

## Standard Operating Procedures

### Determination of Anions and/or Cations Extracted from Nylon® Filters by Ion Chromatography (IC)

**Analytical Sciences Department**  
Discovery Sciences  
RTI International\*  
Research Triangle Park, NC

Prepared by: \_\_\_\_\_ Date:\_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date:\_\_\_\_\_

Approved by: \_\_\_\_\_ Date:\_\_\_\_\_

\*RTI International is a registered trademark and a trade name of Research Triangle Institute.

## Table of Contents

	Page
<b>1.0 SCOPE AND APPLICATION .....</b>	<b>4</b>
<b>2.0 SUMMARY OF THE METHOD.....</b>	<b>4</b>
<b>3.0 CONTAMINATION CONTROL.....</b>	<b>4</b>
<b>4.0 SAFETY.....</b>	<b>5</b>
<b>5.0 SAMPLE RECEIPT, STORAGE AND RECORDKEEPING.....</b>	<b>5</b>
<b>6.0 EQUIPMENT, MATERIALS, REAGENTS, AND STANDARDS .....</b>	<b>5</b>
6.1 Laboratory Equipment .....	5
6.1.1 Labware.....	5
6.1.2 Equipment .....	5
6.2 Preparation of Labware .....	6
6.2.1 General Plastic Labware .....	6
6.2.2 Pipette Tips, Plastic.....	6
6.2.3 Autosampler Tubes.....	6
6.2.4 EasyPrep Autodilutor Deionized Water.....	6
6.3 Micropipettes.....	6
6.4 Refrigerator and Freezer.....	6
6.5 Analytical Balance .....	7
6.6 EasyPrep Autodilution system .....	7
6.7 Reagents.....	7
6.7.1 18.2MΩ-cm deionized water (DI).....	7
6.7.2 Anion Chromatography Reagents.....	7
6.7.3 Anion Calibration and Quality Control (QC) Standards.....	7
6.7.4 Cation Chromatography Reagents .....	8
6.7.5 Cation Calibration and Quality Control (QC) Standards.....	8
<b>7.0 STANDARD AND SAMPLE PREPARATION.....</b>	<b>8</b>
7.1 Quality Control Samples .....	8
7.2 Sample Preparation.....	10
7.3 Calibration Standards .....	11
7.4 Sample Storage.....	12

<b>8.0 ANALYSIS BY IC.....</b>	<b>12</b>
8.1 Calculations and Data Reduction .....	13
<b>9.0 METHOD PERFORMANCE .....</b>	<b>13</b>
9.1 Quality Control Samples .....	13
9.2 Linearity .....	14
<b>10.0 CALCULATIONS .....</b>	<b>14</b>
10.1 Percent Recovery of QC Samples .....	14
<b>11.0 DATA MANAGEMENT.....</b>	<b>14</b>
11.1 Data Processing .....	14
11.2 Data Storage .....	16
<b>12.0 IC OPERATION AND MAINTENANCE .....</b>	<b>16</b>
<b>INSTRUMENT DESCRIPTION .....</b>	<b>16</b>
12.1 Instrument Maintenance.....	16
12.2 Instrument Operation .....	16
<b>13.0 MILLI-Q POLLISHER SYSTEM OPERATION.....</b>	<b>17</b>
<b>14.0 REFERENCES.....</b>	<b>17</b>

## **1.0 SCOPE AND APPLICATION**

The method described will be used for the quantitative determination of Anions (defined as chloride ( $\text{Cl}^-$ ), nitrite ( $\text{NO}_2^-$ ), nitrate ( $\text{NO}_3^-$ ), and sulfate ( $\text{SO}_4^{2-}$ )) and/or cations (defined as sodium ( $\text{Na}^+$ ), ammonium ( $\text{NH}_4^+$ ), potassium ( $\text{K}^+$ ), calcium ( $\text{Ca}^{2+}$ ), and magnesium ( $\text{Mg}^{2+}$ )) levels in air quality samples collected on Nylon® filters. The method will be conducted in accordance with applicable SOPs cited herein. Samples will be processed by extracting each filter with deionized water. Deionized water will be added using the EasyPrep Autodilutor. The samples will be sonicated for 30 minutes following the addition of deionized water and allowed to sit overnight at room temperature. Samples will be stored in the refrigerator following equilibration overnight at room temperature. The samples will remain in the refrigerator overnight prior to analysis. The extracts will be analyzed for Anions and/or Cations using Ion Chromatography (IC).

## **2.0 SUMMARY OF THE METHOD**

Nylon filters for collection of anions and cations do not require pre-treatment and are extracted with deionized water. Extraction with deionized water makes it possible to analyze for both anions and cations.

Sample extracts are passed through columns coated with quaternary ammonium active sites for anion analysis and through columns coated with carboxyl active sites for cation analysis. During passage through the column, ion separation occurs due to the different affinities of the ions at the active resin sites. Following separation, the ions pass through suppressors which lower background levels of eluent ions. Species are detected and quantified by a conductivity detector. Accuracy and precision will be monitored routinely by analysis of quality control (QC) samples.

## **3.0 CONTAMINATION CONTROL**

Contaminants in reagents, plastic labware and other components of sample processing, as well as environmental sources, have the potential to cause erroneously high results. Therefore, all samples, Quality Control samples and standards will be prepared in plastic labware rinsed with deionized water. Analysts will use gloves rinsed in deionized water when handling filters, extracts, calibration standards and QC standards. Samples will be capped following the addition of deionized water and will not be uncapped except during the measurement procedure. Samples will be recapped as soon as possible after the analysis.

## **4.0 SAFETY**

All laboratory personnel involved in handling, transporting, and measurement of these samples will wear gloves and eye protection with side shields, in addition to following the normal safety requirements in the RTI Safety and Occupational Health Manual.

## **5.0 SAMPLE RECEIPT, STORAGE AND RECORDKEEPING**

Filters to be extracted are received in sets of 400 and given a set number by Crocker Nuclear Laboratory (CNR) at University of California. The laboratory will label the samples with the same set number as CNR. The set number, and date received are recorded on a Sample Tracking and Extraction log. The sample identification is transferred from the letter received from Crocker on the Sample Tracking and Extraction log. A Sample Receipt Form will be filled out when samples are received by the laboratory. These records will accompany records for the samples from the point of extraction through analysis. Samples will arrive at room temperature and will be stored frozen at -20° in a freezer until extraction. Samples will be extracted at room temperature and allowed to sit overnight immediately following extraction and sonication. They will be moved into refrigerated storage and remain overnight prior to analysis. Unused sample portions will be stored refrigerated for 2 years beginning from the archival date. Samples will be disposed following the two year archival period.

## **6.0 EQUIPMENT, MATERIALS, REAGENTS, AND STANDARDS**

### **6.1 Laboratory Equipment**

#### **6.1.1 Labware**

- Volumetric flasks, Nalgene, various sizes
- Pipette tips, clear plastic, disposable
- Ion chromatography vials (SCP Science)
- Storage bottles of various sizes, HDPE or Teflon
- Disposable flat bottom tubes with screw caps, 50 mL, polypropylene
- Graduated cylinders, polymer, and glass various sizes
- Tweezers

#### **6.1.2 Equipment**

- Micropipettes (micropipets), fixed and variable volume

- Refrigerator (4°C–10°C, nominal)
- Freezer ( $\leq$  -20° C, nominal)
- Ultrasonic bath fitted with epoxy-coated test tube rack to hold flat bottom tubes.
- EasyPrep Autodilution System (SCP Science)
- Ion Chromatography (Dionex ICS-2000, 3000 and Aquion systems)

## 6.2 Preparation of Labware

### 6.2.1 General Plastic Labware

- Volumetric labware will be filled with deionized water and stored capped/covered.
- Devices such as plastic rods and spatulas for aliquoting samples will be rinsed in deionized water.

### 6.2.2 Pipette Tips, Plastic

- Only plastic pipette tips that are free of Ions contamination will be used. If quality control blank analyses consistently show measurable Ions, contamination due to the pipette tip will be considered.

### 6.2.3 Autosampler Tubes

- Vials for use with Dionex equipment are available commercially and are rinsed 3 times with DI water and dried before use.

### 6.2.4 EasyPrep Autodilutor Deionized Water

- The container used to deliver deionized water into the vials for extraction will be rinsed and refilled prior to beginning the extraction.

## 6.3 Micropipettes

Micropipettes used in this analysis will be calibrated in accordance with SOP 100-EQP-020.4, "Gravimetric Calibration Verification and Maintenance of Liquid Dispensing Devices."<sup>1</sup>. No uncalibrated pipettes will be used for transfers that are intended to be quantitative.

## 6.4 Refrigerator and Freezer

Any refrigerator used for this work will be maintained in accordance with SOPs 100-EQP-007.5, "Refrigerator and Freezer Monitoring, Maintenance and Operation with Storage Condition Definitions."<sup>2</sup>, and SOP 100-EQP-009.5 "Calibration of Temperature Measuring Devices."<sup>3</sup>

A -20°C (nominal) freezer will be used at all times.

## 6.5 Analytical Balance

Any analytical balance used for this work will be calibrated and maintained in accordance with SOP 100-EQP-004.5, “Calibration, Use and Maintenance of Balances.”<sup>4</sup>

- Analytical balance capable of one (1) g readability
- Analytical balance capable of two decimal place (.01) g readability

## 6.6 EasyPrep Autodilution system

The EasyPrep Autodilution system will be maintained in accordance with SOP Extractant Via EasyPrep Instrument.

## 6.7 Reagents

6.7.1 18.2MΩ-cm deionized water (DI)

6.7.2 Anion Chromatography Reagents

*Note: Use ACS reagent-grade chemicals and 18.2MΩ-cm deionized water for the preparation of all solutions.*

1. Concentrated eluent (100X), 30 mM NaHCO<sub>3</sub>/270 mM Na<sub>2</sub>CO<sub>3</sub>: Dissolve 2.5209 g NaHCO<sub>3</sub>(commercially purchased) and 28.6178 g Na<sub>2</sub>CO<sub>3</sub>(commercially purchased) in 1 L of deionized water. (Note: Do NOT dry the salts that are used to prepare the eluent.)
2. Working eluent, 0.3 mM NaHCO<sub>3</sub>/2.7 mM Na<sub>2</sub>CO<sub>3</sub>: Dilute 200 mL concentrated eluent to 20 L with deionized water.
3. Regenerant, 0.025 N H<sub>2</sub>SO<sub>4</sub>: Dilute 100 mL of commercially purchased 5.0N H<sub>2</sub>SO<sub>4</sub> to 20 L with deionized water. (Note: This reagent is not used for an IC system equipped with a self-regenerating suppressor.)

6.7.3 Anion Calibration and Quality Control (QC) Standards

Note: Calibration standards are prepared from a commercially purchased National Institute of Standards and Technology (NIST) traceable stock standard. (Spex Certiprep or a verified source) and QC standards are prepared from commercially purchased NIST traceable stock

standards (NSI stock standards or a verified source). Two different sources should always be used when preparing the calibration and QC standards.

- Anion stocks purchased from Spex Certiprep, 1000 ppm or 10000 ppm each for Chloride, Nitrite, Nitrate, and Sulfate
- Anions stocks purchased from NSI 1000 ppm each for Chloride, Nitrite, Nitrate and Sulfate.

#### 6.7.4 Cation Chromatography Reagents

*Note: Use ACS reagent-grade chemicals and 18.2MΩ-cm deionized water for the preparation of all solutions.*

- Concentrated Eluent Stock Solution: 5 N H<sub>2</sub>SO<sub>4</sub>, purchased from VWR Scientific
- Working Eluent, 22 mN Sulfuric Acid: Dilute 8.8 mL 5N H<sub>2</sub>SO<sub>4</sub> to 2 liter using deionized water.

#### 6.7.5 Cation Calibration and Quality Control (QC) Standards

Note: Calibration standards are prepared from commercially purchased NIST traceable stocks (Spec Certiprep or a verified source) and QC standards are prepared from commercially purchased NIST traceable stock standards (NSI stock standards or a verified source). Two different sources should always be used when preparing the calibration and QC standards.

- Cation stocks from Spex Certiprep, 1000 ppm each or 10000 ppm each for Sodium, Ammonium, and Potassium, Magnesium and Calcium
- Cations stocks purchased from NSI 1000 ppm each for Sodium, Ammonium and Potassium, Magnesium and Calcium

### 7.0 STANDARD AND SAMPLE PREPARATION

#### 7.1 Quality Control Samples

7.1.1 Quality control standards (QCS) are prepared in deionized water at low, mid and high range as applicable to the calibration.

Preparation of Intermediate Anion QC Standard and Intermediate Cation QC Standard

Intermediate solutions are stable for at least six (6) months.

Anions 1000 ppm, NIST-traceable, commercial SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, and NO<sub>2</sub><sup>-</sup> and Cl<sup>-</sup> solutions will be used to prepare the Intermediate Anion QC Standard. A 15.0 mL aliquot of 1000 ppm SO<sub>4</sub><sup>2-</sup>, a 7.5

mL aliquot of the 1000 ppm  $\text{NO}_3^-$ , a 5.0 mL aliquot of  $\text{NO}_2^-$  and a 2.5 mL aliquot of the 1000 ppm  $\text{Cl}^-$  will be diluted to two hundred and fifty (250) mL in deionized water to prepare a 60 ppm  $\text{SO}_4^{2-}$ , 30 ppm  $\text{NO}_3^-$ , 20 ppm  $\text{NO}_2^-$  and 10 ppm ( $\text{Cl}^-$ ) Intermediate Anion QC Standard. A list of QC standards and concentrations for each of the ions are shown in Table 1.

**Table 1. Anion QC Standards**

Anions	Vol of Intermediate QC Standard added	Diluted Volume	Final Concentration mg/L $\text{SO}_4^{2-}$	Final Concentration mg/L $\text{NO}_3^-$	Final Concentration mg/L $\text{NO}_2^-$	Final Concentration mg/L $\text{Cl}^-$
QC- LOW	5.00	250	1.20	0.600	0.400	0.200
QC- MED HI	10.0	100	6.00	3.00	2.00	1.00
QC-MED	12.5	250	3.00	1.50	1.00	0.500
QC-HIGH	20.0	100	12.0	6.00	4.00	2.00

Cations 1000 ppm, NIST-traceable, commercial  $\text{Na}^+$ ,  $\text{K}^+$ , and  $\text{NH}_4^+$  will be used to prepare the Intermediate Cation QC Standard. A 25.0 mL aliquot of 1000 ppm  $\text{Na}^+$ ,  $\text{K}^+$ , and  $\text{NH}_4^+$  will be diluted to two hundred and fifty (250) mL in deionized water to prepare the 100 ppm  $\text{Na}^+$   $\text{K}^+$ , and  $\text{NH}_4^+$  Intermediate Cation QC Standard. A list of QC standards and concentrations for each of the ions are shown in Table 2.

**Table 2. Cations**

Cations	Vol of Intermediate QC Standard added	Diluted Volume	Final Concentration mg/L $\text{Na}^+$ , $\text{K}^+$ , $\text{NH}_4^+$ $\text{Mg}^{2+}$ $\text{Ca}^{2+}$
0.040QC	0.040	100	0.040
0.150QC	0.150	100	0.150
0.400QC	0.400	100	0.400
2.000QC	1.00	100	2.00

Laboratory Control Samples (LCS) are prepared during the extraction of the samples by pipetting known concentrations into 50 mL flat bottom tubes and diluting them with the same volume of deionized water used to extract filters. Target concentrations for LCS solutions are listed in Table 3.

**Table 3. Target Concentrations for LCS Solutions**

Final Conc. (PPM)	Final Volume (mL)	LCS Spiking Solution Aliquot (mL)
<b>LCS low</b> $\text{Cl}^- = 0.196$ $\text{NO}_2^- = 0.392$ $\text{NO}_3^- = 0.588$ $\text{SO}_4^{2-} = 1.18$	20.4	0.400 mL
<b>LCS med</b> $\text{Cl}^- = 0.476$ $\text{NO}_2^- = 0.95$ $\text{NO}_3^- = 1.43$ $\text{SO}_4^{2-} = 2.86$	21.0	1.0 mL
<b>LCS high</b> $\text{Cl}^- = 2.00$ $\text{NO}_2^- = 4.00$ $\text{NO}_3^- = 6.00$ $\text{SO}_4^{2-} = 12.0$	25.0	5.0 mL

Method Blanks are prepared during the extraction of samples. An empty 50 mL flat bottom tube is filled with the same volume of deionized water used to extract filters using the autodilutor.

## 7.2 Sample Preparation

### Filter Extraction Procedure

- Label flat bottom tubes with moisture-resistant labels that have been pre-printed with the filter identification for the sample batch to be extracted. Carefully place the label near the top of the flat bottom tube to prevent loss during the sonication procedure.
- Remove filters to be extracted from the freezer and allow them to equilibrate to room temperature.
- Put gloves on hands, rinse well with deionized water, shake dry and wipe away residual water with clean Kimwipe prior to handling tweezers or samples.
- Using tweezers, place each filter in a flat bottom tube that has been labeled with the sample I.D. (Note: Be sure that the label on the flat bottom tube matches the label on the Petri dish.)

- Transfer the flat bottom tube containing the filter into the sample racks used for the autodilution system.
- When the rack for the autodilution system is filled, remove the caps from the flat bottom tubes and place them face up in order on a Kimwipe next to the EasyPrep autodilutor.
- Following procedures from the analytical method for the autodilutor add 20 mL of deionized water to each flat bottom tube.
- Screw the cap tightly on the flat bottom tube.
- Place the sample racks containing the flat bottom tubes in the ultrasonic bath and sonicate for 30 minutes.
- Remove the rack containing the tubes from the bath and allow the extracts to sit at room temperature overnight, and then refrigerate for at least 24 hours prior to analysis.

### 7.3 Calibration Standards

Preparation of Intermediate Standards (Intermediate Standard A and Intermediate Standard 3C) Intermediate solutions are stable for at least six (6) months.

A minimum of eight (8) calibration standards will be prepared directly from Intermediate Standard A for anions as shown in Table 4. A minimum of 6 calibration standards will be prepared directly from Intermediate 3C for cations as shown in Table 5. These standards will either be used that day or refrigerated (for no more than sixty [60] days).

Anions 1000 ppm, NIST-traceable, commercial  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ , and  $\text{NO}_2^-$  commercial  $\text{Cl}^-$  solutions will be used to prepare the Intermediate Standard A. A 10.0 mL aliquot of the 1000 ppm  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ , and  $\text{NO}_2^-$  and a 2.5 mL aliquot of the 1000 ppm  $\text{Cl}^-$  will be diluted to one hundred (100) mL in deionized water to prepare a 100 ppm ( $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ , and  $\text{NO}_2^-$ ) and 20 ppm ( $\text{Cl}^-$ ) Intermediate Standard A.

Table 4. Anions

Anions	Vol of Intermediate A added	Diluted Volume	Final Concentration mg/L $\text{SO}_4^{2-}$ , $\text{NO}_3^-$ , $\text{NO}_2^-$	Final Concentration mg/L $\text{Cl}^-$
Level 1	0.100 mL	200 mL	0.05	0.0100
Level 2	0.200 mL	200 mL	0.100	0.0200

Level 3	0.200 mL	100 mL	0.200	0.0400
Level 4	0.500 mL	100 mL	0.500	0.100
Level 5	2.00 mL	200 mL	1.00	0.200
Level 6	3.00 mL	100 mL	3.00	0.600
Level 7	20.0 mL	200 mL	10.0	2.00
Level 8	25.0 mL	100 mL	25.0	5.00

Cations 10,000 ppm, NIST-traceable, commercial Na<sup>+</sup>, K<sup>+</sup>, and NH<sub>4</sub><sup>+</sup> will be used to prepare the Intermediate Standard 3C. A 1.0 mL aliquot of 10,000 ppm Na<sup>+</sup>, K<sup>+</sup>, and NH<sub>4</sub><sup>+</sup> will be diluted to 100 mL in deionized water to prepare the 100 ppm Na<sup>+</sup>, K<sup>+</sup>, and NH<sub>4</sub><sup>+</sup> Intermediate Standard 3C.

**Table 5. Cations**

Cations	Vol of Intermediate A added	Diluted Volume	Final Concentration mg/L Na <sup>+</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , Mg <sup>2+</sup> , Ca <sup>2+</sup>
Cal Standard 1	0.050 mL	100 mL	0.05
Cal Standard 2	0.100 mL	100 mL	0.100
Cal Standard 3	0.200 mL	100 mL	0.200
Cal Standard 4	0.500 mL	100 mL	0.500
Cal Standard 5	1.00 mL	100 mL	1.00
Cal Standard 6	10.0 mL	100 mL	3.00

#### 7.4 Sample Storage

Extracts will remain refrigerated at 2–8 °C for a minimum of two years following analysis.

### 8.0 ANALYSIS BY IC

The analysis will be set up to run a complete calibration curve at the beginning of the run. Deionized water blanks will be run prior to the calibration curve for sample loop rinsing. QC samples are analyzed at the beginning and end of the sample queue and after every ten samples to ensure instrument stability. Typically, 50 samples complete an analytical batch. Three duplicates and two matrix spikes (prepared by spiking 0.2 mL of a known concentration into 3 mL of sample when using the AS40 autosamplers and spiking 0.05 mL of a known concentration into 0.750 mL of sample when using the AS-AP autosampler) are included with each batch of 50 samples. The Dionex PeakNet® software is set up using a quadratic function for calibration of all anions. The Dionex Chromeleon® software is set up using quadratic functions for the calibration of all anions and cations except for

ammonium which is a cubic fit function. Dionex recommends using a cubic function for the calibration of ammonium.

### **8.1 Calculations and Data Reduction**

Peak areas are entered into the computer where calculations are performed using a quadratic fit to the calibration data. The quadratic fit yields the following:

$$y = ax^2 + bx + c$$

where:

y = the instrument response

x = the calculated anion concentration,  $\mu\text{g/L}$

a = curvature

b = slope

c = offset

Initially, the calibration curve from 0.05 to 10.0 ppm is used for the calculation of the extract nitrite, nitrate, and sulfate concentrations and from 0.01 to 2.0 ppm for chloride. All nitrite, nitrate, and/or sulfate concentrations that exceed 10 ppm are recalculated with the 25.0 ppm standard added to the calibration curve all chloride concentrations exceeding the 2.0 ppm standard are recalculated with the 5.0 ppm standard added to the calibration curve. If recalculated values exceed the highest standard, the extract is diluted appropriately (usually 5-fold) to bring the ion concentration into the calibration range and reanalyzed.

## **9.0 METHOD PERFORMANCE**

### **9.1 Quality Control Samples**

Upper and lower control limits for QC standards and matrix spikes are set at  $\pm 10$  percent for ions with concentrations above 0.050 mg/L when ion concentrations in the QC standards fall below 0.050 mg/L, the acceptable range is  $\pm 35$  percent. If a QC standard sample fails, a second QC sample may be analyzed to verify the calibration. If this sample fails, samples bracketed by the failed QC are reanalyzed. If the reason that a QC standard fails because it was prepared incorrectly and all other QC's in the batch have acceptable recoveries, this is documented as a failure and data are not repeated.

The acceptance criterion for duplicates is based on the sample concentration. Near the detection limit variability will increase and therefore limits are  $\pm$  200 percent. For sample concentrations greater than ten times the detection limit acceptable ranges are  $\pm$  10 percent. For sample spikes, recoveries within 90 to 110 % of target values are acceptable. When QC criteria fail for duplicates or matrix spikes, the sample impacted is reanalyzed as are 5 % of the samples analyzed within the entire sample queue are reanalyzed to verify precision and ascertain if more than one sample was impacted. If other samples reanalyzed fail to meet the duplicate criteria, the entire set is reanalyzed.

## **9.2 Linearity**

The correlation coefficient of the calibration curve must be  $\geq$  0.999 for calibration curves using calibration standards 1 – 7 and  $\geq$  0.995 for calibration curves using calibration standards 1 -8.

# **10.0 CALCULATIONS**

## **10.1 Percent Recovery of QC Samples**

- Measured QC recovery is calculated as measured/target \* 100
- Duplicate precision is calculated as the relative percent difference: Difference/Average and reported at Relative Percent Difference when multiplied by 100.
- For a dilution, samples are multiplied by the volume of sample/total volume.
- Concentrations measured on the IC systems are converted from  $\mu\text{g/L}$  to  $\mu\text{g/filter}$  by multiplying the result by the extraction volume 20 mL/1000.

Duplicate precision is calculated as the relative percent difference: Difference/Average and reported at Relative Percent Difference when multiplied by 100.

# **11.0 DATA MANAGEMENT**

## **11.1 Data Processing**

The instrument software is capable of processing and producing concentration data as both printed and electronic output.

Data are transferred from the instrument using Microsoft Excel electronically. A report template is utilized within the Chromatography software. The template provides the sample injection

ID, and the amount of chloride, nitrite, nitrate and sulfate in  $\mu\text{g/L}$ . The Excel file is stored temporarily onto a thumb drive. The data are copied from the thumb drive and stored onto a secured server. Two copies of the report template are removed from the instrument for each set of data. One copy includes all results with calibration standards 1 – 7. A second copy of the data includes all results with calibration standards 1 – 8. Only results with calibration standards 1 – 8 for any ion that exceed the concentration limit of calibration standard 7 and is less than the calibration standard 8 are used from this copy. A database file is prepared with all results from the appropriate standard curve. This file is imported into the Ion Lab Data Review Application. The database program allows for a review of each batch or sample individually and includes sample name, sample type, project, extraction volume of the sample. The database includes entry points from the reviewer that can be edited during data review. These fields include sample flags and sample comments, or selections to exclude data. The database also enables the data reviewer to review a QA report which shows duplicate percent differences, spiked sample recoveries and QC sample recoveries. A QC report for each analytical batch is printed and maintained with hard copy files of the analytical queues, the COC received from UC Davis, the sample receipt form, extraction records, and a sample extraction log. The database converts the concentration for each ion from  $\mu\text{g/L}$  to  $\text{ug/filter}$ . After the data reviewer completes review of the dataset for an entire batch, the packet of data is passed to QA to review. The QA reviewer verifies that all notes listed on the queues and sample extraction log are noted in the database. They confirm that level 0 and level 1 reviews have been completed, QC checks have all met criteria, reanalyzed samples have met data quality objectives. They also check 5% of the data concentrations in the original excel file match the data listed in the database. They check to ensure that samples requiring the high standard have the appropriate result. They confirm all QC issues are addressed by the level 0 or level 1 reviewers. They also confirm that the data packet is complete. The reviewed packet is transferred back to the data reviewer and data are exported in an excel file and prepared for export to UC Davis. To export data, the data reviewer imports the list of samples received from UC Davis and selects the analysis dates for samples on the list. This file is compared to the sample list provided on the COC from UC Davis to verify that all samples listed on the COC have results entered. The datafile is saved as in csv. format and sent to UC Davis via email.

## **11.2 Data Storage**

All raw data acquired by the instrument will be stored on the computer hard-drive, along with the processed data. At the completion of the study, or at least quarterly, data will be transferred from the instrument hard drive to a secondary storage device used solely for this project.

# **12.0 IC OPERATION AND MAINTENANCE**

## **Instrument Description**

Each system is configured with a conductivity detector, chromatography module, pump system and computer software that is specific to each model. All systems utilize a Dionex AS40 or AS-AP Autosampler. A variety of eluent solutions, analytical column types and other components may be added to achieve detection of the analytes of interest.

## **12.1 Instrument Maintenance**

The operator should be familiar with the instrument and the system software. All analysts will be trained by the ions laboratory manager on instruments operation and maintenance. Service contracts are in place on instruments and the service engineers perform annual preventative maintenance and major failure maintenance as needed. Laboratory analysts perform maintenance associated with changing consumables, changing tubing and fixing leaks as needed. The ions laboratory manager trains all personnel on these routine maintenance procedures and ensures that analysts are adequately trained before they perform maintenance on their own. All maintenance performed by the service engineer or laboratory analysts is recorded on an instrument maintenance record maintained for each instrument.

## **12.2 Instrument Operation**

Ensure that eluent reservoirs are filled daily and check at the end of the day to ensure enough eluent is present to complete the analysis.

Turn on the pump and the suppressor for the system. Allow at least 1 hour for the baseline to stabilize before running standards. Verify that there are no leaks in the DC or pump modules. Wipe up any spilled eluent. Ensure that waste containers have space available for overnight operation, empty as needed.

Ensure that the pump pressure and background conductivity have had time to stabilize before beginning the calibration. The pump pressure and background conductivity are recorded for each

batch. It may be recorded at the time that the instrument is started or recorded during data review by reviewing the audit trail record for the instrument. This information is recorded in the instrument log book and also on the level 0 review record (hard copy) that is stored with each batch of samples processed. Analysts are trained on how to set up queues and begin analyses before they begin analyzing samples on their own. Laboratory analysts show demonstration of capability by performing analyses under the supervision of a trained analyst and have been able to meet QC requirements set forth in Section 9.1.

### **13.0 MILLI-Q POLLISHER SYSTEM OPERATION**

Laboratory analysts are trained to use the Milli-Q polisher systems which provide a secondary filtration of the building deionized water supply which is managed by facilities. The reading on the polisher system should be 18.2 megaohms ( $M\Omega$ ). The reading on the system is recorded daily and analysts are trained to notify the ions laboratory manager or analytical sciences laboratory manager when the reading is below 18.2  $M\Omega$ . DI water from the Milli-Q polisher system is not used until the filters have been serviced and the reading is back to 18.2 $M\Omega$ .

### **14.0 REFERENCES**

1. SOP 100-EQP-020.4, "Gravimetric Calibration Verification and Maintenance of Liquid Dispensing Devices."
2. 100-EQP-007.5, "Refrigerator and Freezer Monitoring, Maintenance and Operation with Storage Condition Definitions."
3. SOP 100-EQP-009.5, "Calibration of Temperature Measuring Devices."
4. SOP 100-EQP-004.5, "Calibration, Use and Maintenance of Balances."
5. SOP "Extractant Via EasyPrep Instrument."

### REVIEW & REVISION HISTORY

Version	Describe Major Changes or Indicate “Reviewed with No Revisions”	Effective Date/ Review Date	New Review Date
7	Editorial changes. Established revision record and added in data review, maintenance and operation of ICs and Milli-Q system. Also expanded QA section.	2/16/2018	2/27/2020

Instructions:

- For revisions, authors increment the version number and add the description of change to this form. Upon receipt of the signed, revised SOP, the SOP Coordinator assigns the new effective date.
- For reviews with no revisions, the SOP Coordinator updates this page and assigns the next date for review upon receipt of a completed review notice.