimwipes. Items needed in the work area for unpacking are as follows: a bag of clean forceps; 30 and 60 mL bottles; Teflon, nylon, and  $K_2CO_3$ -impregnated cellulose fraction and sample tracking labels (T, N, W, S labels); a large plastic tub; and paper for the label comparison sheet.
- 5.2.4 Label the bottles with the preprinted computer fraction labels. Attach the blue W fraction label to the 60 mL bottle. The other fraction labels are for the 30 mL bottles. Verify that the sample numbers on the bottles match the sample number recorded on the SSRF form.
- 5.2.5 Remove the filter pack from the resealable plastic bag, and remove the top and bottom end caps.
- 5.2.6 Begin the label comparison page by removing the Filter Pack ID label from the filter pack and attaching it to one side of a sheet of paper. Next to the Filter Pack ID label, attach the corresponding S label. The S label should agree with the sample fraction labels attached to the small sample bottles in Step 5.2.4. Record the received date at the top of the label comparison page (see Figure 5).

Figure 5 Label Comparison Page

08-27-10

1034001-46 S SITE: LRL117  
 On: 08/17/10

1034001-47 S SITE: LYK123  
 On: 08/17/10

1034001-62 S SITE: PRK134  
 On: 08/17/10

1034001-68 S SITE: SAN189  
 On: 08/17/10

1034001-70 S SITE: SHN418  
 On: 08/17/10

1034001-71 S SITE: SND152  
 On: 08/17/10

1034001-73 S SITE: STK138  
 On: 08/17/10

ON DATE 08-17-10  
Filter pack  
SITE: LRL117 Sampled: 08/17/10

1034001-46

Filter pack  
SITE: LYK123 Sampled: 08/17/10

1034001-47

Filter pack  
SITE: PRK134 Sampled: 08/17/10

1034001-62

Filter pack  
SITE: SAN189 Sampled: 08/17/10

1034001-68

Filter pack  
SITE: SHN418 Sampled: 08/17/10

1034001-70

Filter pack  
SITE: SND152 Sampled: 08/17/10

1034001-71

Filter pack  
SITE: STK138 Sampled: 08/17/10

1034001-73

code  
T1, w3

- 5.2.7 Put the gray clamp nut on the packing/unpacking stand (see Figure 6). Loosen the filter pack with the green vinyl packing wrench. Remove the clear Teflon cap, and place it in the plastic tub to be washed later. Be careful not to let the filter stick to the Teflon cap during removal.

**Figure 6** Unpacking Stand



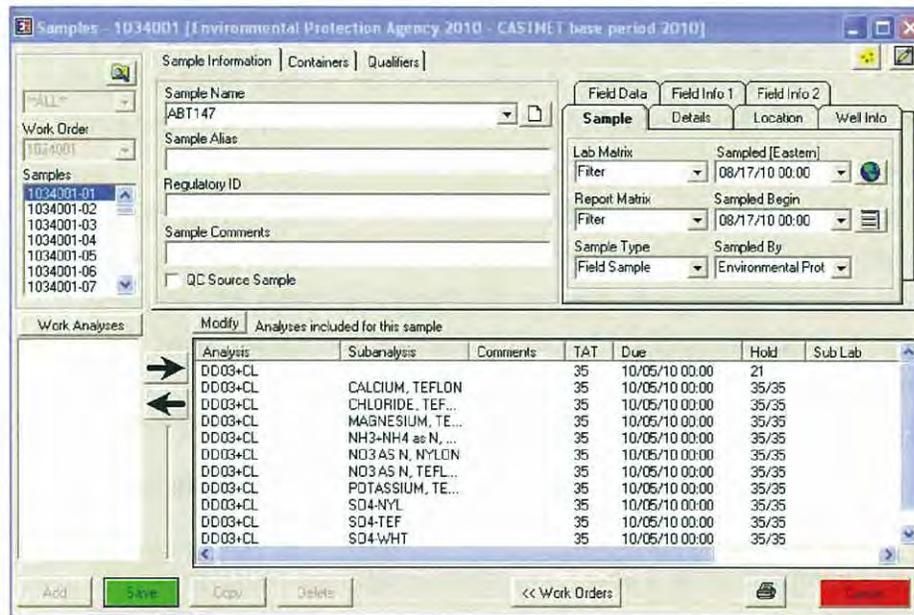
- 5.2.8 Using clean forceps for each filter type and each sample, remove the filters one at a time. The first filter removed is the Teflon filter; it is placed in the white T fraction label bottle. The second filter removed is the nylon filter; it is placed in the orange-label N fraction bottle. The last two filters are the cellulose filters, both filters are placed in one bottle with a blue W fraction label.
- 5.2.9 During the unpacking process, observe the filters for possible filter, or filter pack problems. Possible problems include the following: torn/ripped filters, excessively dirty filters, extremely wet filters, raised grids, bugs or anything else that is not normally observed. Record on the label comparison page any observations noted, using the CASTNET Comment Codes (Attachment B) as a guide.
- 5.2.10 Place all filter parts and used forceps in a pan to be washed.
- 5.2.11 Put caps securely on the bottles and place samples in the appropriate sample box in the cold room.
- 5.2.12 After the unpacking is completed, the Label Comparison page and SSRF are given to the sample log-in technician.

### **5.3 Sample Log In:**

- 5.3.1 Scanning arriving samples into Element (LIMS).  
5.3.1.1 Select *Sample Control*, select *Custody*

- 5.3.1.2 Click on **Container ID**, hit spacebar to clear
  - 5.3.1.3 Begin scanning barcode **S** labels of sample arrivals
  - 5.3.1.4 When finished scanning last sample of each received date, right click, **Select All**, right click, select **Edit Internal Custody**; pick from the drop-down the **Custody Date**, from the drop-down change **Disposition** to **Available (In)**, pick from the drop-down initials of who **Checked In/Out**, click **Apply**.
  - 5.3.1.5 Right click, select **Update Pending**. **Status** of the samples will change to **Received**.
- 5.3.2 Update Work Order
- 5.3.2.1 Select Sample Control then Samples.
  - 5.3.2.2 The work order that was logged in will appear under Work Order. Using the Label Comparison page, select the sample to be updated.
  - 5.3.2.3 Click Edit at the bottom of the screen.
  - 5.3.2.4 On the Sample tab in the upper right hand corner of the screen, check the sampled date (see Figure 7). If different than the actual sampled date, use the drop down to access the calendar and select the correct date.

**Figure 7** Sample Tab



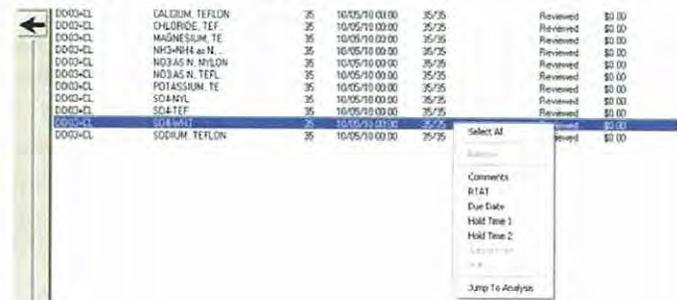
- 5.3.2.5 Go to the Details tab and check the sample Logged In date (see Figure 8). Update using the drop down calendar.

**Figure 8** Details Tab

The 'Details' tab contains the following information:

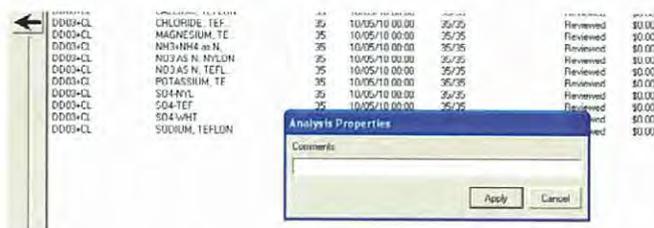
|                  |                     |
|------------------|---------------------|
| Sample Received  | Received By         |
| 08/26/10 13:00   | Helen Reed          |
| Sample Logged In | Sample Logged In By |
| 08/31/10 12:47   | RWw/yrosdick        |

**Figure 9** Comments Menu



- 5.3.2.6 Check Received By and update by using the drop down and selecting the correct name.
- 5.3.2.7 Check Sample Logged In by and update by using the drop down and selecting the correct name.
- 5.3.2.8 For each succeeding sample, highlight and roll the center of the mouse to enter the correct information.
- 5.3.2.9 To enter filter pack comment codes, select the filter type and hold down the CTRL key until all have been highlighted. Right click and select **Comment** from the list (see Figure 9). “Analysis Properties” pop-up will appear (see Figure 10). Type in the correct code(s) from the Label Comparison page and click Apply then Save.

**Figure 10** Analysis Properties



- 5.3.3 When all samples have been updated, select **Done**.
- 5.3.4 Print Custody Log.
  - 5.3.4.1 Under the **Print** tab, select **Custody Log**.
  - 5.3.4.2 Print the Internal Chains of Custody (see Figure 11) for samples just entered using **ico\_AMEC.rpt**.
- 5.3.5 The Label Comparison page is stored with the field group folder in the filter pack prep area.

**Figure 11** Internal Chain of Custody

**AMEC E&I, Inc.**  
**Internal Chain of Custody**

**1135001**

|  |                             |                              |
|--|-----------------------------|------------------------------|
| Client: Environmental Protection Agency 2011 | Received: 09/13/11 10:50:20 |                              |
| Project: CASTNET IV base period 2011         | Received By: HCR            |                              |
| Number: 6064063003                           | Temp (°C):                  | Printed: 11/3/2011 3:46:56PM |

|  |                          |                          |
|--|--------------------------|--------------------------|
| <b>1135001-01 (ABT147) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-01 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/09/11 11:12:44 by HCR |
| <b>1135001-02 (ACA416) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-02 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/12/11 11:37:48 by HCR |
| <b>1135001-03 (ALC188) Sampled 08/31/11 00:00:00</b> |                          |                          |
| <i>1135001-03 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/13/11 14:10:58 by HCR |
| <b>1135001-04 (ALH157) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-04 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/09/11 11:12:44 by HCR |
| <b>1135001-05 (ANA115) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-05 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/19/11 14:14:46 by HCR |
| <b>1135001-06 (ARE128) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-06 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/09/11 11:12:44 by HCR |
| <b>1135001-07 (ASH135) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-07 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/12/11 11:37:48 by HCR |
| <b>1135001-08 (BBE401) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-08 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/09/11 11:12:44 by HCR |
| <b>1135001-09 (BEL116) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-09 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/09/11 11:12:44 by HCR |
| <b>1135001-10 (BFT142) Sampled 08/31/11 00:00:00</b> |                          |                          |
| <i>1135001-10 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/08/11 09:18:49 by HCR |
| <b>1135001-11 (BVL130) Sampled 08/30/11 00:00:00</b> |                          |                          |
| <i>1135001-11 A [Filter Pack]</i>                    | <i>Out</i>               | <i>In</i>                |
| 6064   | 08/18/11 07:49:21 by HCR | 09/13/11 14:10:58 by HCR |

## 6.0 REFERENCES

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

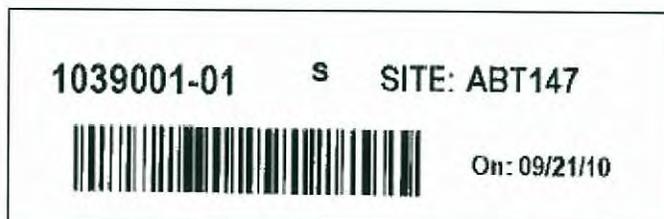
## **7.0 ATTACHMENTS**

Attachment A – Labels

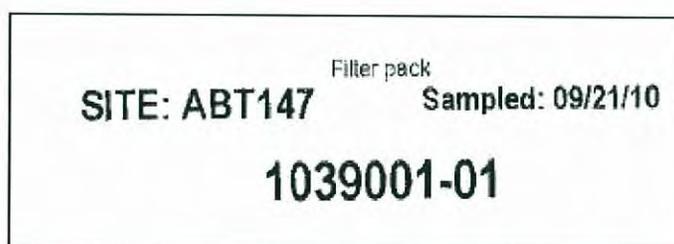
Attachment B – CASTNET Filter Pack Comment Codes

## ATTACHMENT A Labels

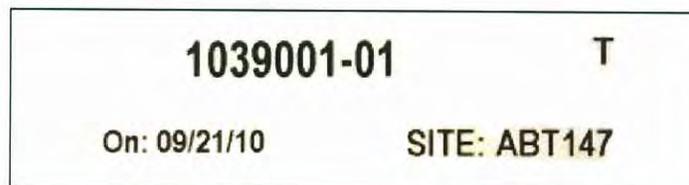
S Label



Filter Pack Label



Teflon Label



Nylon Label



Cellulose Label



## ATTACHMENT B CASTNET Filter Pack Comment Codes

- 1 – Unidentified debris or particles on filter.
- 2 – Torn, hole, or ripped filter noted during unpacking.
- 3 – Excessively wet filter noted during unpacking.
- 4 – Excessively dirty filter noted during unpacking.
- 5 – Filter pack loose upon arrival, possible leakage during sampling period.
- 6 – Apparent solenoid problem.
- 7 – Filter pack end caps cracked or missing upon receipt.
- 8 – Outside of filter pack excessively dirty upon receipt.
- 9 – Support screen raised up, noted during unpacking.
- 10 – Insect inside filter pack, noted during unpacking.
- 11 – Laboratory accident.
- 12 – Filter pack on tower incorrect length of time.
- 13 – Filter pack not run in field.
- 14 – Unusual odor noticed during unpacking.
- 15 – Low extraction efficiency off filter.
- 16 – On/off dates and times are assumed.
- 17 – Filter given to EPA for analysis.
- 18 – Field accident.
- 19 – Field equipment problem.
- 20 – Filter used for special study.
- 21 – Forest fire or agricultural activity in area.
- 22 – Site closed down.
- 23 – Site Status Report Form (SSRF) not received with filter pack.
- 24 – Chain of Custody incomplete or incorrect.
- 25 – Filter pack run on wrong week.

T – Teflon filter

N – Nylon filter

W – K<sub>2</sub>CO<sub>3</sub> impregnated filter

**TITLE: PREPARING AND SHIPPING THE CLEAN AIR STATUS AND TRENDS NETWORK (CASTNET) THREE-STAGE FILTER PACKS**

Effective Date: 11-9-11

Prepared by: Kathy W. Barry  
 Laboratory Chemist *Kathy W. Barry*

Reviewed by: Garry L. Price  
 Laboratory Operations Manager *Garry L. Price*

Reviewed by: Marcus O. Stewart  
 QA Manager *Marcus O. Stewart*

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Procedure
- 6.0 References
- 7.0 Attachments

| Annual Review |               |                 |                          |
|---------------|---------------|-----------------|--------------------------|
| Reviewed by:  | Title:        | Date:           | Signature:               |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/26/12</i> | <i>Marcus O. Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/31/14</i> | <i>Marcus O. Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/16/15</i> | <i>Marcus O. Stewart</i> |
|               |               |                 |                          |
|               |               |                 |                          |

## **PREPARING AND SHIPPING THE CLEAN AIR STATUS AND TRENDS NETWORK (CASTNET) THREE-STAGE FILTER PACKS**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to AMEC E&I, Inc. (AMEC) laboratory personnel for the preparing and shipping of three-stage filter packs and associated use of the laboratory information management system (LIMS).

### **2.0 SCOPE**

This SOP applies to all three-stage filter packs prepared for CASTNET and CASTNET-like ambient air projects.

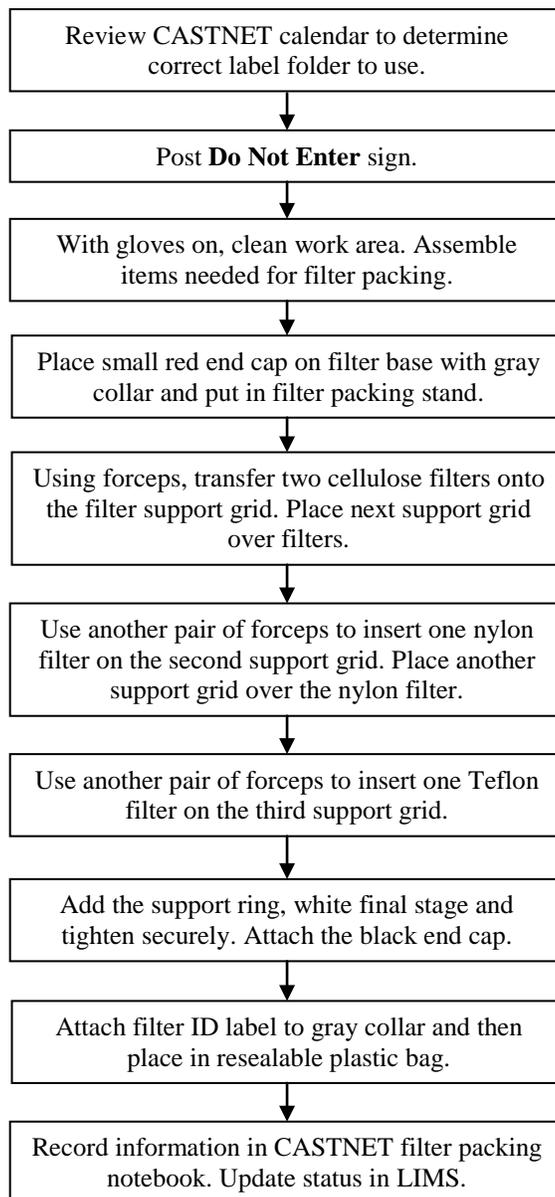
### **3.0 SUMMARY OF METHOD**

A general overview of the steps described in this SOP is depicted Figure 1. Deviations from the analytical method described in this SOP are not permitted.

### **4.0 MATERIALS**

- Powder-free gloves
- Pre-cleaned plastic forceps
- Filter packing assembly/ disassembly device
- Clean filter packs, support rings with grid, Teflon filter support ring and end caps
- Field group folder (see Attachment A):
  - Site Status Report Form (SSRF) Chain of Custody label
  - Filter pack identification (ID) label
  - Shipping Tube/FedEx Site ID Label
- CASTNET calendar
- Resealable plastic bags
- Polyvinyl chloride (PVC) and cardboard shipping tubes
- Packing notebook
- Filters:
  - Teflon filters that have passed acceptance testing
  - Nylon filters that have passed acceptance testing
  - Potassium carbonate (K<sub>2</sub>CO<sub>3</sub>)-impregnated Whatman cellulose No. 41 filters that have passed acceptance testing
- SSRF
- United States Postal Service (USPS) prepaid return label
- Kimwipes

**Figure 1** Overview – Laboratory Operations Procedures for Preparing Filter Packs



## 5.0 PROCEDURE

### 5.1 Preparing the three-stage filter pack

5.1.1 Prior to beginning filter assembly, post the Do Not Enter Sign (see Figure 2).

**Figure 2** Do Not Enter Sign



5.1.2 Review the CASTNET calendar (see Figure 3) to determine the correct field group folder to use for packing. Starting with the first Tuesday of the year, each week is assigned a CASTNET sample week number. This sample week number is used to determine the correct field group folder to use. The Laboratory Information System (LIMS) Lab ID numbers are set up with this pattern:

|           |     |   |                               |
|-----------|-----|---|-------------------------------|
| XXYY001-Z | 001 | = | CASTNET Base Program          |
|           | XX  | = | Year (last two digits)        |
|           | YY  | = | Week number(1-52) *           |
|           | Z   | = | Assigned number for each site |

\* If the calendar year starts on Wednesday through Saturday, the second calendar week will be the first CASTNET week and numbering will be 2-53.

Each field group folder contains the AMEC field log sheet, sample fraction labels, chain of custody labels, shipping/FedEx Site ID labels, and filter pack ID labels. Remove the filter pack ID labels, chain of custody labels, and shipping/contents labels from the folder and place in the designated filter packing area.

Figure 3 CASTNET Calendar

| August 2010 |           |           |           |           |           |           |
|-------------|-----------|-----------|-----------|-----------|-----------|-----------|
| Sunday      | Monday    | Tuesday   | Wednesday | Thursday  | Friday    | Saturday  |
| 1<br>213    | 2<br>214  | 3<br>215  | 4<br>216  | 5<br>217  | 6<br>218  | 7<br>219  |
| 8<br>220    | 9<br>221  | 10<br>222 | 11<br>223 | 12<br>224 | 13<br>225 | 14<br>226 |
| 15<br>227   | 16<br>228 | 17<br>229 | 18<br>230 | 19<br>231 | 20<br>232 | 21<br>233 |
| 22<br>234   | 23<br>235 | 24<br>236 | 25<br>237 | 26<br>238 | 27<br>239 | 28<br>240 |
| 29<br>241   | 30<br>242 | 31<br>243 |           |           |           |           |

CDN Sample Week Number (9:00 am local standard time) Site Operator Call In Mail Logbook Pages (from previous month)

5.1.3 Attach one chain of custody label to the SSRF in the designated location (see Figure 4).

Figure 4 SSRF with Chain of Custody Label Attached

| CASTNET   |  | SITE STATUS REPORT FORM (SSRF)  |  |
|---|--|---|--|
| <b>DAS AND OZONE</b>  |  | <b>FILTER PACK INFO</b>   |  |
| SITE NAME / NO  |  | ACTUAL SAMPLE RUN   |  |
| VISIT DATE / DAY  |  | FILTER PACK ID #  |  |
| FIELD OPERATIONS COORDINATOR CALLED:  |  | DATE  |  |
| (Name)  |  | TIME  |  |
| DAS PRIMARY BACKUP  |  | MFC (PUMP OFF)  |  |
| JULIAN DATE   |  | MFC LEAK CHECK  |  |
| TIME (2400 hr)  |  | DAS FLOW (LPM)  |  |
| DATA DOWNLOADED   |  | ROTAMETER (LPM)   |  |
| FILES BACKED UP   |  | ELAPSED TIME (HRS)  |  |
| FILE NAMES  |  | RETURN SHIPMENT PREPARED BY   |  |
| OZONE CHECKS  |  | DATE  |  |
| SAMPLE FREQUENCY  |  | EXPECTED SHIPMENT DATE  |  |
| NOISE   |  | LAB USE ONLY  |  |
| CELL PRESSURE (mmHg)  |  | LAB SAMPLE NO.  |  |
| CELL TEMPERATURE (°C)   |  | LAB TECH RECEIPT  |  |
| SAMPLE FLOW (LPM)   |  | COMMENTS:   |  |
| OUTSIDE FILTER CHANGED  |  | CHAIN-OF-CUSTODY LABEL  |  |
| INSIDE FILTER CHANGED   |  | <p>WEEK 38 2010</p> <p>Filter Pack ID #: 1039001-05 Site: ANA115</p> <p>Scheduled On Date: 09/21/2010</p> <p>SHIPMENT OPENED BY: DATE:</p> <p>PACKED BY: 3578</p> |  |
| WATER IN LINE/TAP   |  | <p>WIND DIRECTION (DEGREES)</p>   |  |
| REMOTE MODE ON  |  | <p>WIND SPEED (MPH)</p>   |  |
| PVT CONNECTION ON   |  | <p>TEMPERATURE (°C)</p>   |  |
| OFFSET OF BACKGROUND  |  | <p>BLOWER WORKING</p>   |  |
| SPAN OR COEFFICIENT   |  | <p>DELTA TEMPERATURE (°C)</p>   |  |
| <p>AUTO CAL ZERO ppb 400 ppb 90 ppb</p> <p>DATE</p> <p>DMC USE ONLY</p> <p>REVIEWED BY / ON</p> |  | <p>RELATIVE HUMIDITY (%)</p>  |  |
|   |  | <p>SOLAR RADIATION (W/M²)</p>   |  |
|   |  | <p>WETNESS SENSOR</p>   |  |
|   |  | <p>CLEAN TEST RESPONSE</p>  |  |
|   |  | <p>NOTES:</p>   |  |
|   |  | <p>RAINFALL (INCHES)</p>  |  |
|   |  | <p>CLEAN &amp; LEVEL</p>  |  |
|   |  | <p>10-TIP CHECK</p>   |  |
|   |  | <p>WIND DIRECTION (DEGREES)</p>   |  |
|   |  | <p>WIND SPEED (MPH)</p>   |  |
|   |  | <p>TEMPERATURE (°C)</p>   |  |
|   |  | <p>BLOWER WORKING</p>   |  |
|   |  | <p>DELTA TEMPERATURE (°C)</p>   |  |
|   |  | <p>RELATIVE HUMIDITY (%)</p>  |  |
|   |  | <p>SOLAR RADIATION (W/M²)</p>   |  |
|   |  | <p>WETNESS SENSOR</p>   |  |
|   |  | <p>CLEAN TEST RESPONSE</p>  |  |
|   |  | <p>NOTES:</p>   |  |

- 5.1.4 Keep forms, labels, and filter packs in order to avoid any mix-ups.
- 5.1.5 With gloves on, clean off the filter-packing stand and surrounding work area using Kimwipes. Organize all items needed around the packing area, including filters, filter packs, screens, forceps, resealable plastic bags, laboratory cart, filter pack end caps, and notebook (see Figure 5).

**Figure 5** Filter Pack Components



- 5.1.6 Verify the orientation of the Teflon filters by manually tearing the top filter in the box. Also, re-verify the Teflon orientation halfway through the packing procedure.
- 5.1.7 As each filter pack is assembled, inspect each part for physical damage that could permit air leaks (cracks, thread damage, etc.). If there is any filter part damage, send the part to the Laboratory Operations Manager.

**Note:** Pay particular attention to the filter pack outlet near the threaded quick connector. This plastic part is prone to stress fatigue damage. The damage may appear as tiny cracks. Inspect this part carefully. Discard this part at the first indication of damage or impending failure.

- 5.1.8 Begin the filter packing process by removing one filter base and gray collar from the storage container. Place a small red end cap on the base fitting and place the filter base into the filter packing stand (see Figure 6).

**Figure 6** Filter Packing Stand



- 5.1.9 Prior to placing each filter in the filter pack, inspect it for physical defects (pinholes, thin or thick areas, tears, and abnormal color) Discard any filters with defects. Care must be taken to avoid contamination during the inspection.
- 5.1.10 Using clean forceps, carefully transfer two  $K_2CO_3$ -impregnated cellulose filters into the filter support ring with grid. Place the next support ring with grid over the filters, ensuring that it has mated properly with the stage below.
- 5.1.11 Using another pair of clean forceps, insert one nylon filter on the second support grid. Place the next support grid on top, ensuring that it has mated properly with the stage below.
- 5.1.12 Using another pair of clean forceps, insert one Teflon filter on the third support ring with grid. Ensure proper orientation of the Teflon filter - Teflon side (dull side) facing up, toward the ambient air (shiny surface should be facing down). An additional support ring for the Teflon filter is then added.
- 5.1.13 Place the white final stage of the filter assembly onto the base and tighten securely.
- 5.1.14 Remove the filter pack from the filter-packing stand and attach the black end cap.
- 5.1.15 Attach the filter ID label to the gray collar. Cover the label with tape to prevent damage due to rain. Place in a resealable plastic bag.
- 5.1.16 Record the required information in the CASTNET Filter Packing Notebook (see Figure 7).

Figure 7 CASTNET Filter Pack Notebook



CASTNet Filter Pack Preparation

| Teflon | TAT Samples   | Lot Number | CASTNet Samples         |
|--------|---------------|------------|-------------------------|
|        | 1025006-21-24 | 80317500   | LB1, 1038001-01-84, LB2 |

| Nylon | NAT Samples   | Lot Number | CASTNet Samples    |
|-------|---------------|------------|--------------------|
|       | 1034005-05-08 | T93268     | LB1, 1038001-01-39 |
|       | 1034005-09-12 | T01177     | LB2, 1038001-40-84 |

| Whatman | WAT Samples   | Impreg. Date | Group | Logbook-Page | CASTNet Samples    |
|---------|---------------|--------------|-------|--------------|--------------------|
|         | 1042006-09-16 | 07-26-10     | B     | 20           | LB1, 1038001-01-55 |
|         | 1047007-01-08 | 08-13-10     | A     | 021          | LB2, 1038001-56-84 |

| LB: 10#002-01 - 02 |         |            |           |            |   |           |         |   |           |         | Week (##) 37 |
|--------------------|---------|------------|-----------|------------|---|-----------|---------|---|-----------|---------|--------------|
| Lab ID             | Site ID | ✓          | Lab ID    | Site ID    | ✓ | Lab ID    | Site ID | ✓ | Lab ID    | Site ID | ✓            |
| 10#001-01          | ABT147  | ✓          | 10#001-25 | CTH110     | ✓ | 10#001-49 | MCK131  | ✓ | 10#001-73 | STK138  | ✓            |
| 10#001-02          | ACA416  | ✓          | 10#001-26 | CVL151     | ✓ | 10#001-50 | MCK231  | ✓ | 10#001-74 | SUM156  | ✓            |
| 10#001-03          | ALC188  | ✓          | 10#001-27 | DCP114     | ✓ | 10#001-51 | MEV405  | ✓ | 10#001-75 | THR422  | ✓            |
| 10#001-04          | ALH157  | ✓          | 10#001-28 | DEN417     | 2 | 10#001-52 | MKG113  | ✓ | 10#001-76 | UVL124  | ✓            |
| 10#001-05          | ANA115  | ✓          | 10#001-29 | EGB181     | ✓ | 10#001-53 | MOR409  | ✓ | 10#001-77 | VIN140  | ✓            |
| 10#001-06          | ARE128  | ✓          | 10#001-30 | ESP127     | ✓ | 10#001-54 | OXF122  | ✓ | 10#001-78 | VOY413  | ✓            |
| 10#001-07          | ASH135  | ✓          | 10#001-31 | EVE419     | 3 | 10#001-55 | PAL190  | ✓ | 10#001-79 | VPI120  | ✓            |
| 10#001-08          | BBE401  | ✓          | 10#001-32 | GAS153     | ✓ | 10#001-56 | PAR107  | ✓ | 10#001-80 | WNC429  | ✓            |
| 10#001-09          | BEL116  | ✓          | 10#001-33 | GLR468     | ✓ | 10#001-57 | PED108  | ✓ | 10#001-81 | WSP144  | ✓            |
| 10#001-10          | BFT142  | ✓          | 10#001-34 | GRB411     | ✓ | 10#001-58 | PET427  | ✓ | 10#001-82 | WST109  | ✓            |
| 10#001-11          | BVL130  | ✓          | 10#001-35 | GRC474     | ✓ | 10#001-59 | PIN414  | ✓ | 10#001-83 | YEL408  | ✓            |
| 10#001-12          | BWR139  | ✓          | 10#001-36 | GRS420     | ✓ | 10#001-60 | PND165  | ✓ | 10#001-84 | YOS404  | ✓            |
| 10#001-13          | CAD150  | ✓          | 10#001-37 | GTH161     | ✓ | 10#001-61 | PNF126  | ✓ |           |         |              |
| 10#001-14          | CAN407  | 1          | 10#001-38 | HOW132     | ✓ | 10#001-62 | PRK134  | ✓ |           |         |              |
| 10#001-15          | CAT175  | ✓          | 10#001-39 | HOX148     | ✓ | 10#001-63 | PSU106  | ✓ |           |         |              |
| 10#001-16          | CDR119  | ✓          | 10#001-40 | HWT187     | ✓ | 10#001-64 | QAR172  | ✓ |           |         |              |
| 10#001-17          | CDZ171  | ✓          | 10#001-41 | IRL141     | ✓ | 10#001-65 | ROM206  | ✓ |           |         |              |
| 10#001-18          | CHA467  | ✓          | 10#001-42 | JOT403     | ✓ | 10#001-66 | ROM406  | ✓ |           |         |              |
| 10#001-19          | CHE185  | ✓          | 10#001-43 | KEF112     | ✓ | 10#001-67 | SAL183  | ✓ |           |         |              |
| 10#001-20          | CKT136  | ✓          | 10#001-44 | KNZ184     | ✓ | 10#001-68 | SAN189  | ✓ |           |         |              |
| 10#001-21          | CND125  | ✓          | 10#001-45 | LAY410     | ✓ | 10#001-69 | SEK430  | ✓ |           |         |              |
| 10#001-22          | CNT169  | ✓          | 10#001-46 | LRL117     | ✓ | 10#001-70 | SHN418  | ✓ |           |         |              |
| 10#001-23          | CON186  | ✓          | 10#001-47 | LYK123     | ✓ | 10#001-71 | SND152  | ✓ |           |         |              |
| 10#001-24          | COW137  | ✓          | 10#001-48 | MAC426     | ✓ | 10#001-72 | SPD111  | ✓ |           |         |              |
| 1 08-30-10         |         | 2 07-29-10 |           | 3 08-26-10 |   |           |         |   |           |         |              |

Notes:

1038

Packed By H. Reed Date: 09-02-10 Shipped via FED-EX Date: 09-02-10  
 Book # 1881 Page 037

## 5.2 Preparation of Laboratory Blanks

5.2.1 Prepare two laboratory blanks weekly for each filter type. The laboratory blank LIMS Lab ID follows the same format as the filter pack field groups:

XXYY002 - 01, 02 002 = Laboratory Blank  
XX = Year (last two digits)  
YY = Week number(1-52)  
01 = First Laboratory Blank  
02 = Second Laboratory Blank

5.2.2 Place one Teflon, one nylon, and two cellulose acceptance tested filters into the appropriate bottles labeled correctly for each filter type and the corresponding week number. Use filters from the same lots as were used in packing the filter packs for that week.

5.2.3 Record the date of preparation on the field log sheet. Record the required information in the CASTNET Filter Packing Notebook.

5.2.4 Store the bottles at 4 degrees Celsius (°C) until the samples for that week are ready for extraction.

## 5.3 Preparing the Shipping Tubes for Shipment

5.3.1 Each cardboard shipping tube must contain the PVC sleeve, filter pack, an SSRF, and a USPS prepaid return label. Foam end-pieces or bubble wrap are used to prevent damage to the filter pack and to the shipping tube. Attach the end-caps securely to both tubes to prevent the filter pack from falling out during shipment (see Figure 8).

**Figure 8** Filter Shipping Tubes



- 5.3.2 Attach the shipping tube/FedEx Site ID label to the outside of the tube. This label identifies the destination of the tube to the shipping technician. FedEx is used to ship the tubes to the site operators.
- 5.3.3 Keep the filter packs, SSRF, and shipping/contents labels in a set order to ensure that the correct filter pack is matched up with the correct SSRF and that the PVC shipping tube is sent to the correct site according to the shipping/contents label.
- 5.3.4 The date the shipment is given to the shipping technician is recorded at the bottom of the CASTNET Filter Packing Notebook.

#### 5.4 Update status of schedule samples in LIMS when shipped

- 5.4.1 Select Sample Control, select Custody
- 5.4.2 Highlight the first sample ID of the work order being shipped, hold the Shift Key and highlight the last one
- 5.4.3 Click the arrow pointing to the right moving samples to that section, right click, Select-All.
- 5.4.4 Right click, select Edit Internal Custody; pick the Custody Date shipped from the drop-down; from the drop-down change Disposition to Active (Out), pick from the drop-down the initials of shipper in Checked In/Out, click Apply.
- 5.4.5 Print Sample Custody (see Figure 9).

**Figure 9** Sample Custody

**Sample Custody**  
By Helen Reed  
Printed 09/02/10 14:34

| Lab ID       | Container   | Last Owner | Department | Location | Home Loc | Status  | Disposition  | Custody Date   |
|--------------|-------------|------------|------------|----------|----------|---------|--------------|----------------|
| 1038001-01 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-02 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-03 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-04 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-05 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-06 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-07 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-08 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-09 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-10 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-11 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-12 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-13 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-14 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-15 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-16 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-17 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-18 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-19 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-20 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |
| 1038001-21 A | Filter Pack | HCR        | 6064       |          |          | Pending | Active (Out) | 09/02/10 14:34 |

## **6.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

## **7.0 ATTACHMENTS**

Attachment A – Labels

## ATTACHMENT A Examples of Labels

### SSRF Chain of Custody Label

|                                      |                     |
|--------------------------------------|---------------------|
| <b>WEEK 38 2010</b>                  |                     |
| Filter Pack ID #: <b>1039001-01</b>  | Site: <b>ABT147</b> |
| Scheduled On Date: <b>09/21/2010</b> |                     |
| =====                                |                     |
| SHIPMENT OPENED BY:                  | DATE:               |
| -----                                |                     |
| PACKED BY: 3578                      |                     |

### Filter Pack ID Label

|                     |                          |
|---------------------|--------------------------|
| Filter pack         |                          |
| <b>SITE: ABT147</b> | <b>Sampled: 09/21/10</b> |
| <b>1039001-01</b>   |                          |

### Shipping Tube/FedEx Site ID Label

|                           |                          |
|---------------------------|--------------------------|
| <b>FEDEX-GOVT</b>         |                          |
| <b>LAB ID: 1039001-01</b> | <b>Sampled: 09/21/10</b> |
| <b>SITE: ABT147</b>       |                          |

**TITLE: PROCEDURE FOR SAMPLE INFORMATION CHANGES AND ADDITIONS**

Effective

Date: 11-4-11

Prepared by: Kathy W. Barry  
Laboratory Chemist

Kathy W Barry

Reviewed by: Garry L. Price  
Laboratory Operations Manager

Garry L Price

Reviewed by: Marcus O. Stewart  
QA Manager

Marcus O. Stewart

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Procedure
- 4.0 References

| Annual Review |        |          |                |
|---------------|--------|----------|----------------|
| Reviewed by:  | Title: | Date:    | Signature:     |
| MS            | QA Mgr | 10/26/12 | Marcus Stewart |
| MS            | QA Mgr | 10/31/14 | Marcus Stewart |
| MS            | QA Mgr | 10/16/15 | Marcus Stewart |
|               |        |          |                |
|               |        |          |                |

## **PROCEDURE FOR SAMPLE INFORMATION CHANGES AND ADDITIONS**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for changing or adding sample information in the Laboratory Information Management System (LIMS) Element by Promium.

### **2.0 SCOPE**

This SOP applies to all samples received by the Gainesville Laboratory and is used whenever changes and/or additions need to be made to existing sample information stored in the LIMS.

### **3.0 PROCEDURE**

- A request for changes and additions to sample information is initiated by an e-mail request sent to the Laboratory Operations Manager (LOM), copying the QA Manager and login technician.
- All requests must be printed, dated, and signed.
- The following items are required for each request: Project, lab identification (ID) number(s), date of request, requestor's name, and a clear description of the changes and/or additions to be made along with the reason for the request. Before changes are made, the LOM's signature is required. If the changes affect a sample already reported, the QA Manager also needs to sign.
- After the appropriate changes have been made, the request is signed and dated by the data technician.
- The request is then filed in the appropriate Project File.
- Communication of the change(s) is the responsibility of the person requesting the change(s).

### **4.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

### **5.0 ATTACHMENTS**

This SOP does not contain attachments.

**TITLE: PROCEDURE FOR DATA ENTRY ERROR CORRECTION**

Effective  
Date: 11-4-11

Prepared by: Kathy W. Barry  
Laboratory Chemist *Kathy W. Barry*

Reviewed by: Garry L. Price  
Laboratory Operations Manager *Garry L. Price*

Reviewed by: Marcus O. Stewart  
QA Manager *Marcus O. Stewart*

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Procedure
- 4.0 References
- 5.0 Attachment

| Annual Review |               |                 |                          |
|---------------|---------------|-----------------|--------------------------|
| Reviewed by:  | Title:        | Date:           | Signature:               |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/26/12</i> | <i>Marcus O. Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/31/14</i> | <i>Marcus O. Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>1/21/15</i>  | <i>Marcus O. Stewart</i> |
|               |               |                 |                          |
|               |               |                 |                          |

## **PROCEDURE FOR DATA ENTRY ERROR CORRECTION**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for correcting errors made during documentation of procedures used in sample chain-of-custody, sample records, standard and spike preparation, data transcription, data reduction, and data reporting.

### **2.0 SCOPE**

This SOP applies to all changes on data or data entries performed at the Gainesville Laboratory including studies conducted under the Good Laboratory Practice (GLP) guidelines specified in 40 Code of Federal Regulations (CFR), Parts 160 and 792.

### **3.0 PROCEDURE**

When an error is made or discovered in the documentation, the personnel making the correction shall strike a single line through the error so as not to obscure the original entry, make the correction, and initial and date the correction and include the appropriate code (see Attachment A).

### **4.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

CFR 40, Part 160, FIFRA - Good Laboratory Practice Standards (7/01/90).

CFR 40, Part 792, TSCA - Good Laboratory Practice Standards (7/01/90).

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

### **5.0 ATTACHMENTS**

Attachment A – Data Entry Error Correction Codes.

## ATTACHMENT A Data Entry Error Correction Codes

### ERROR CORRECTION CODES

These codes used on all documentation provided in this data batch.

| CODE | EXPLANATION                            |
|------|--|
| WP   | Entry made in the wrong place.         |
| WI   | Wrong information recorded.            |
| CE   | Calculation error.                     |
| SP   | Spelling error.                        |
| IW   | Illegible handwriting.                 |
| FO   | Data changed with further observation. |
| TE   | Transcription error.                   |
| FE   | Further explanation provided.          |

**TITLE: DEIONIZED WATER QUALITY**

Effective

Date: 11-9-11

Prepared by: Kathy W. Barry  
 Laboratory Chemist

Kathy W Barry

Reviewed by: Garry L. Price  
 Laboratory Operations Manager

Garry L Price

Reviewed by: Marcus O. Stewart  
 QA Manager

Marcus O. Stewart

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Summary of Method
- 4.0 Materials
- 5.0 Safety
- 6.0 Procedure
- 7.0 References
- 8.0 Attachments

| Annual Review |        |          |                       |
|---------------|--------|----------|-----------------------|
| Reviewed by:  | Title: | Date:    | Signature:            |
| MS            | QA Mgr | 10/26/12 | <u>Marcus Stewart</u> |
| MS            | QA Mgr | 10/31/14 | <u>Marcus Stewart</u> |
| MS            | QA Mgr | 10/16/15 | <u>Marcus Stewart</u> |
|               |        |          |                       |
|               |        |          |                       |

## DEIONIZED WATER QUALITY

### 1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide assurance that the AMEC E&I, Inc. (AMEC) laboratory deionized (DI) water system is regularly monitored, and the quality of water produced meets the required specifications.

### 2.0 SCOPE

This SOP applies to the DI water system located adjacent to the analytical laboratory building of the Gainesville AMEC campus.

### 3.0 SUMMARY OF METHOD

The DI system is checked on weekdays (excluding holidays) to insure that the quality lights are on and the resistivity meets criteria.

### 4.0 MATERIALS

The laboratory DI system consists of anion, cation, mixed bed and carbon tanks; a meter to monitor the output water resistivity and a meter to display the volume of water used (see Figure 1).

**Figure 1** DI Water System



## 5.0 SAFETY

No chemicals are used in this method; however, laboratory personnel should exercise caution when stepping up on the platform to take the daily readings.

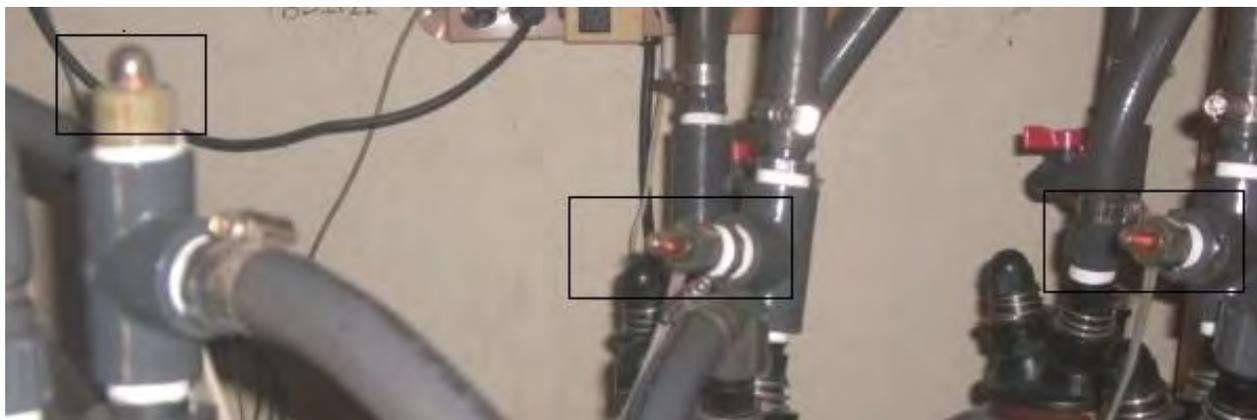
## 6.0 PROCEDURE

### 6.1 Monitoring

6.1.1 Monitoring is done on a daily basis, excluding weekends and holidays, and recorded in the DI Water System Operation Log (see Attachment A).

6.1.2 Verify that all of the tank quality lights are on (see Figure 2).

**Figure 2** Tank Quality Lights



6.1.3 Open the valve next to the water meter (see Figures 3 and 4).

**Figure 3** Valve Closed



**Figure 4** Valve Opened



6.1.4 After approximately 30 seconds, record the resistivity (see Figure 5). The resistivity should be equal to or greater than 15 megohms per centimeter (Mohm/cm). Close the valve opened in the previous step.

**Figure 5** Resistivity Meter



6.1.5 Record the quantity of DI used (see Figure 6).

**Figure 6** Water Meter



## **6.2 Quality Control**

- 6.2.1 If one or more tank lights are off, but the resistivity is acceptable ( $\geq 15$  Mohm/cm), the tanks need to be replaced but the water may still be used. The Laboratory Operations Manager (LOM) must be notified. The water system service company must be contacted and requested to have the tanks changed within the next 24 hours. The time the service company call was made must be marked in the DI Water System Operation Log. Once the tanks are changed by the water system service company, the LOM must be notified. The date and time that they were replaced must be noted in the DI Water System Operation Log.
- 6.2.2 If the tank lights are off and resistivity has dropped below the minimum specification ( $< 15$  Mohm/cm), it indicates the tanks have expired. The DI water system must be shut down and the LOM must be notified immediately. The water system service company must be contacted and requested to have the tanks changed immediately. The time of the service company call must be marked in the DI Water System Operation Log. Once the tanks have been changed by the water system company, all DI faucets in the building must be purged for a minimum of 30 seconds. Once the water system has been purged and the resistivity is greater than 15 Mohm/cm, the LOM must be notified that the water system is within criteria and can now be used. The date and time that the DI water system is operational must be noted in the DI Water System Operation Log.

## **6.3 Corrective Action**

When the resistivity falls below 15 Mohm/cm, the water system must be purged after the tanks are changed.

## **6.4 System Maintenance**

The LOM must be notified immediately of any maintenance work to be done on the water system, whether planned or unplanned. The time the system was brought down, what work was done, and the time the system was brought back up must be recorded in the operating log. When any work is performed on the system that could adversely affect water quality, the system must be purged.

## **7.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

## **8.0 ATTACHMENTS**

Attachment A – DI Water System Operation Log

## ATTACHMENT A DI WATER SYSTEM OPERATION LOG

MACTEC Engineering and Consulting  
 CASTNet Laboratory Deionized Water System Operation Log

| Equipment Check or Reading      | Monday   | Tuesday  | Wednesday | Thursday | Friday |
|---------------------------------|----------|----------|-----------|----------|--------|
| Date                            | 08-16-10 | 08-17-10 | 08-18-10  |          |        |
| Checked By                      | DCL      | DCL      | DCL       |          |        |
| Bank C/A                        | ON       | ON       | ON        |          |        |
| Mixed Bed Quality Light (Left)  | ↓        | ↓        | ↓         |          |        |
| Mixed Bed Quality Light (Right) | ↓        | ↓        | ↓         |          |        |
| Resistance Meter (≥ 15 Mohm/cm) | 17.28    | 17.25    | 17.23     |          |        |
| Water Meter (in gallons)        | 2019430  | 2019440  | 2019460   |          |        |

Any exception to the operating parameters or service performed on the system shall be noted below.

| Date | Initials | Comments |
|------|----------|----------|
|      |          |          |
|      |          |          |
|      |          |          |
|      |          |          |
|      |          |          |
|      |          |          |
|      |          |          |
|      |          |          |
|      |          |          |
|      |          |          |

**TITLE: ELEMENT® BATCH PREPARATION – DATA UPLOAD**

Effective Date: July 13<sup>th</sup>, 2015

Prepared by: Kathy W. Barry  
 Laboratory Chemist 

Reviewed by: Garry L. Price  
 Laboratory Operations Manager 

Reviewed by: Marcus O. Stewart  
 QA Manager 

**TABLE OF CONTENTS**

- 1.0 Purpose
- 2.0 Scope
- 3.0 Procedure
- 4.0 Troubleshooting and Corrective Actions
- 5.0 References
- 6.0 Attachments

| Annual Review |        |       |            |
|---------------|--------|-------|------------|
| Reviewed by:  | Title: | Date: | Signature: |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |
|               |        |       |            |

## ELEMENT<sup>®</sup> BATCH PREPARATION

### 1.0 PURPOSE

The purpose of the SOP is to provide guidance for the preparation of electronic data batches in Element DataSystem<sup>®</sup> Laboratory Information Management System (LIMS) for all laboratory methods that require uploading instrument export files.

### 2.0 SCOPE

This SOP covers the automated upload data procedures for laboratory instrumentation, troubleshooting and error correction steps.

### 3.0 PROCEDURE

#### 3.1 Prepare to upload.

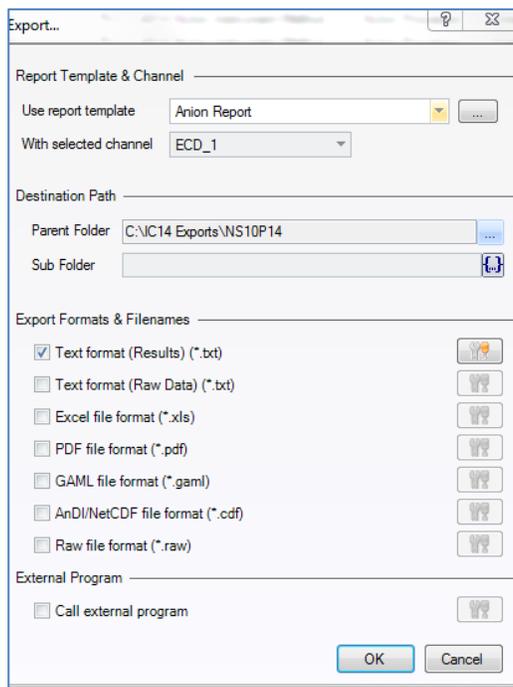
Prepare an export file. Do not include any samples requiring a dilution unless the diluted sample has been analyzed. If any required dilutions have been performed and analyzed, do not include any diluted values for samples in the automated upload file; all dilution values must be entered manually.

*Prepare export files only after the raw data has been printed and evaluated for errors, including incorrect sample laboratory IDs. Be sure to check the entire file for correct spelling and sample identification and correct any errors found before proceeding.*

##### 3.1.1 Chromeleon 7.2

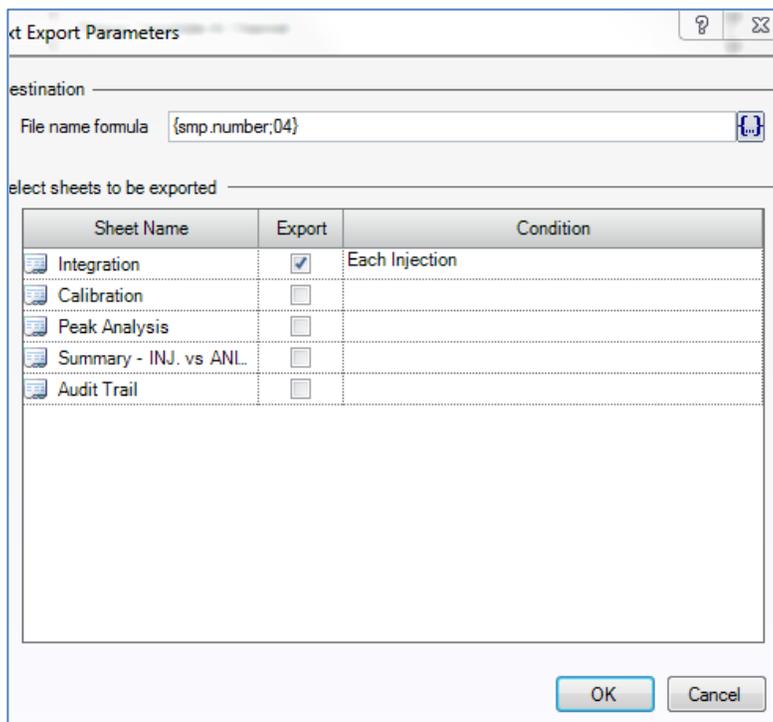
- A. On the Chromeleon console, select the **Data** section. Right click on the sequence to be printed. Select **Print**. Print the checked items only, click **OK** to print.
- B. At the bottom of the screen, open the Instrument Method. Left click on the Chromeleon icon and select **Quick Print**. Repeat this for the Processing Method.
- C. Open the **Logbook Form**. Go to the **Sequence Printout** tab and adjust the information to fit the logbook page. Note: changes are made the same as in an Excel spreadsheet.
- D. Highlight the samples for exporting. Do not include the calibration curve or shutdown method.
- E. Right click and select **Export**. The following screen (Figure 1) will open:

**Figure 1. Chromeleon 7.2 Data Export Screen - 1**



- F. The **Text format (Results) (\*.txt)** box should be the only one checked. Change the sequence ID in the **Parent Folder** section to match your sequence.
- G. Click on the tool button that is highlighted. Verify that **Integration** is the only item checked (Figure 2). Click **OK** to close. Then **OK** on the original screen.

**Figure 2. Chromeleon 7.2 Data Export Screen - 2**



- H. Click **OK** to create the export files. Each sample will have a corresponding text file and will be created in a directory with the same name as the sequence file in item F (extension.seq) located in an export folder on the instrument computer.
- I. Transfer the entire export file to an Element<sup>®</sup> - enabled workstation via rewriteable compact disc or USB thumb-drive to process your Element<sup>®</sup> AutoBatch.

### 3.1.2 Bran+Luebbe AACE 6.03

**Figure 3.** Bran+Luebbe AACE 6.03 Export Screen



- A. From the main **AACE 6.03** screen on the instrument PC select **File** and then choose **Export to** from the drop-down list. Select **ASCII File** to open the **Select Run** screen.
- B. On the **Select Run** screen, select the following data path: **C:\Program Files\Bran+Luebbe\Aace 6.03\Data\NH3+NH4.**

- C. Select the appropriate **.run** file on the right side of the **Select Run** screen and click **OK** to open the **Configure Export** screen.\

*It should be noted that the following instructions are only required if the software has not been used for an export prior to this. An example would be a new software installation. If the previous analysis has been exported, the default settings may be used.*

- D. On the **Configure Export** screen, click the **Export** tab. The following information should be selected as indicated by a check mark next to each option (see Figure 3):

|                      |             |                         |
|----------------------|-------------|-------------------------|
| Export Header Design | Method      | Export Data Fields      |
| Analysis             | Unit        | Use Real Channel Format |
| Run Name             | Base + Gain | Result                  |
| Run Date             |             |                         |
| Operator             |             |                         |
| Comment              |             |                         |
| Calibrant Values     |             |                         |
| Sample ID            |             |                         |
| Peak Number          |             |                         |
| Cup Position         |             |                         |
| Cup Type             |             |                         |

- E. On this same screen click the **Format** tab and select the following by clicking on the radio button next to each option: Text Format (ASCII)

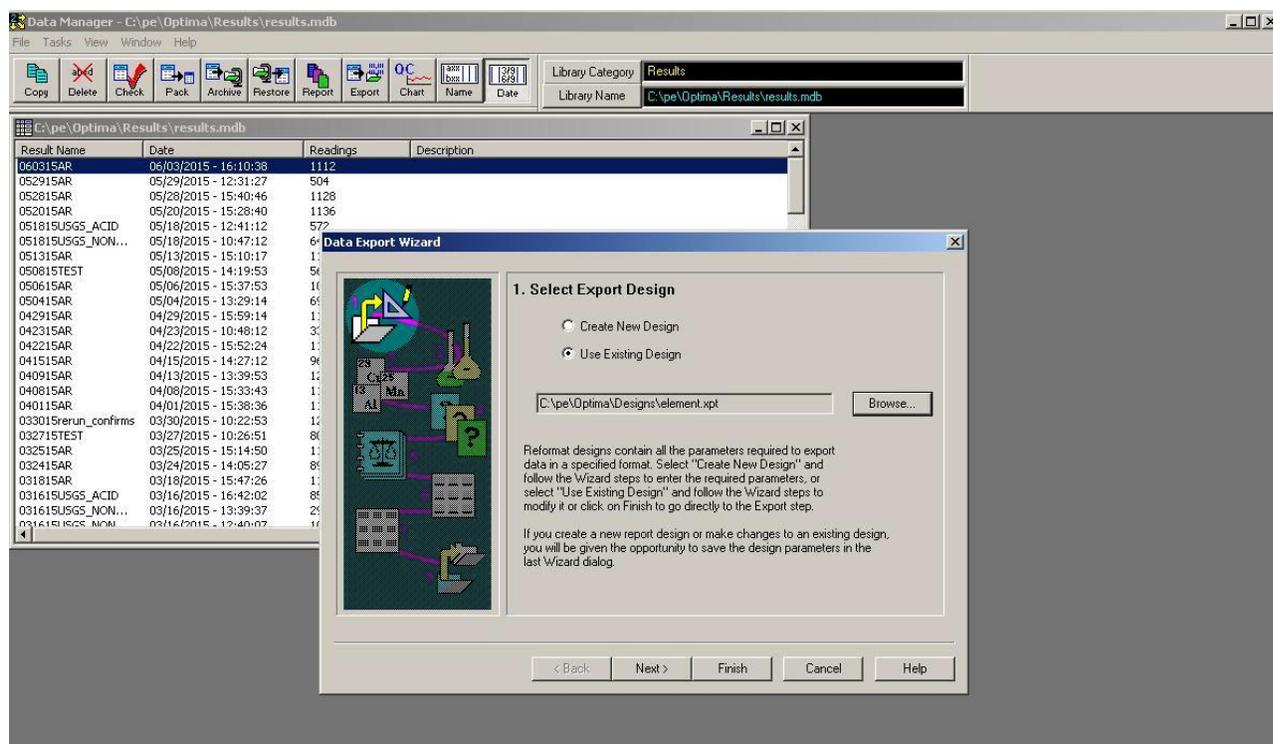
**i. Comma**

**ii. Double Inverted Comma**

- F. Click **Export** at the bottom of the **Configure Export** screen to create the export file (“**.txt**” format). The **AACE Message** window will open to indicate that the file was successfully created; click **OK**. Click **Cancel** to exit the **Configure Export** screen.
- G. The file is automatically saved to the following address: **P:\Lab\Projects\AA3\** (file name which is always the year, the month, the day, and an alpha character for discrete instrument run of the day – see Figure 3 for example – **080722A.run**)

### 3.1.3 Perkin Elmer WinLab32

**Figure 4.** Perkin Elmer WinLab32 Export Screen



- A. From the main WinLab32 screen on the instrument PC click on **File** in the upper left corner and select **Utilities** from the drop-down list.
- B. Select **Data Manager** to open the screen. Choose the results file that contains the desired data by clicking on the filename.
- C. Select **Export** to open the **Data Export Wizard** (see Figure 4). Under Select Export Design choose **Use Existing Design** and select **Browse**.
- D. Choose **element.xpt** from the pop-up window and click **Open** to return to the **Select Export Design** window. Click **Next** to open the **Select Samples to Export** screen.
- E. To select or deselect sample data files, choose **Enable/Disable** on the **Select Samples to Export** screen to open the **Enable/Disable** window. A red check mark in the Enable column indicates that a particular sample will be included in the export file. Click in the Enable column to add or remove the check marks.
- F. Click **OK** once all samples have been selected to return to the **Select Samples to Export** screen. Click on **Next** to open the **Select Analytes to Export** window.

G. Ensure that the all analytes of analysis are on the right. Select by clicking **Add** on the **Select Analytes to Export** screen. When all analytes are selected, choose **Next** to open the **Select Export Options** screen.

H. On the screen, the settings should be as follows:

Name – Same as data set file name, Extension - .prn, Directory is set to E:\ or D:\.

Use a USB thumb drive for drive **E:\** and a CDRW for drive **D:\**.

Select **Next** to proceed.

*It should be noted that the following instructions are only required if the software has not been used for an export prior. An example would be a new software installation. If the previous analysis has been exported, the default settings may be used.*

I. On the **Select Sample Parameters** screen, choose **Sample ID** by highlighting and clicking on **Add** to move the selection to the right side of the screen. Choose **Next**.

J. On the **Select Mean-Related Parameters** screen, select the following options by highlighting and clicking **Add** to move the selections to the right side of the screen:

|                                   |                             |
|-----------------------------------|-----------------------------|
| Analyte Name                      | Data and Time               |
| Concentration (Samp. Units)       | Element Symbol              |
| Element Wavelength                | Reported Conc. (Samp.Units) |
| Concentration (Calibration Units) | Units (Sample)              |
| Units (Calibration)               |                             |

Select **Next**.

K. On the **Select Replicate-Related Parameters** screen, repeat item J for the following parameters:

Replicate Number and Date and Time

Click **Next** to open the **Export Data Set** window.

L. Select **Export Data** at the top of the **Export Data Set** window to create the export file on the floppy disc. After the file is written select **Finish**.

M. Transfer the export file to an Element™-enabled workstation via floppy disk or rewriteable CDRW.

N. The export file is archived at the following address:

**P:\Lab\Projects\Optima\Results\Element**

### 3.1.4 AutoWeight V36 – See also Section 7.5.14 in GLM3180-002

**Figure 5.** AutoWeight V36 Export Screen

| Analyst           | Units | Timestamp             | Sample ID | Filter ID | Final weight mg | Initial weight mg | Net mg | Selected?                           |
|-------------------|-------|-----------------------|-----------|-----------|-----------------|-------------------|--------|-------------------------------------|
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:05:44 AM | DI08-13-  | D08-00022 | 268.291         | 268.258           | 0.033  | <input checked="" type="checkbox"/> |
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:09:50 AM | DI08-13-  | D08-00023 | 272.027         | 271.933           | 0.094  | <input checked="" type="checkbox"/> |
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:11:57 AM | DI08-13-  | D08-00024 | 270.55          | 270.304           | 0.246  | <input checked="" type="checkbox"/> |
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:13:43 AM | DI08-13-  | D08-00025 | 275.855         | 275.704           | 0.151  | <input checked="" type="checkbox"/> |
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:15:55 AM | DI08-13-  | D08-00026 | 265.743         | 265.54            | 0.203  | <input checked="" type="checkbox"/> |
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:38:31 AM | FRM08-14- | F99-33346 | 144.424         | 144.191           | 0.233  | <input checked="" type="checkbox"/> |
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:40:33 AM | FRM08-14- | F99-33347 | 148.071         | 147.738           | 0.333  | <input checked="" type="checkbox"/> |
| Ruby W. Wyrosdick | mg    | 7/28/2008 10:42:06 AM | FRM08-14- | F99-33348 | 138.460         | 138.460           | 0.000  | <input checked="" type="checkbox"/> |

- A. From the **Options** menu in **AutoWeight** , select **Export Data** (see Figure 5).
- B. Select a starting date and an ending date. Click the **Update Report File** button.
  - 1) To edit the report, select **Review and Edit Data** from the Options form.
  - 2) Select Initial/Final and Yesterday/Today. Press **Start**.
  - 3) Edit the form. All fields except Analyst and Sample Weight are available for editing. **Save** after every change.
  - 4) Close form.
- C. Print using the **Print Form** button.
- D. Click the **Export Data** button. Enter a filename at the prompt; do not enter a file extension. The file is automatically saved to the following address:
- E. **G:\GNVCASTNET\ELEMENT\AutoWeight Export files**

## 3.2 Element<sup>®</sup> Login and Data Upload

3.2.1 Double click on the Element<sup>®</sup> icon on the pc desktop

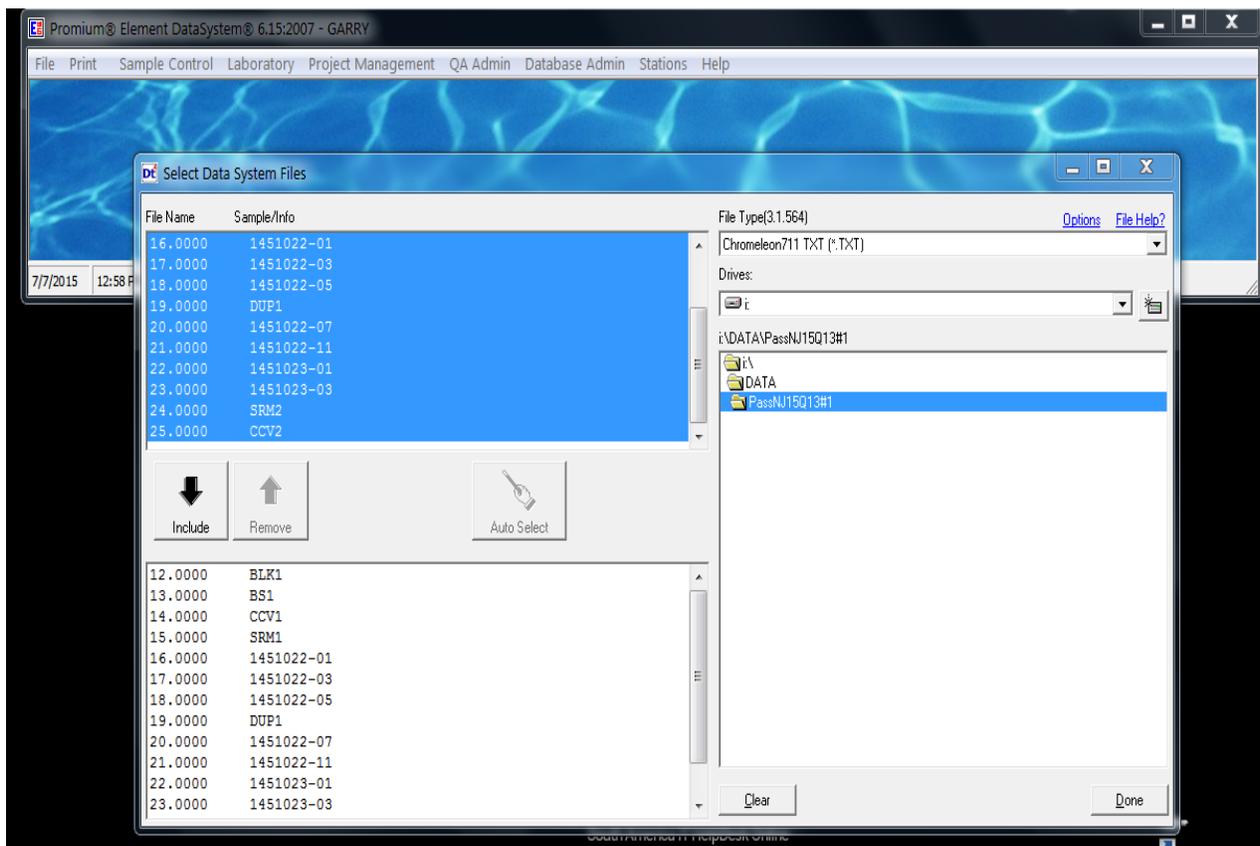
3.2.2 Enter your analyst ID and password

3.2.3 **Data Tool** – Creating a Bench Sheet

- A. From the **Laboratory** drop-down and open **Data Tool**.
- B. Clear any data visible on the left side of the **Data Tool – Main** screen by clicking on the **Clear** button for that side of the screen.
- C. Select the **Browse** button on the **right** side of the screen to open the **Select Data System Files** screen. Clear any data visible on the bottom left side of the screen by using the **Clear** button.
- D. From the drop-down list under **File Type** select as shown below:

|                         |                           |
|-------------------------|---------------------------|
| Chromleon711 (Chrom7.2) | Chromleon711 (*.txt)      |
| Bran+Luebbe AACE 6.03   | Braun + Luebbe (*.txt)    |
| Perkin Elmer WinLab32   | PE WinLab-Final (*.prn)   |
| AutoWeight v36          | Excel Spreadsheet (*.xls) |
- E. Select the appropriate drive and pathway.
- F. Choose the correct export file from those displayed in the bottom right section of the **Select Data System Files** screen by double-clicking on the filename. For Chromeleon 7.2, chose the appropriate directory. The samples in that file or directory should appear in the upper left section of the **Select Data System Files** screen.
- G. Highlight all samples to be uploaded. Do not include any calibration standards that may have inadvertently been exported with the sample data. Use the Include button to move the highlighted samples to the lower left section of the Select Data System Files screen. Verify that all desired samples have been included and any undesired samples excluded before proceeding. See an example of DataTool in Figure 6 on the following page.

**Figure 6.** Element<sup>®</sup> DataTool Screen



- H. Select **Done** to move the samples to the right side of the **Data Tool – Main** screen. If any samples appear on the left side of this screen select **Clear** at the top of the left side of the screen.
- I. Verify that the **CrossTable** displayed at the bottom of the screen corresponds to the correct instrument and/or analysis. Correct the **CrossTable** by selecting **File** followed by **Select Cross Table**. Verify that the data path is **C:\ELEMENT** before selecting the appropriate **CrossTable**.

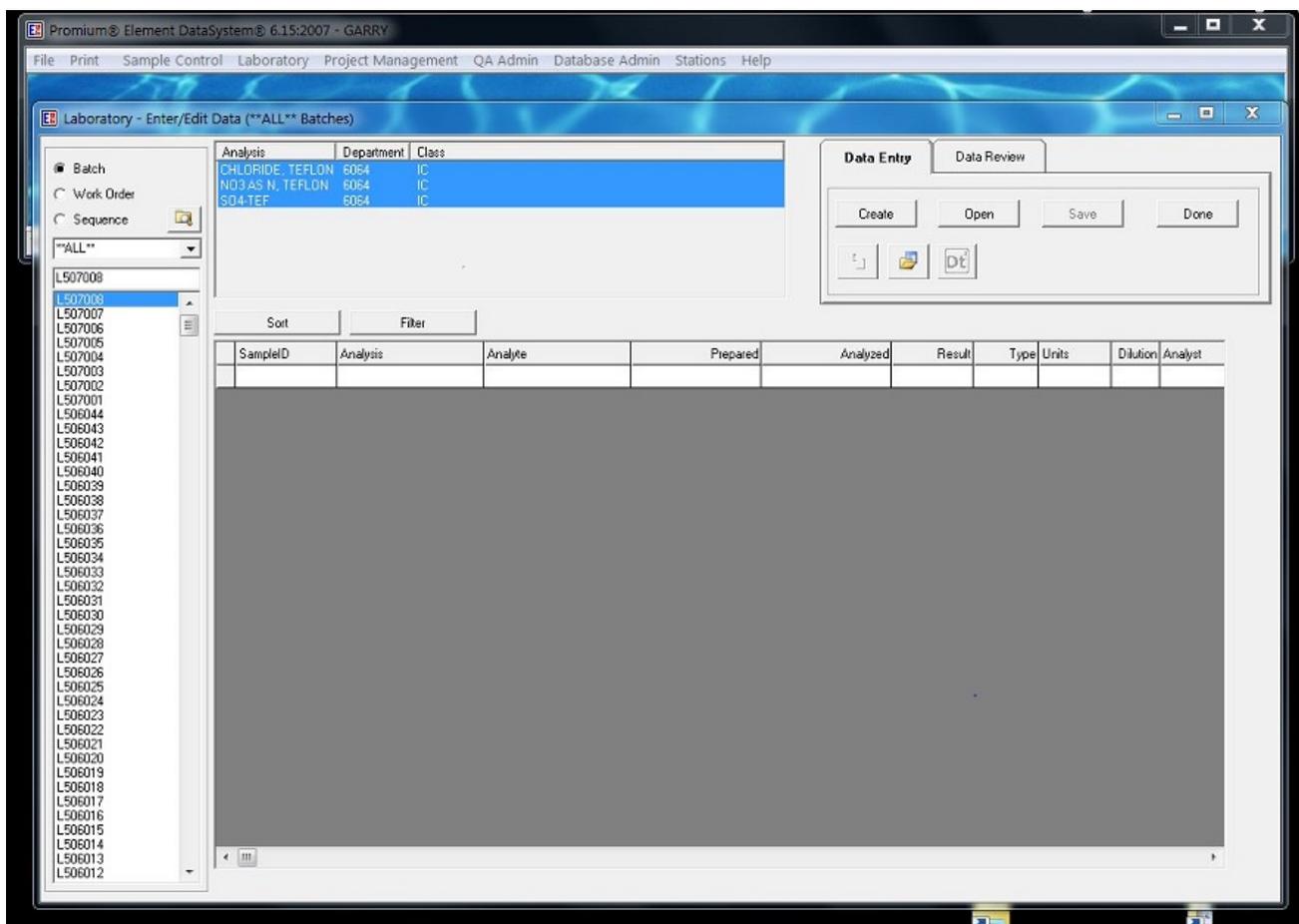
**Note:** A pop-up will open stating “The selected filename does not match the expected name. Are you sure you want to use it?” Choose **Yes**.

- J. Click **Merge Files** once the **CrossTable** is correct to bring up the **Data Transfer** screen. Select the **Instrument Data** tab to confirm that data has been entered. **Save** to the following pathway with a unique filename: **C:\ELEMENT\UserFiles**
- K. Click **Done** and close the **Data Tool – Main** screen by clicking on the [X] at the top right corner of the screen.

### 3.2.4 AutoBatch

- 3.2.4.1 Under **Laboratory**, select **Data Entry/Review**; under the **Data Entry** tab, choose the yellow folder button as shown here . Select the appropriate merged upload file saved from **Data Tool** and **Open**.

**Figure 7.** Data Entry/Review

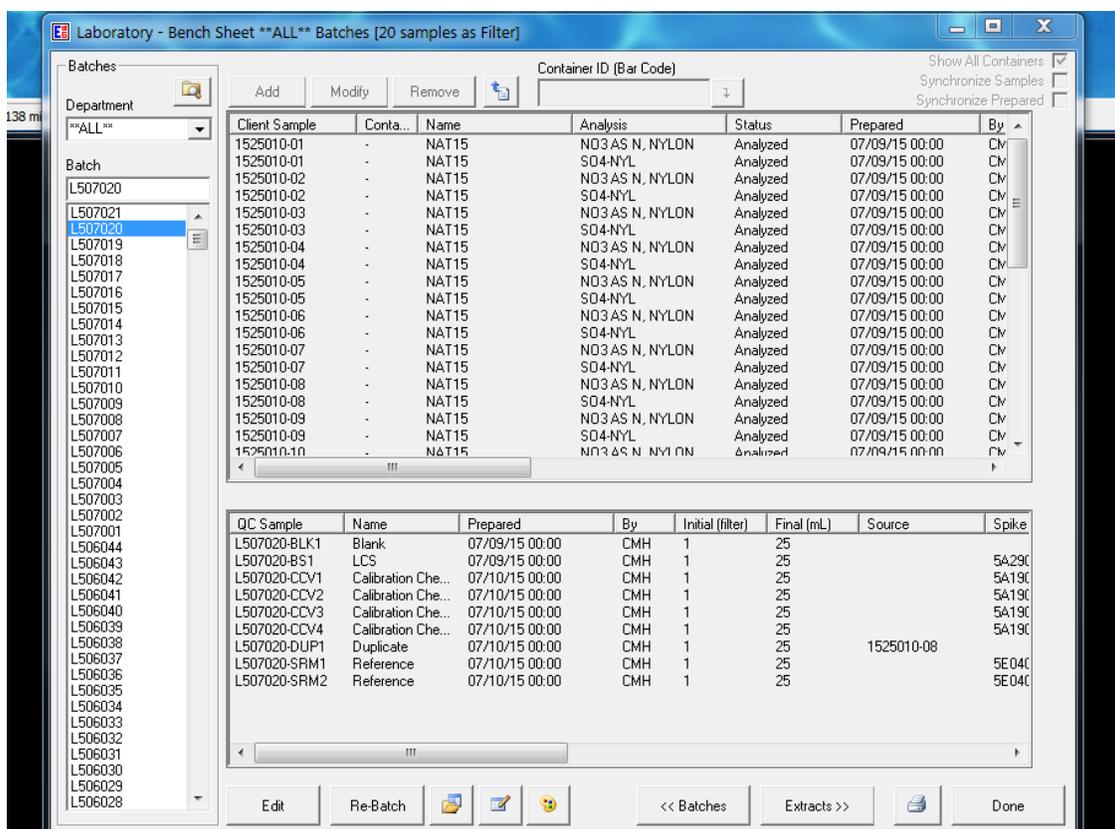


3.2.4.2 Select the appropriate parameters from the **Batch Analysis** pop-up window by clicking in the box to the left of each parameter name.

*Note: Only the parameters available for the samples included in the upload file will be available in the pop-up window.*

Close this window by clicking on the [X] at the top right corner of the window to open the **Bench Sheet**. Record the batch number.

**Figure 8.** Bench Sheet



3.2.4.3 On the **Bench Sheet**, select the **Edit** button to enter Edit Mode.

- A. In the upper window (real samples), verify that all desired samples were included. If any samples are missing, it is likely that they were misnamed. Check the Laboratory ID against the label on the extract bottle and make any necessary corrections to the raw data and upload files before proceeding.
- B. Still in the upper window, highlight a sample, right click and choose **Select All** to

highlight all samples. Right click again and select **Prepared** to correct the extraction date (if applicable). Repeat and select **Prepared By** to correct the extractor name if different than that of the analyst preparing the data batch.

- C. In the lower window (QC samples), repeat the steps in item B, correcting the **Prepared** date to the date of extraction for the method blank (**BLKI**) and any other extracted QC samples (such as **BSI**). Correct the **Prepared** date to the date of analysis for any non-extracted QC samples such as **CCV** and **SRM**.
- D. Highlight all **CCV**, right click and select **Spike 1 ID**. Select the appropriate Element<sup>®</sup> Standard assigned ID by double-clicking. Repeat right-click while all **CCV** are still highlighted and select **Spike 1 Type**. Right-click again and choose **Static** from the drop-down list. Repeat right-click again and select **Spike 1 Amount** Set this value to **1**
- H. Repeat item D for **SRM** samples.
- I. Repeat item D for **BSI** sample. Choose **Pre-Prep** under **Spike 1 Type** and either **1000** or **2000** (the later for 50 mL extracts only) for **Spike 1 Amount**.
- J. Highlight DUP1. Right click and select **Source**. Using the information from the run log, choose the correct Laboratory ID for the duplicated sample. Repeat for the remaining **DUP** samples in the batch.
- K. **Save** the changes and select **Done** to open the **Data Transfer** screen.

3.2.4.4 Any unmatched parameters appearing in red in the upper left corner of the **Data Transfer** screen must be added to the **CrossTable**.

- A. Double-click the first unmatched analyte in red to open the **CrossTable**.
- B. The **CSAnalyte** and **ELMAnalyte** columns **must** match for every entry in the table; the ELMAnalysis column should be **ignored**.
- C. To match, click in the unmatched **ELMAnalyte** column and select the correct match from the drop-down list.
- D. The **CSAnalyte** column must be either matched to the **ELMAnalyte** column or listed as **\*Non-Target\***.
- E. Verify that the upper range of the method is correct.
- F. **Save** and **Close**.

3.2.4.5 On the **Data Entry/Review** screen, select the *Analyst* column by clicking on the appropriate header tab. Right click and choose *Fill Cells*. Select the appropriate analyst initials from the drop-down list and click *Apply* and *Save*.

3.2.4.6 Click *OK* to close the **Data Post Results** pop-up window.

### **3.3 Review and Print Batch**

3.3.1 From **Laboratory** in the Element<sup>®</sup> taskbar, select *Data Entry/Review*, highlight your batch number, and under the **Data Review** tab choose *Query*.

3.3.2 Review all data for correctness. Verify that all desired samples are listed. Samples for which the database detects an error or flag will be coded in red. The parameter in question will also be coded red. Investigate all red-coded samples for corrections.

*Note: If no red-coded parameters are initially viewable, reveal all details for the samples by clicking on the grey square in the uppermost left corner of the data display to select all samples, right click, and choose “View Details.” Scroll to the right until the red-coded parameter comes into view.*

*Note: All flags and errors must be addressed in the batch checklist.*

3.3.3 After all necessary corrections have been made, select all by clicking on the grey square in the uppermost left corner of the data display. Right click and select *Update Status* from drop-down and select *Update to Analyzed*.

3.3.4 Select *Lock* to lock the data from further editing.

3.3.5 Print the batch report using the correct report format as indicated below.

IC and ICP – rev\_AMEC\_R3.rpt

AA3 – rev\_AMEC\_NH4.rpt

Mass analyses – rev\_AMEC\_PM25.rpt / rev\_AMEC\_PMHiVol.rpt

### **3.4 Standard Update**

3.4.1 Update the **Standards** table for QC used in analysis. This step only need be completed when new standard solutions or masses are being used.

3.4.1.1 Under **Laboratory** on the main Element<sup>®</sup> screen, select **Standards** to open the **Standards** table. On the left side of the table are the standard numbers assigned by Element<sup>®</sup>; on the right are the descriptions of each standard.

3.4.1.2 Choose the number for a previous standard of the type required and click **Copy** to create another entry of the same type. To create a standard from scratch click **Add** and specify the type as follows:

For stocks (purchased or laboratory prepared), purchased standards, and mass standards:

1 – Specify each analyte and its concentration.

For diluted standards:

2 – Combine and/or dilute existing standards.

3.4.1.3 **For copied standards**, update the entry to reflect the correct information for the standard.

A. For purchased stocks and standards:

- 1) The **Prepared Date** and the **Reference Date** should be changed to reflect the date the stock or standard was made. If this date is not available from the manufacturer's documentation, use the received date. For masses, use the certification date.
- 2) The **Expiration Date** should reflect the date specified by the Certificate of Analysis. If no expiration date is given, the solution expires one year from the date of receipt. For masses, the **Expiration Date** is one year from the date of certification.
- 3) Select the radio button next to **Purchased** and enter the Received date and **Opened date** in the appropriate fields.
- 4) Update **Prepared By** with **\*\*Vendor\*\*** and **Vendor** to the correct manufacturer source.
- 5) Update the lot number both in the **Vendor Lot** field and in the **Comment** field.

B. For laboratory prepared stocks:

- 1) The **Prepared Date** and the **Reference Date** should be changed to reflect the date the stock was made.
- 2) The **Expiration Date** is one year from the date the stock was prepared.
- 3) Select the **Prepared** radio button.
- 4) Update the **Vendor Lot** field and in the **Comment** field with the solution number.

Include a specific reference to the logbook and page number in the Comment field.

***Note: Each laboratory prepared standard should be documented in an appropriate logbook and assigned a unique solution number that references both the logbook and page number. For example, 180701IA references the first solution documented in section I of page 01 in logbook 1807. For logbooks designed to document only solution per page, the logbook number followed by the page number should be sufficient, eg. 1857-01.***

C. For diluted standard solutions:

- 1) The ***Prepared Date*** should be changed to reflect the date the diluted standard was made.
- 2) The ***Reference Date*** should be changed to reflect the date the ***stock*** was made. If no prepared date was supplied with a purchased stock, use the receipt date.
- 3) The ***Expiration Date*** is assigned as follows:

|                       |                                 |
|-----------------------|---------------------------------|
| ICP standards:        | 1 month from date of dilution.  |
| Most other standards: | 3 months from date of dilution. |
| High-level standards: | 6 months from date of dilution. |
- 4) Select the ***Prepared*** radio button.
- 5) Update the ***Vendor Lot*** field and in the ***Comment*** field with the ***stock*** lot number for standards diluted from purchased stocks or the ***diluted standard*** solution number for standards diluted from prepared stocks. Include a specific reference to the logbook and page number in the Comment field.
- 6) In the left-hand portion of the screen below the Comment field, right click on the displayed Element<sup>®</sup> standard number and select ***Browse*** to open the list of all Element<sup>®</sup> assigned standard ID numbers. Select the standard number of the stock solution used for the dilution.

***Note: Only the information in the Comment field is visible when selecting the correct “Spike 1 ID” in the Bench Sheet. Any information necessary to identify the correct standard – such as lot number, expiration date, and/or preparation date – should be included in this field.***

3.4.1.4 **For newly created standards**, update the entry to reflect the correct information for the standard.

A. For purchased stocks and standards or standard masses (Type 1):

- 1) Edit the dates as in Section 3.5.1.3.1 – 3. 5.1.3.2.
- 2) Select the **Vendor** from the drop-down list or enter an unlisted vendor by hand. Enter the **Vendor Lot** number.
- 3) Select the radio button next to **Purchased** and enter the **Received date** and **Opened date** in the appropriate fields.
- 4) Enter the **Volume** of the standard and the **Concentration Units**. Set the Concentration Units for masses to **NA**. The **Solvent Lot#** is also **NA** for masses.
- 5) Select the analytes. Highlight the desired parameters in the left-hand portion of the screen below the **Comment** field and click the “→” button to move the selected analyte to form an analyte table on the right side of the screen.
- 6) Enter the targets for each analyte in the column next to the parameter name. This column will be named based on the **Concentration Units**. Verify that the correct control limits have been entered.
- 7) Select the radio button next to **Reference** under **Standard Type**. Choose **Other** for mass standards.

B. For laboratory prepared stocks (Type 1):

- 1) Edit the dates as in Sections 3.5.1.3.3.
- 2) Select the radio button next to **Prepared**.
- 3) Update the **Vendor Lot** field and in the **Comment** field with the solution number. Include a specific reference to the logbook and page number in the **Comment** field.
- 4) Follow Sections 3.1.3.4.1.4 – 3.1.3.4.1.7.

C. For diluted standard solutions (Type 2):

- 1) Edit the dates as in Sections 3.1.3.3.3.1 – 3.1.3.3.3.2.
- 2) Select the radio button next to **Prepared**.
- 3) Update the **Vendor Lot** field and in the **Comment** field with the **stock** lot number for standards diluted from purchased stocks or the **diluted standard** solution number for standards diluted from prepared stocks. Include a specific reference to the

logbook and page number in the *Comment* field.

- 4) In the left-hand portion of the screen below the *Comment* field, right click on the displayed Element<sup>®</sup> standard number and select *Browse* to open the list of all Element<sup>®</sup> assigned standard ID numbers. Select the standard number of the stock solution used for the dilution.
- 5) Select the radio button next to *Reference* under *Standard Type*.

3.4.1.5 *Save* the changes.

#### 4.0 TROUBLESHOOTING AND CORRECTIVE ACTIONS

Most Element<sup>®</sup> errors occur because of misidentified samples. The Element<sup>®</sup> Laboratory IDs are purely numeric and can be *very* similar for some samples. Careful attention is required at every stage of analysis to ensure that each sample is correctly identified. It is strongly recommended that instrument schedules be prepared directly from the samples and not from another schedule or list as this creates an opportunity for errors to be introduced in manner that is extremely difficult to detect.

- 4.1 Before submitting the batch, verify that every sample included in the export file has been uploaded into Element<sup>®</sup>. If a sample field or QC is missing, check the instrument schedule or run log for obvious errors, such as alphabetical characters interchanged with numerals. If the schedule appears correct, check the export file for inadvertently excluded samples. If this is correct, verify the schedule against the sample bottle labels.
- 4.2 If the missing sample is a misidentified QC sample, follow the procedure in Section 4.3, but select the appropriate QC sample type instead of *Client Sample (Container)* as specified in Section 4.3.3. See Section 3.3.2.2 for a list of the selection choices for each QC sample type.

*Note: Most often a sample will be misidentified as a sample for which the data has already been locked in Element<sup>®</sup>. In this case a pop-up window will appear during upload indicating that a sample already has data entered for the parameters included in the batch. The sample will not be uploaded into Element<sup>®</sup>. The specific sample affected is not named and must be identified by comparing the uploaded samples with the instrument run log.*

**4.3** If a misidentified sample is indicated, correct the export file and add it to the data batch.

4.3.1 Open the export file and make the appropriate correction, making sure to save the file. Alternatively, the export file can be recreated after the sample identification has been corrected.

**Note: The raw data, both electronic (where possible) and the hard-copies must be corrected before the batch can be submitted.**

4.3.2 Open the **Laboratory** tab and select **Data Entry/Review**. Select the correct batch number and **Query**. **Make sure all data are locked**. See Section 3.4.4. If data are left unlocked it could be overwritten and additional compounding errors introduced.

4.3.3 Open the **Bench Sheet** and select the appropriate batch. Enter **Edit Mode**. Highlight any sample that should **not** be in the batch and click **Remove**. Click **Add** to and select **Client Sample (Container)** to bring up a list of available samples. Double-click the appropriate sample and analysis to add it to the bench sheet. Close the window by clicking on the [X] in the upper right corner of the screen.

4.3.4 Highlight the added sample and right-click. Select **Prepared** and change the prepared date to match the rest of the samples in the batch. **Save** and **Done** to exit the **Bench Sheet**.

4.3.5 Open **Laboratory** tab and select **Data Entry/Review**; select the appropriate batch. Under the **Data Entry** tab, click **Create** and then **Export** as an Excel file. Assign the file a unique name that is different from the original export file for this batch but is related. Save to the following directory: **C:\ELEMENT\UserFiles**. Click **Cancel** and close the screen.

4.3.6 Open **Data Tool** through Element<sup>®</sup>. **Clear** the left side of the screen of samples. Click **Browse** on the **left side** of the **Data Tool – Main** window. Open the Excel file created in Section 4.3.5 so that the samples are listed in the left pan of the window.

4.3.7 Click **Browse** on the **right side** of the window. **Clear** any existing samples displayed on the **Select Data System Files** screen. Select the original export file so that the samples are displayed in the upper left portion of the screen. Highlight the desired sample and

move it to the lower left portion of the screen using the ↓/Include button. Click **Done** to return to the **Data Tool – Main** screen.

- 4.3.8 Click **Merge Files**. Click the Instrument tab on the **Data Transfer** screen to display the instrument data.
- 4.3.9 **Save** as an Excel file to the same destination as in Section 4.3.5 with a new related filename.
- 4.3.10 Open the **Laboratory** tab and select **Data Entry/Review** screen and select the correct batch. Click **Open** and select the file saved in Section 4.3.9. **Save** and **Query** to review. In cases where data have not been updated to *Reviewed* status, it is currently possible to overwrite data for batched samples *even if the data have been locked*. When this happens, the older sample will be deleted from its original batch. To correct this problem it is therefore also necessary to add the erased sample back into its original batch as well as correct the misidentified sample in the current batch.
- 4.3.11 If a misidentified sample has resulted in data from previous batches being erased, delete the offending sample from the most recent batch from the Bench Sheet.
- 4.3.12 Replace the deleted sample in its original batch following the steps outlined in Sections 4.3.2 – 4.3.10. Resubmit the batch including a Batch Update Request Form. No status change is required. See Attachment C.
- 4.3.13 Repeat the entirety of Section 4.3 for the most recent batch.

- 4.4** If a sample in the current batch is correctly identified but is excluded from the upload because a sample in a previous batch has been misidentified with the same Laboratory ID, the data in the older batch must be corrected before the current sample can be uploaded properly.
- 4.4.1 Determine the original batch's number and submit a Batch Update Request Form to have the status changed back to "Analyzed" by the Laboratory Manager.
- 4.4.2 Using the archived data files, follow the steps in Section 4.3 for the original batch and resubmit the batch, including the Batch Update Request Form.
- 4.4.3 Re-upload the current batch.
- 4.4.4 Using the archived data files, follow the steps in Section 4.3 for the original batch and resubmit the batch, including the Batch Update Request Form.
- 4.4.5 Re-upload the current batch.

## **5.0 REFERENCES**

AMEC Environment & Infrastructure, Inc. (AMEC). 2014. Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan (QAPP) Revision 8.2. Prepared for U.S. Environmental Protection Agency (EPA), Office of Air and Radiation, Clean Air Markets Division, Washington, DC. Contract No. EP-W-09-028. Gainesville, FL. <http://java.epa.gov/castnet/documents.do>.

U.S. Environmental Protection Agency (EPA). 2007. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/B-07/001, April.

## **6.0 ATTACHMENTS**

Attachment A – General Batch Narrative Format.

Attachment B – Mass Analyses Batch Format.

Attachment C – Example Batch Update Request Form.

Attachment D – Revisions Table

**ATTACHMENT A**  
**General Batch Narrative Format**

Batch Comments      L802041                      02/21/08

1. Comment 1.

2. Comment 2.

|          |       |      |       |
|----------|-------|------|-------|
| Analyst  | _____ | Date | _____ |
| Reviewer | _____ | Date | _____ |
|          | _____ |      | _____ |

**ATTACHMENT B**  
**Mass Analyses Batch Format**  
(See also Attachment E of GLM3180-002 for PM<sub>2.5</sub> analyses)

Batch: \_\_\_\_\_ Date: \_\_\_\_\_

Room Temp.: \_\_\_\_\_ Humidity: \_\_\_\_\_

Please note the following observations for filters in this batch:

Sample Name (Filter #) Code and description (repeat this information for all samples identified with a code).

Note: The following filters are field blanks:

Sample Name (Filter #), ....

Analyst Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewer Signatures: \_\_\_\_\_ Date: \_\_\_\_\_  
\_\_\_\_\_ Date: \_\_\_\_\_  
\_\_\_\_\_ Date: \_\_\_\_\_

## ATTACHMENT C

### Example Batch Update Request Form



#### Data Batch Update Request

|          |     |       |
|----------|-----|-------|
| Batch #: | By: | Date: |
|----------|-----|-------|

|                        |
|------------------------|
| Request Justification: |
|------------------------|

|                      |       |       |       |
|----------------------|-------|-------|-------|
| Status Changed By:   | _____ | Date: | _____ |
| Changes Made By:     | _____ | Date: | _____ |
| Updates Reviewed By: | _____ | Date: | _____ |
|                      | _____ | Date: | _____ |
| Status Changed By:   | _____ | Date: | _____ |

**ATTACHMENT D**  
**Revisions Table**

| <b>Document Revision No.</b> | <b>Section</b>                        | <b>Revision</b>   |
|------------------------------|---------------------------------------|---|
| 4.0                          | <del>Appendix 1</del><br>Attachment D | Addition of this table  |
|                              | Figure 2                              | Updated Dionex Chromeleon 6.8 Data Export Screenshot  |
|                              | 3.1.1.5                               | Text changed to:<br>G:\GNVCASTNET\ELEMENT\AutoWeight Export files   |
|                              | 3.2                                   | (* .csv) changed to (*filename Dtool.xls)   |
|                              | 3.4.5.1                               | Lowercase amec changed to AMEC  |
|                              | 3.4.5.2                               | Lowercase amec changed to AMEC  |
|                              | 3.4.5.3                               | Lowercase amec changed to AMEC  |
|                              | 3.4.5.4                               | Lowercase amec changed to AMEC  |
| 5.0                          | Attachment D                          | Corrected reference in revisions table row 1.   |
|                              | All                                   | Chromeleon 6.8 references updated to 7.2 except where references are to external processor function names instead of the actual software. |
|                              | 3.1.1.1                               | The section referencing PeakNet 5.1 was replaced by Chromeleon 7.2.   |
| 6.0                          | 3.3                                   | Manual Entry batches was removed and a separate SOP for manual entry Element® created   |
|                              |                                       | Figures 6, 7, and 8 added   |
|                              | 5.0                                   | Section 5 updated with the latest QAPP reference  |
|                              | Attachment C                          | Example Batch Update Request Form updated   |

## PROCEDURE FOR GENERATION OF CONTROL CHARTS

Effective

Date: 11-10-11

Prepared by: Kathy W. Barry  
 Laboratory Chemist Kathy W Barry

Reviewed by: Garry L. Price  
 Laboratory Operations Manager Garry Price

Reviewed by: Marcus O. Stewart  
 QA Manager Marcus Stewart

### TABLE OF CONTENTS

- 1.0 Purpose
- 2.0 Scope
- 3.0 Procedure
- 4.0 References
- 5.0 Attachments

| Annual Review |        |          |                |
|---------------|--------|----------|----------------|
| Reviewed by:  | Title: | Date:    | Signature:     |
| MS            | QA Mgr | 10/26/11 | Marcus Stewart |
| MS            | QA Mgr | 10/31/11 | Marcus Stewart |
| MS            | QA Mgr | 10/1/11  | Marcus Stewart |
|               |        |          |                |
|               |        |          |                |

## **PROCEDURE FOR GENERATION OF CONTROL CHARTS**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for generation of control charts for CASTNET and CASTNET-like projects.

### **2.0 SCOPE**

This SOP applies to Laboratory Control Samples (LCS), Continuing Calibration Standards (CCV) and Standard Reference Materials (SRM) analyzed at the Gainesville Laboratory. The charts generated will be used as a tool to assist in detection of trends, bias or other problems at the bench.

### **3.0 PROCEDURE**

#### **3.1 Chart generation**

Charts will be generated quarterly by the 15<sup>th</sup> of the month following the end of the previous quarter using data in the Laboratory Information Management System (LIMS). For example, the fourth quarter of 2010 will be generated by 1/15/2011. New charts will start where the previous set ended.

- 3.1.1 Log into Element and access “Control Charts” under QA Admin (see Figure 1).
- 3.1.2 Select the appropriate analyte from the list on the left side of the screen.
- 3.1.3 Set the dates to cover a three month range starting with the day after the end of the last set generated.
- 3.1.4 Select “All” for Data Points.
- 3.1.5 Query.
- 3.1.6 Right click on the graph and print.
- 3.1.7 LCS %R, SRM %R and CCV %R are printed for each analyte.

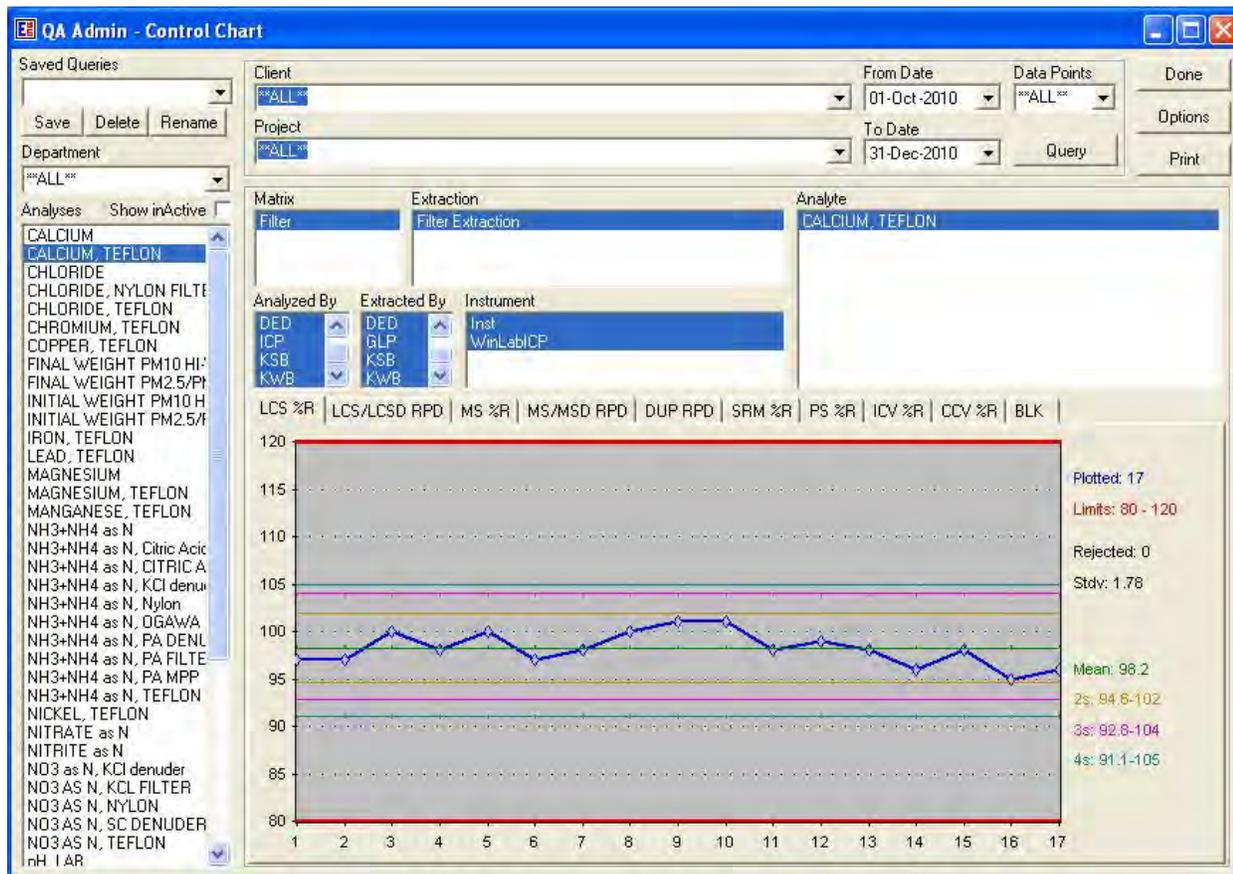
*Note: Moving averages will not be calculated and limits will not be continually adjusted.*

#### **3.2 Analyst review**

Copies of the charts will be maintained in each laboratory. The analyst will compare batch results with charts to check for situations including, but not limited to:

- Excursions beyond three standard deviations from the mean;
- Five or more consecutive points increasing or decreasing;
- Step function changes (see Attachments C & D for examples).

**FIGURE 1** Control Chart Screen in Element



### 3.3 Corrective actions

Suspect results will be reported to the Laboratory Operations Manager (LOM). Justification will be documented in the data batch comments as per the LOM's direction.

### 4.0 REFERENCES

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

## **5.0 ATTACHMENTS**

Attachment A – SO<sup>4</sup> Cellulose CCV

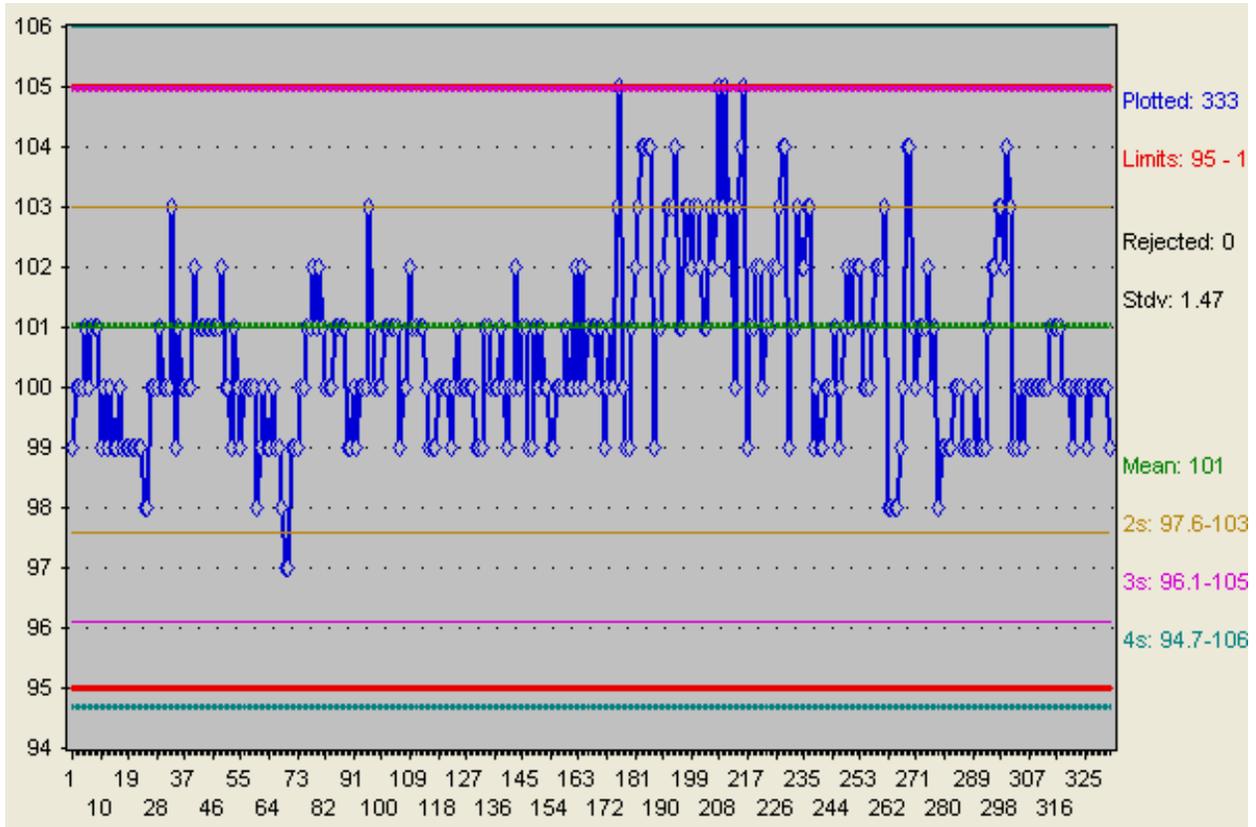
Attachment B – SO<sup>4</sup> Cellulose LCS

Attachment C – SO<sup>4</sup> Cellulose SRM

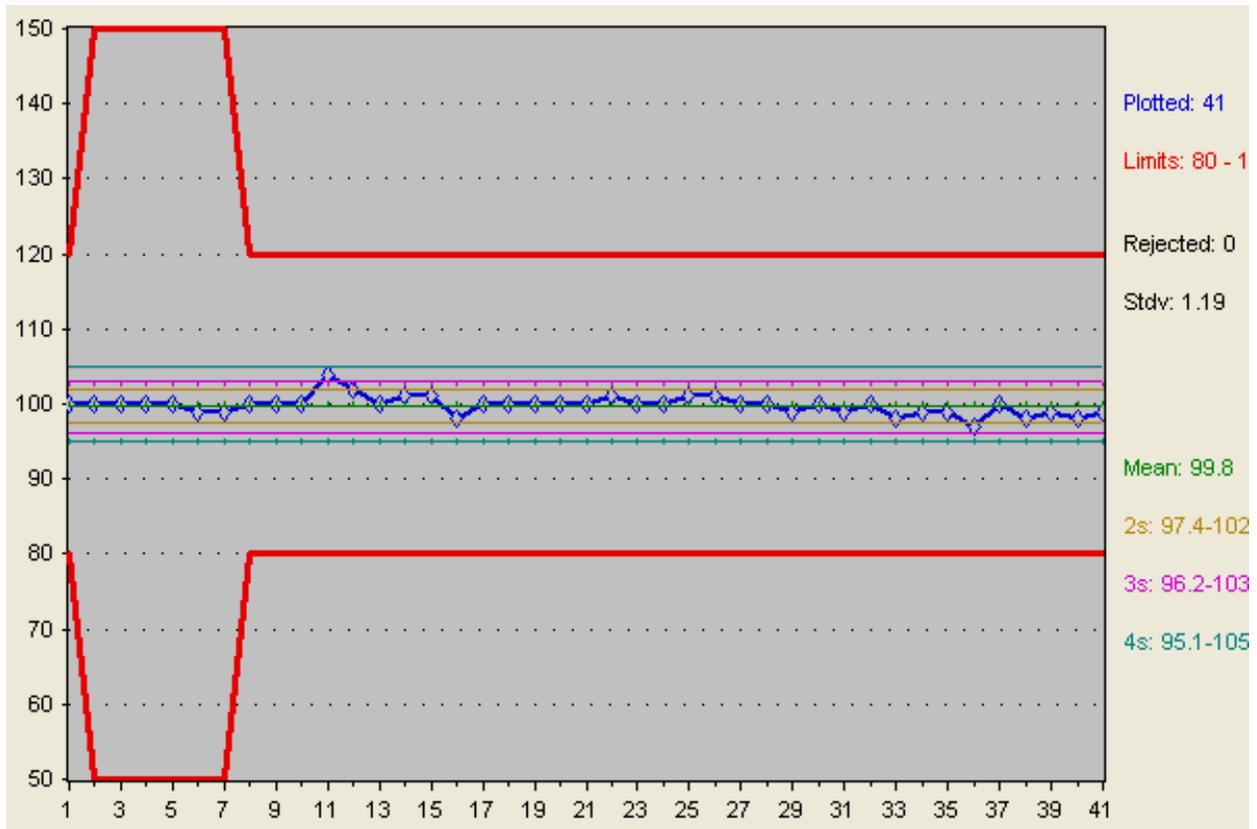
Attachment D – NO<sup>3</sup> as N Teflon SRM

### ATTACHMENT A

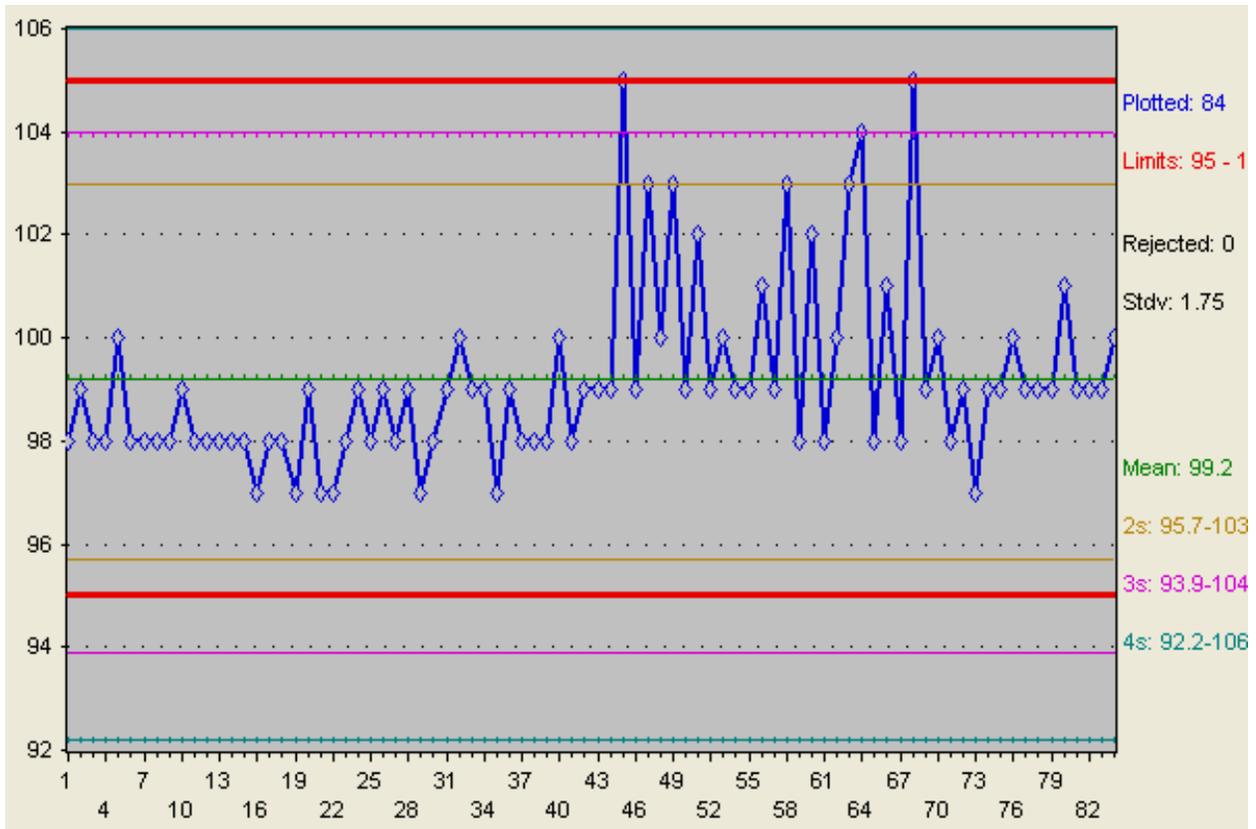
#### SO<sup>4</sup> Cellulose CCV



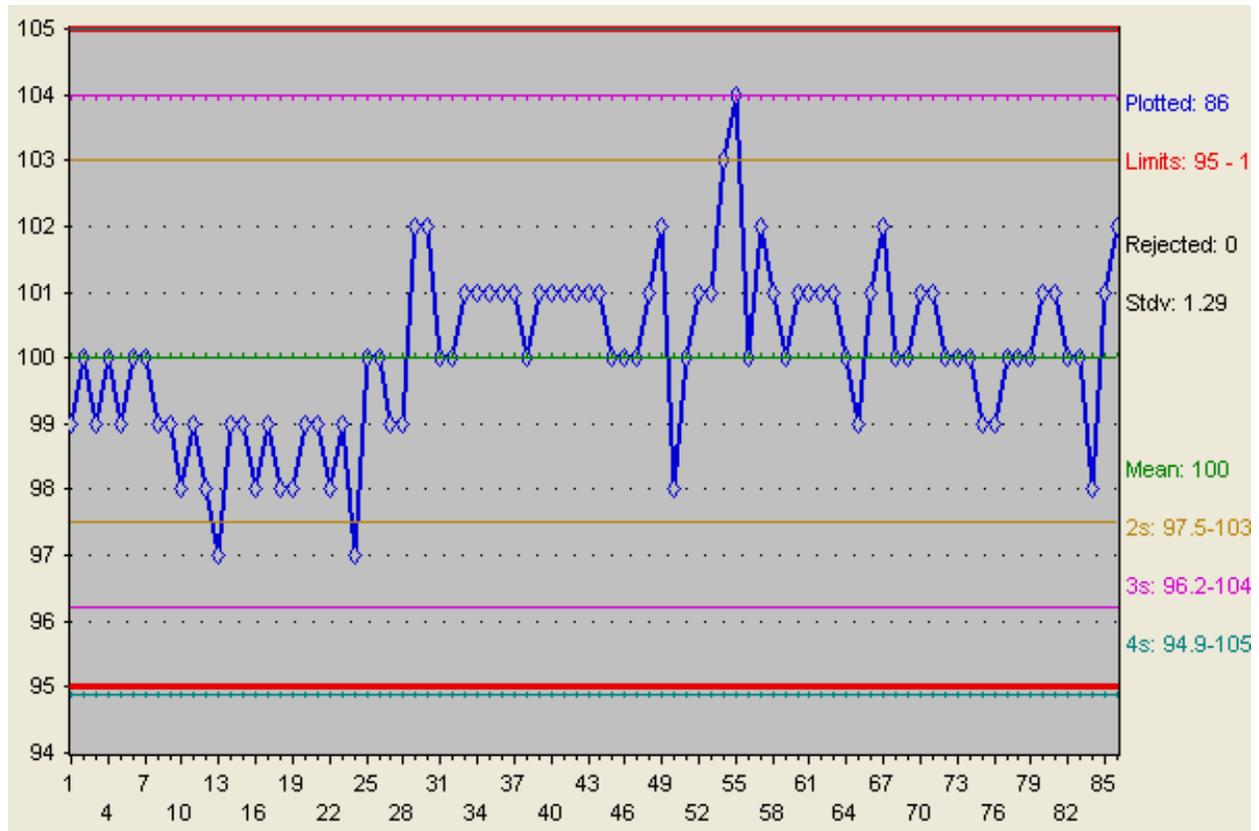
### ATTACHMENT B SO<sup>4</sup> Cellulose CCV



### ATTACHMENT C SO<sup>4</sup> Cellulose SRM



### ATTACHMENT D NO<sup>3</sup> as N Teflon SRM



## PROCEDURE FOR LABELING AND EXPIRATION OF STANDARDS

Effective

Date: 11-10-11

Prepared by: Kathy W. Barry  
Laboratory Chemist

Kathy W Barry

Reviewed by: Garry L. Price  
Laboratory Operations Manager

Garry L Price

Reviewed by: Marcus O. Stewart  
QA Manager

Marcus O Stewart

### TABLE OF CONTENTS

- 1.0 Purpose
- 2.0 Scope
- 3.0 Procedure
- 4.0 References

| Annual Review |        |          |                  |
|---------------|--------|----------|------------------|
| Reviewed by:  | Title: | Date:    | Signature:       |
| MS            | QA Mgr | 10/29/12 | Marcus O Stewart |
| MS            | QA Mgr | 10/31/14 | Marcus O Stewart |
| MS            | QA Mgr | 10/16/15 | Marcus O Stewart |
|               |        |          |                  |
|               |        |          |                  |

## PROCEDURE FOR LABELING AND EXPIRATION OF STANDARDS

### 1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for labeling and determining expiration dates for standards and reagents used in sample preparation and quantitative sample analysis.

### 2.0 SCOPE

This SOP is a general guideline for determining expiration dates for standards, all dilutions of standards, and reagents. This includes standards and reagents purchased or prepared for use on all projects at AMEC.

### 3.0 PROCEDURE

#### 3.1 Vendor prepared standards and reagents

Standards and reagents purchased from vendors should have an expiration date on the label or certificate of analysis. If the vendor does not supply an expiration date for a reagent, the lab will use a date of five years from receipt.

#### 3.2 Lab prepared solutions

Inorganic analyte calibration and quality control solutions for Ion Chromatography (IC) and Inductively Coupled Argon Plasma-Atomic Emission Spectrometry (ICP-AES) will have an expiration date one month after preparation.

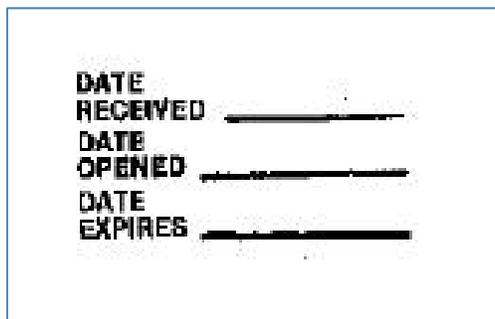
Stock conductivity solutions with a concentration of 0.05 molar (M), or greater, will have an expiration date of one year from preparation.

Working conductivity solutions with a concentration less than 0.05 M will have an expiration date of six months from preparation.

#### 3.3 Labeling

All vendor prepared standards and reagents and lab prepared solutions will be labeled with date received, date opened and expiration date. See Figure 1 for an example of the label that will be affixed to the bottle.

**Figure 1.** Bottle Label



#### **4.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

## PROCEDURE FOR CALIBRATION OF PIPETTORS

Effective  
Date: 11-10-11

Prepared by: Kathy W. Barry *Kathy W. Barry*  
Laboratory Chemist

Reviewed by: Garry L. Price *Garry L. Price*  
Laboratory Operations Manager

Reviewed by: Marcus O. Stewart *Marcus O. Stewart*  
QA Manager

### TABLE OF CONTENTS

- 1.0 Purpose
- 2.0 Scope
- 3.0 Materials
- 4.0 Procedure
- 5.0 References
- 6.0 Attachments

| Annual Review |               |                 |                       |
|---------------|---------------|-----------------|-----------------------|
| Reviewed by:  | Title:        | Date:           | Signature:            |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/26/12</i> | <i>Marcus Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/31/14</i> | <i>Marcus Stewart</i> |
| <i>MS</i>     | <i>QA Mgr</i> | <i>10/16/15</i> | <i>Marcus Stewart</i> |
|               |               |                 |                       |
|               |               |                 |                       |

## **PROCEDURE FOR CALIBRATION OF PIPETTORS**

### **1.0 PURPOSE**

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to the AMEC E&I, Inc. (AMEC) laboratory personnel for calibrating pipettors.

### **2.0 SCOPE**

This SOP is applicable to all pipettors which are used to prepare solutions used by the laboratory.

### **3.0 MATERIALS**

Pipettor, adjustable or fixed.

Analytical balance capable of accurately weighing to the nearest 0.0001 gram (g)

Weigh boat or disposable beaker.

### **4.0 PROCEDURE**

4.1 Pipettors must be calibrated each day of use.

4.2 Calibration is performed as follows:

4.2.1 Place a weigh boat or disposable cup on the analytical balance and tare the balance.

4.2.2 Using the pipettor to be calibrated, draw the needed volume of deionized water.

4.2.3 Release the deionized water into the tared weigh boat. Record the weight in the appropriate notebook for the balance (see Attachment A).

4.2.4 Tare the weight again and repeat steps 4.2.2 and 4.2.3 two more times, recording the weight each time.

4.2.5 Three consecutive weights must be within criteria for the pipettor to be considered calibrated.

4.3 The pipettor is acceptable if the calibration shows that they can deliver within a +/- 1% accuracy. For example, a pipettor calibrated to one mL, must deliver a volume between 0.9900 and 1.0100 mL to be acceptable.

4.4 If the pipettor fails criteria and is adjustable, it should be adjusted and the procedure repeated until the three weights are within criteria. Adjustment procedures vary with pipettor type and manufacturer. Follow the pipettor manual or manufacturer's instructions for proper adjustment.

4.5 If the pipettor is not adjustable and fails criteria or an adjustable pipettor that can not be properly calibrated, it must be removed from service.

## **5.0 REFERENCES**

AMEC E&I, Inc. (AMEC). 2011. *Clean Air Status and Trends Network (CASTNET) Quality Assurance Project Plan, Revision 8.0*. Prepared for U.S. Environmental Protection Agency (EPA), Washington, DC. Contract No. EP-W-09-028. Gainesville, FL.

U.S. Environmental Protection Agency (EPA). 1995. *Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents*. EPA/600/R-96/027, November.

## **6.0 ATTACHMENTS**

Attachment A – Example Logbook Page

### ATTACHMENT A

#### Pipettor Calibration Logbook Page

| DATE     | INIT. | PIPETTE No. | READING 1 | READING 2 | READING 3 |
|----------|-------|-------------|-----------|-----------|-----------|
| 1/3/11   | KSB   | 261184      | 0.9987 g. | 1.0011 g. | 1.0018 g. |
| 1/4/11   | KWB   | 4337114     | 0.9995 g  | 0.9999 g  | 0.9998 g  |
| 1/4/11   | KSB   | 261184      | 1.0023 g  | 1.0025 g. | 1.0037 g. |
| 1/5/11   | KWB   | 4337114     | 0.9998 g  | 0.9999 g  | 1.0002 g  |
| 1/10/11  | KWB   | 4337114     | 1.0002 g  | 1.0005 g  | 1.0001 g  |
| 01/11/11 | DCL   | B600134     | 1.0006g   | 0.9994g   | 1.0003g   |
| 1/11/11  | KWB   | 4337114     | 1.0002 g  | 1.0005g   | 1.0002g   |
| 1/12/11  | KSB   | 261184      | 0.9994 g. | 1.0014 g. | 1.0021 g. |
| 1/13/11  | KSB   | 261184      | 0.9997 g  | 1.0003 g. | 0.9994 g. |
| 1/15/11  | KSB   | 261184      | 0.9998 g  | 0.5006g   | 0.5011 g. |
| 1/18/11  | KWB   | 4337114     | 1.0004 g  | 1.0002 g  | 1.0006 g  |
| 01/24/11 | DCL   | B600134     | 1.0010g   | 1.0003g   | 1.0006g   |
| 1/25/11  | KWB   | 4337114     | 1.0001 g  | 1.0000g   | 1.0003g   |
| 1/28/11  | DCL   | B600134     | 1.0010g   | 1.0013g   | 1.0009g   |
| 1/28/11  | KWB   | 4337114     | 0.9999 g  | 0.9998g   | 1.0002g   |
| 1/31/11  | KSB   | 261184      | 1.0003 g. | 1.0005g.  | 1.0010 g. |
| 02/01/11 | DCL   | B600134     | 1.0015g   | 1.0011g   | 1.0007g   |
| 2/1/11   | KWB   | 4337114     | 1.0002g   | 1.0000g   | 1.0005g   |
| 2/2/11   | KSB   | 261184      | 1.0005g   | 1.0003 g. | 1.0002 g. |
| 2/2/11   | KWB   | 4337114     | 1.0002    | 1.0002    | 1.0000 g  |
| 2/2/11   | KWB   | 4337114     | 0.9998g   | 1.0001g   | 1.0000 g  |
| 2/2/11   | DCL   | B600134     | 1.0021g   | 1.0026g   | 1.0019g   |
| 2/14/11  | KWB   | 4337114     | 1.0001g   | 1.0000 g  | 1.0003g   |
| 2/14/11  | KSB   | 261184      | 1.0015g.  | 1.0020 g. | 1.0016 g. |
| 02/15-11 | DCL   | B600134     | 1.0010g   | 1.0006g   | 1.0010g   |
| 2/15/11  | KWB   | 4337114     | 1.0004 g  | 1.0003g   | 1.0001g   |
| 2/16/11  | KSB   | 261184      | 1.0002g   | 1.0012 g  | 1.0010 g. |
| 2/21/11  | KSB   | 261184      | 1.0010 g  | 0.9998 g  | 1.0006 g. |
| 2/21/11  | KSB   | 261184      | 0.5002 g. | 0.4999 g  | 0.5004 g. |
| 2/21/11  | KWB   | 4337114     | 1.0005 g  | 1.0002 g  | 1.0004g   |
| 2/22/11  | KSB   | 261184      | 1.0002 g. | 0.9991 g. | 1.0004g   |
| 2/28/11  | KWB   | 4337114     | 1.0001 g  | 1.0003 g  | 1.0003g.  |