

**Standard Operating Procedure**  
**RTI SOP # Ions1**

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

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## 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 cations (defined as sodium ( $\text{Na}^+$ ), ammonium ( $\text{NH}_4^+$ ), and potassium ( $\text{K}^+$ )), 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 60 minutes following the addition of deionized water and shaken for 8 hours while at 4° C and allowed to sit overnight at 4°C. The analysis will be performed at least 24 hours after samples have been placed in the refrigerator. The extracts will be analyzed for Anions and 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 DEFINITIONS

- IC- Ion Chromatography
- QC – Quality Control
- DI – Deionized Water; 18.2 MΩ deionized water from the Milli-Q system
- SOP – Standard Operating Procedure; established methods to be followed routinely for the performance of designated operations
- HDPE – High Density Polyethylene
- ACS – American Chemical Society

- NIST – National Institute of Standards & Technology

#### **4.0 CONTAMINATION CONTROL**

Contaminants in reagents, plastic labware and other components of sample processing, as well as environmental sources, typically 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.

#### **5.0 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.

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

Filters to be extracted when received will be labelled, inventoried and will be stored frozen at -20° C (or colder) in a freezer until extraction. The set number, and date received are recorded on a 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 be extracted at room temperature and stored at 4° C overnight following extraction. Unused sample portions will be stored refrigerated for a minimum of 6 months beginning from the archival date. Samples will be disposed when data have been transferred and validated by UC Davis.

#### **7.0 EQUIPMENT, REAGENTS, AND MATRIX**

##### **7.1 Laboratory Equipment**

###### 7.1.1 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 centrifuge tubes with screw caps, 50 mL, polypropylene
- Graduated cylinders, polymer, various sizes
- Tweezers

#### 7.1.2 Equipment

- Micropipettes (micropipets), fixed and variable volume
- Refrigerator (4°C–10°C, nominal)
- Freezer ( $\leq -20^{\circ}$  C, nominal)
- Analytical balance capable of one (1) g readability
- Analytical balance capable of two decimal place (.01) g readability
- Ultrasonic bath fitted with epoxy-coated test tube rack to hold centrifuge tubes.
- EasyPrep Autodilution System.(SCP Science)
- Ion Chromatography (Dionex ICS-2000, ICS-3000, & Aquion) systems

#### 7.1.3 Reagents

- 18.2M $\Omega$ -cm deionized water (DI)

## 7.2 Preparation of Labware

### 7.2.1 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.

### 7.2.2 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.

#### 7.2.3 Autosampler Tubes

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

#### 7.2.4 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. The reading for polisher DI reading is 18.2 MΩ. When the reading drops below this level, DI will not be used until filters have been changed and the water quality reaches 18.2 MΩ.

### 7.3 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.

### 7.4 Refrigerator and Freezer

- Any refrigerator and freezer used for this work will be maintained in accordance with SOPs 100-EQP-007.4, "Refrigerator and Freezer Monitoring, Maintenance and Operation with Storage Condition Definitions."<sup>2</sup>, and SOP 100-EQP-009.4, "Calibration of Temperature Measuring Devices."<sup>3</sup>
- A -20°C (nominal) freezer will be used at all times.

### 7.5 Analytical Balance

- Any analytical balance used for this work will be calibrated and maintained in accordance with SOP 100-EQP-004.4, "Calibration, Use and Maintenance of Balances."<sup>4</sup>

### 7.6 EasyPrep Autodilution system

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

## 7.7 Reagents

### 7.7.1 Anion Chromatography Reagents

Note: Use ACS reagent-grade chemicals and 18.2M $\Omega$ -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.)

### 7.7.2 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 each for Chloride, Nitrate, and Sulfate
- Anions stocks purchased from NSI 1000 ppm each for Chloride, Nitrate and Sulfate.

### 7.7.3 Cation Chromatography Reagents

Note: Use ACS reagent-grade chemicals and 18.2M $\Omega$ -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 4.4 mL 5N H<sub>2</sub>SO<sub>4</sub> to 1 liter using deionized water.



#### 7.7.4 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 for Sodium, Ammonium, and Potassium
- Cations stocks purchased from NSI 1000 ppm each for Sodium, Ammonium and Potassium

## 8.0 STANDARD AND SAMPLE PREPARATION

### 8.1 Quality Control Samples

8.1.1 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  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ , and  $\text{Cl}^-$  solutions will be used to prepare the Intermediate Anion QC Standard. A 15.0 mL aliquot of 1000 ppm  $\text{SO}_4^{2-}$ , a 7.5 mL aliquot of the 1000 ppm  $\text{NO}_3^-$ , 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  $\text{NO}_3^-$ , 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 $SO_4^{2-}$	Final Concentration mg/L $NO_3^-$	Final Concentration mg/L $Cl^-$
QC LOW	2.00	100	1.20	0.600	0.200
QC MED	12.5	250	3.00	1.50	0.500
QC MED HI	10.0	100	6.00	3.00	1.00
QC HIGH	20.0	10	12.0	6.00	2.00

Cations 1000 ppm, NIST-traceable, commercial  $Na^+$ ,  $K^+$ , and  $NH_4^+$  will be used to prepare the Intermediate Cation QC Standard. A 10.0 mL aliquot of 1000 ppm  $Na^+$ ,  $K^+$ , and  $NH_4^+$  will be diluted to one hundred (100) mL in deionized water to prepare the 100 ppm  $Na^+$ ,  $K^+$ , and  $NH_4^+$  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 $Na^+$ , $K^+$ , $NH_4^+$
QC Low	0.020	100	0.200
QC Med	0.250	100	0.250
QC Med Hi	0.750	100	0.750
QC High	2.00	100	2.00

Laboratory Control Samples (LCS) are prepared during the extraction of the samples by pipetting known concentrations into 50 mL centrifuge 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 Anions in LCS Solutions**

Final Conc. (ppm)	Final Volume (mL)	LCS spiking Solution Concentration (ppm)	LCS spiking Solution Aliquot (mL)
<b>LCS low</b> Cl <sup>-</sup> = 0.196 NO <sub>3</sub> <sup>-</sup> = 0.588 SO <sub>4</sub> <sup>2-</sup> = 1.18	25.5	Cl <sup>-</sup> = 10 ppm NO <sub>3</sub> <sup>-</sup> = 30 ppm SO <sub>4</sub> <sup>2-</sup> = 60 ppm	0.500 mL
<b>LCS med</b> Cl <sup>-</sup> = 0.476 NO <sub>3</sub> <sup>-</sup> = 1.43 SO <sub>4</sub> <sup>2-</sup> = 2.86	26.25	Cl <sup>-</sup> = 10 ppm NO <sub>3</sub> <sup>-</sup> = 30 ppm SO <sub>4</sub> <sup>2-</sup> = 60 ppm	1.25 mL
<b>LCS high</b> Cl <sup>-</sup> = 2.00 NO <sub>3</sub> <sup>-</sup> = 6.00 SO <sub>4</sub> <sup>2-</sup> = 12.0	31.25	Cl <sup>-</sup> = 10 ppm NO <sub>3</sub> <sup>-</sup> = 30 ppm SO <sub>4</sub> <sup>2-</sup> = 60 ppm	6.25 mL

**Table 4. Target Concentrations for Cations in LCS Solutions**

Final Conc. (PPM) Na <sup>+</sup> , K <sup>+</sup> , & NH <sub>4</sub> <sup>+</sup>	Final Volume (mL)	LCS spiking Solution Concentration (ppm) Na <sup>+</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup>	LCS spiking Solution Aliquot (mL)
<b>LCS low</b> 0.020 ppm	25.025	20 ppm	0.025 mL
<b>LCS med</b> 0.276	25.350	20 ppm	0.350 mL
<b>LCS high</b> 0.769	26.0	20 ppm	1.0 mL

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

## 8.2 Sample Preparation

### Filter Extraction Procedure

- Label centrifuge 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 centrifuge 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 centrifuge tube that has been labeled with the sample I.D. (Note: Be sure that the label on the centrifuge tube matches the label on the Petri dish.)
- Transfer the centrifuge tube with containing the filter from the Styrofoam box into the sample racks used for the autodilution system.
- When the rack for the autodilution system is filled, remove the caps from the centrifuge 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 25 mL of deionized water to each centrifuge tube.
- Screw the cap tightly on the centrifuge tube.
- Place the sample racks containing the centrifuge tubes in the ultrasonic bath and sonicate for 60 minutes.
- Remove the rack containing the tubes from the bath and place samples on shaker table for 8 hours. Shaker tables are used in cold storage room maintained at 4° C. Sample will be maintained at 4°C until analysis.

### **8.3 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 5. A minimum of 6 calibration standards will be prepared directly from Intermediate Standard C for cations as shown in Table 6. These standards will either be used that day or refrigerated (for no more than thirty [60] days).

Anions 1000 ppm, NIST-traceable, commercial  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ , and  $\text{Cl}^-$  and 1000 ppm, 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 a

2.0 mL aliquot of  $\text{Cl}^-$  will be diluted to one hundred (100) mL in deionized water to prepare a 100 ppm ( $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ ) and 20 ppm ( $\text{Cl}^-$ ) Intermediate Standard A.

**Table 5. Anions**

Anions	Vol of Intermediate A added (mL)	Diluted Volume (mL)	Final Concentration mg/L $\text{SO}_4^{2-}$ , $\text{NO}_3^-$	Final Concentration mg/L $\text{Cl}^-$
Level 1	0.100 mL	200 mL	0.05	0.010
Level 2	0.200 mL	200 mL	0.100	0.020
Level 3	0.200 mL	100 mL	0.200	0.040
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 1000 ppm, NIST-traceable, commercial  $\text{Na}^+$ ,  $\text{K}^+$ , and  $\text{NH}_4^+$  will be used to prepare the Intermediate Standard C. A 10.0 mL aliquot of 1000 ppm  $\text{Na}^+$ ,  $\text{K}^+$ , and  $\text{NH}_4^+$  will be diluted to 100 mL in deionized water to prepare the 100 pm  $\text{Na}^+$ ,  $\text{K}^+$ , and  $\text{NH}_4^+$  Intermediate Standard C.

**Table 6. Cations**

Cations	Vol of Intermediate C added	Diluted Volume	Final Concentration mg/L $\text{Na}^+$ , $\text{K}^+$ , $\text{NH}_4^+$
Standard 1	0.010 mL	100 mL	0.01
Standard 2	0.050 mL	100 mL	0.050
Standard 3	0.100 mL	100 mL	0.100
Standard 4	0.200 mL	100 mL	0.200
Standard 5	0.300 mL	100 mL	0.300
Standard 6	0.500 mL	100 mL	0.500
Standard 7	1.00 mL	100 mL	1.00
Standard 8	3.00 mL	100 mL	3.00

#### 8.4 Sample Storage

Extracts will remain refrigerated at 2–8 °C for a minimum of 6 months following analysis.

## 9.0 ANALYSIS BY IC

The analyst will follow maintenance and operation procedures listed the RTI SOP Analysis of Samples by Ion Chromatography. 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) are included with each batch of 50 samples. 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.

### 9.1 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

The cubic fit yields the following equation:

$$Y = zx^3 + ax^2 + bx + c$$

where:

y = the instrument response

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

z = cubic coefficient

a = curvature

b = slope

c = offset

Initially, the calibration curve is used for the calculation of the extract chloride, nitrate, sulfate, sodium, ammonium, and potassium. When measured concentrations of any ion exceeds the highest standards listed in Tables 5 and 6, the extract is diluted to bring the ion concentration into the calibration range.

## **10.0 METHOD PERFORMANCE**

### **10.1 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.

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.

### **10.2 Linearity**

The correlation coefficient of the calibration curve must be  $\geq 0.999$  when the instruments are calibrated up to calibration standard 7, when calibration standard 8 is used to calibrate for samples which exceed calibration standard 7 concentrations, the acceptable coefficient is  $\geq 0.995$ .

## **11.0 CALCULATIONS**

### **11.1 Percent Recovery of QC Samples**

- Measured concentration/Target Concentration multiplied by 100

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

## **12.0 DATA MANAGEMENT**

### **12.1 Data Processing**

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

### **12.2 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.

## **13.0 REFERENCES**

1. SOP 100-EQP-020.4, "Gravimetric Calibration Verification and Maintenance of Liquid Dispensing Devices."
2. 100-EQP-007.4, "Refrigerator and Freezer Monitoring, Maintenance and Operation with Storage Condition Definitions."
3. SOP 100-EQP-009.4, "Calibration of Temperature Measuring Devices."
4. SOP 100-EQP-004.4, "Calibration, Use and Maintenance of Balances."
5. SOP "Extractant Via EasyPrep Instrument."
6. SOP 203-EQP-008.1 "Operation and Maintenance of Dionex Ion Chromatography Systems."



