

IMPROVE Anion/Cation
Revision 11
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Standard Operating Procedures

RTI SOP # Ion2

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

In Support of the Interagency Monitoring of Protected Visual Environments (IMPROVE)
Program

Prepared by: Adam Conway Date: 3/5/2025

Reviewed by: Andrea McWilliams Date: 3/5/2025

Approved by: Tracy Dombek Date: 3/5/2025

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1.0 SCOPE AND APPLICATION

The method described will be used for the quantitative determination of Anions (defined as chloride (Cl^-), nitrite (NO_2^-), nitrate (NO_3^-), and sulfate (SO_4^{2-})) and/or cations (defined as sodium (Na^+), ammonium (NH_4^+), potassium (K^+), calcium (Ca^{2+}), and magnesium (Mg^{2+})) levels in air quality samples collected on Nylon® filters in support of the Interagency Monitoring of Protected Visual Environments (IMPROVE) program. 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 SimPrep 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 DEFINITIONS

- **QA**–Quality Assurance. QA is a process that is used to maintain a desired level of quality in a service.
- **QC**–Quality Control. QC is a system of maintaining standards in testing by comparing against specified outputs.
- **QCS**–Quality Control Standards. QCSs are prepared in DI water and spiked with a known concentration at low, mid and high range as applicable to the calibration and are used to verify the calibration curve.

- **DI water**–Deionized Water. For reagent water, 17.9 MΩ of DI water is used from the Milli-Q system.
- **SOP**–Standard Operating Procedure. SOPs are established methods to be followed routinely for the performance of designated operations.
- **CSN** – Chemical Speciation Network.
- **LCS**–Laboratory Control Spike. The purpose of an LCS is to evaluate accuracy and precision for the entire process from extraction through analysis.
- **MB**–Method Blank. The purpose of an MB is to evaluate contamination of the overall process, including sample preparation.
- **SPK**– Matrix Spike. The purpose of an MS is to determine whether the sample matrix contributes bias to the analytical result. The MS is an aliquot of an environmental sample to which known quantities of the method analytes are spiked in the sample.
- **Dup** – Duplicate samples are used to measure uncertainty during the analysis of samples. A duplicate sample is the same sample poured into two distinct analytical vials and analyzed consecutively in the analytical batch.
- **Analytical Batch** – A sample queue containing a total of at least 50 field generated samples, QC's at a rate of at least 1 per every 10 samples, SPK and DUP's at a rate of 1 per every 20 samples, LCS's prepared during the extraction step at a rate of 1 per every 20 samples and MB samples prepared during the extraction at a rate of 1 per every 30 samples.
- **PPB**-parts per billion (µg/L and or ng/mL)
- **PPM**- parts per million. (mg/L and or µg/mL)
- **USB**–Universal Serial Bus. The USB drive is standardized technology for attaching peripheral devices to a computer.
- **HDPE**–High Density Polyethylene
- **ACS**–American Chemical Society
- **NIST**–National Institute of Standards & Technology
- **MDL**–Method Detection Limit. An MDL is the minimum concentration of an analyte that can be measured and reported with 99% confidence that the measured concentration is distinguishable from method blank results.
- **IDL**–Instrument Detection Limit. The IDL is the concentration equivalent to a signal, due to the analyte of interest, which is the smallest signal that can be distinguished from background noise by a particular instrument.

4.0 INTERFERENCES AND 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.

Integration tools are used eliminate chromatographic interferences such as shoulder peaks and co-eluting peaks. Samples are diluted when ion concentrations are so high that they interfere with resolution of adjacent ion peaks.

5.0 HEALTH AND SAFETY WARNINGS

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 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 (see **Figure 1** for an example of the 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 $<0^{\circ}\text{C}$ 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.

7.0 STANDARDS REAGENTS AND EQUIPMENT

Standard stock solutions for each anion and cation may be commercially purchased. The manufacturer's expiration date and storage conditions must be followed. If the manufacturer's date exceeds one year from opening, the laboratory will assign an expiration from the first use. A solution prepared from a stock standard will not have an expiration date past the certified date of the stock standard. Stocks will be purchased from approved vendors which may include Spex CertiPrep, NSI Solutions, and HPS. A primary stock source will be purchased for the preparation of calibration standards and a secondary source will be purchased to prepare quality control solutions used for calibration verification.

Upon first use, the analyst must verify that the certified concentration values are within $\pm 3\%$ of the nominal value. If certified values fall outside this range, the stock standard must not be used. The nominal concentration will be used to calculate calibration standard and quality control sample concentrations.

The laboratory maintains a standard and reagent preparation logbook. The laboratory records the preparation date for all reagents, expiration dates for stocks, reagents, and prepared solutions and standards, lot numbers of reagents and stocks, and serial numbers of pipettes used to prepare solution and standards.

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 flat bottom tubes with screw caps, 50 mL, polypropylene
- Graduated cylinders, polymer, and glass various sizes
- Tweezers

7.1.2 Equipment

- Micropipettes (micropipets), fixed and variable volume
- Refrigerator ($\sim 4^{\circ}\text{C}$, nominal)

- Freezer ($\leq 0^{\circ}\text{C}$, nominal)
- Ultrasonic bath fitted with epoxy-coated test tube rack to hold flat bottom tubes.
- SimPrep Autodilution System (SimPrep Science)
- Ion Chromatography (Dionex ICS-2000, 3000, 6000 and Aquion systems)
 - Analytical and Guard columns – the serial number of the analytical column will be recorded in the instrument logbook when columns are changed to provide traceability.
 - Suppressors

7.1.3 Reagents and Standards

- 17.9 megaohm ($\text{M}\Omega$) DI water
- 5N H_2SO_4 (Scientific Sales or Equivalent)
- Na_2CO_3 (Fisherbrand or Equivalent)
- NaHCO_3 (EMD, or Equivalent)
- 30% H_2O_2
- Cl^- 1000 ppm NIST traceable stock standards (primary and secondary).
- SO_4^{2-} 1000 ppm NIST traceable stock standards (primary and secondary).
- NO_3^- 1000 ppm NIST traceable stock standards (primary and secondary).
- NO_2^- 1000 ppm NIST traceable stock standards (primary and secondary).
- Na^+ 1000 ppm NIST traceable stock standards (primary and secondary).
- NH_4^+ 1000 ppm NIST traceable stock standards (primary and secondary).
- K^+ 1000 ppm NIST traceable stock standards (primary and secondary).

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 SimPrep 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.

7.3 Micropipettes

Micropipettes used in this analysis will be calibrated in accordance with SOP 100-EQP-020, "Gravimetric Calibration Verification and Maintenance of Liquid Dispensing Devices."¹ No uncalibrated pipettes will be used for transfers that are intended to be quantitative.

7.4 Refrigerator and Freezer

Any refrigerator used for this work will be maintained in accordance with SOPs 100-EQP-007, "Refrigerator and Freezer Monitoring, Maintenance and Operation with Storage Condition Definitions."², and SOP 100-EQP-009 "Calibration of Temperature Measuring Devices."³

A $\leq 0^{\circ}\text{C}$ (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, "Calibration, Use and Maintenance of Balances."⁴

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

7.6 SimPrep Autodilution system

The SimPrep Autodilution system will be maintained in accordance with SOP Filter Extraction via SimPrep Autodilution System.

7.7 Chromatography Reagents

7.7.1 Anion Chromatography Reagents

- Concentrated eluent (100X), 30 mM NaHCO₃/270 mM Na₂CO₃: Dissolve 2.5209 g NaHCO₃ and 28.6178 g Na₂CO₃ in 1 L of deionized water. (Note: Do NOT dry the salts that are used to prepare the eluent.)
- Working eluent, 0.3 mM NaHCO₃/2.7 mM Na₂CO₃: Dilute 200 mL concentrated eluent to 20 L with deionized water.

7.7.2 Cation Chromatography Reagents

- Working Eluent, 22 mN Sulfuric Acid: Dilute 8.8 mL 5N H₂SO₄ to 2 L using deionized water.

8.0 STANDARD AND SAMPLE PREPARATION

8.1 Quality Control Samples

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 months.

Anions 1000 ppm, NIST-traceable, commercial SO₄²⁻, NO₃⁻, and NO₂⁻ and Cl⁻ solutions will be used to prepare the Intermediate Anion QC Standard. A 15.0 mL aliquot of 1000 ppm SO₄²⁻, a 7.5 mL aliquot of the 1000 ppm NO₃⁻, a 5.0 mL aliquot of NO₂⁻ and a 2.5 mL aliquot of the 1000 ppm Cl⁻ will be diluted to 250 mL in deionized water to prepare a 60 ppm SO₄²⁻, 30 ppm NO₃⁻, 20 ppm NO₂⁻ and 10 ppm (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 NO_2^-	Final Concentration mg/L Cl^-
QC- LOW	5.0	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 Na^+ , K^+ , and NH_4^+ will be used to prepare the Intermediate Cation QC Standard. A 25.0 mL aliquot of 1000 ppm Na^+ , K^+ , and NH_4^+ will be diluted to 250 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	0.020
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 flat bottom tubes and diluting them with the same volume of deionized water used to extract filters. Target concentrations for Anions and Anions in LCS solutions are listed in Table 3 and Table 4, respectively.

Table 3. Target Concentrations for LCS Solutions

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

Table 1. Target Concentrations for Cations in LCS Solutions

Final Conc. Na^+ , K^+ , & NH_4^+ (ppm)	Final Volume (mL)	LCS spiking Solution Concentration Na^+ , K^+ , NH_4^+ (ppm)	LCS spiking Solution Aliquot (mL)
LCS low 0.020	25.025	20	0.025
LCS med 0.276	25.350	20	0.350
LCS high 0.769	26.000	20	1.000

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.

8.2 Sample Preparation

Filter Extraction Procedure (see Figure 2 for an example of the extraction worksheet).

- 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 SimPrep 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.

8.3 Calibration Standards

Preparation of Intermediate Standards Anion Calibration Standard Intermediate and Cation Calibration Standard Intermediate solutions are stable for at least six (6) months.

Calibration standards will be prepared directly from Anion Calibration Standard Intermediate for anions as shown in Table 5. Calibration standards will be prepared directly from Cation Calibration Standard Intermediate for cations as shown in Table 6. These standards will either be used that day or refrigerated (for no more than 60 days).

Anions 1000 ppm, NIST-traceable, commercial SO_4^{2-} , NO_3^- , and NO_2^- commercial Cl^- solutions will be used to prepare the Intermediate Standard A. A 10.0 mL aliquot of the 1000 ppm SO_4^{2-} , NO_3^- , and NO_2^- and a 2.5 mL aliquot of the 1000 ppm Cl^- will be diluted to one hundred (100)

mL in deionized water to prepare a 100 ppm (SO_4^{2-} , NO_3^- , and NO_2^-) and 20 ppm (Cl^-) Intermediate Standard A.

Table 5. Anions

Anions	Vol of Intermediate A added	Diluted Volume	Final Concentration mg/L SO_4^{2-}, NO_3^-, NO_2^-	Final Concentration mg/L 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 Na^+ , K^+ , and NH_4^+ will be used to prepare the Cation Calibration Standard Intermediate. A 1.0 mL aliquot of 1000 ppm Na^+ , K^+ , and NH_4^+ will be diluted to 100 mL in deionized water to prepare the 100 pm Na^+ , K^+ , and NH_4^+ Cation Calibration Standard Intermediate.

Table 6. Cations

Cations	Vol of Intermediate A added	Diluted Volume	Final Concentration mg/L Na^+, K^+, NH_4^+
Cal Standard 1	0.010 mL	100 mL	0.010
Cal Standard 2	0.050 mL	100 mL	0.050
Cal Standard 3	0.100 mL	100 mL	0.100
Cal Standard 4	0.200 mL	100 mL	0.200
Cal Standard 5	0.300 mL	100 mL	0.300
Cal Standard 6	0.500 mL	100 mL	0.500
Cal Standard 7	1.00 mL	100 mL	1.00
Cal Standard 8	3.00 mL	100 mL	3.00

8.4 Sample Storage

Extracts will remain refrigerated at $\sim 4^\circ\text{C}$ for a minimum of two years following analysis.

9.0 ANALYSIS BY ION CHROMATOGRAPHY

The analysis will be set up to run a complete calibration curve at the beginning of the run. DI 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 AS40 autosamplers and 0.05 mL of a known concentration into 0.75 mL of sample when AS-AP autosamplers are used) 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.

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, µg/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, µg/L

- z = cubic coefficient
- a = curvature
- b = slope
- c = offset

The initial calibration curve consists of seven points and is used to calculate the concentration of chloride, nitrate, nitrite, sulfate, sodium, ammonium, and potassium. If measured concentrations exceed the highest point of the seven-point curve, an eighth calibration standard is added to extend the calibration range. If any ion concentration exceeds the eighth calibration standard listed in Tables 5 and 6, the extract is diluted to bring the ion concentration within 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 ± 10 percent (%) for ions with concentrations above 0.050 milligrams/liter (mg/L). When ion concentrations in the QC standards fall below 0.050 mg/L, the acceptable range is $\pm 35\%$. 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 $\pm 200\%$. For sample concentrations greater than ten times the detection limit, acceptable ranges are $\pm 10\%$. 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 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. The acceptance criteria for DI water blanks added at the start of the calibration must fall at or below 2 times the current MDL.

10.2 Linearity

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

11.0 CALCULATIONS

11.1 Details of Calculations for QC Samples

The expected recovery for QC samples is calculated for recovery by:

$$\%Recovery = \frac{V_{spk} - V_s}{Spk} \times 100$$

Where:

V_{spk} = value observed of the spiked sample

V_s = value observed of the unspiked sample

Spk = value of the spike.

Duplicate precision is calculated as the relative percent difference by:

$$RPD = \frac{(V - V_{dup})}{(V + V_{dup})/2} \times 100$$

Where:

V = value of primary result

V_{dup} = value of duplicate result.

12.0 INSTRUMENT DETECTION LIMITS AND METHOD DETECTION LIMITS

12.1 Instrument Detection Limits

The Instrument Detection Limits (IDL) are calculated by measuring a DI blank sample 7-10 times by IC.

- Calculate the standard deviation of all the results.
- The IDL is calculated using students t (n-1) at a 99% confidence level.

12.2 Method Detection Limits

Method Detection Limit (MDL) is defined as the minimum concentration of an analyte that can be measured and reported with 99% confidence that the measured concentration is distinguishable from method blank results. The following steps are used to derive the MDL for this Chemical Speciation method on multiple instruments:

- Process seven spiked water samples and seven method blank water samples through all steps of the method. With multiple instruments, each instrument must analyze at least two samples and two blanks within the 12-month period.
- Existing data and blanks used for MDL calculation can be used if compliant with the requirements for at least three batches, analyzed on three separate days, and generated within the last twelve months.
- If blanks are collected throughout the year (e.g., batch method blanks), can be used to calculate the MDL Blank (MDLb). Do not use blanks derived from gross failures.
- If not using existing data, samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates.
- Preparation and analysis may be on the same day for the batch.
- Prepare a spike of the target analyte 2 to 5 times greater than the expected MDL each sample.
- Analyze each sample.
- Calculate the standard deviation of the spiked samples.
- The student t-value used to calculate the MDL will represent the number of samples used by the laboratory, which is in accordance with 40 CFR Part 136.3, Appendix B, Revision 2.
- If none of the method blanks give numerical results for an individual analyte, the MDLb does not apply.
- If some (but not all) of the method blanks for an individual analyte give numerical results, set the MDLb equal to the highest method blank results. If more than 100 method blanks are available, set MDLb to the level that is no less than the 99th percentile of the method blank results. For “n” method blanks where $n \geq 100$, sort the method blanks in rank order. The $(n * 0.99)$ ranked method blank result (round to the nearest whole number) is the MDLb.
- If the calculated MDLb is higher than the calculated MDL spike for any element, then it is assumed there is blank contamination, and corrective action must be taken to identify the source of the contamination. The MDL study must be re-done so that the MDLs is higher than the MDLb

- MDLs are determined annually, and corrective action will be taken if the detection limits do not meet the contract-required detection limits.

13.0 DATA MANAGEMENT

13.1 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 $\mu\text{g/filter}$. After the data reviewer completes review of the dataset for an entire batch, the packet of data is passed to an independent data reviewer for review before exporting the data for the client..

The independent data 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 level 1 data reviewer and data are exported in an excel file and prepared for export to UC Davis. To export data, the level 1 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.

13.2 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.

14.0 CORRECTIVE ACTION

14.1 Routine Corrective Actions

Corrective Action is initiated whenever a program QC failure is identified, such as exceeding control limits or contamination issues.

- Before Preliminary Data Reporting
 - Deviation identified before preliminary data is reported to data validators do not require a formal corrective action report.
 - These deviations are documented on the Level 0 and Level 1 reviewer sheets, and analysis are repeated as part of the corrective action.
 - Since the issue is addressed before reporting, the data is not impacted.
- After Data Finalization:
 - If modifications or deviations are discovered after data have been finalized, a formal corrective action report is required.
 - A formal corrective action report will be initiated by the Ions laboratory manager or project manager and provided to the Quality Assurance Manager for approval.

14.2 Non-Routine Corrective Actions

Non-routine failures include PE, CRMs, and audit samples. The Laboratory Manager and QA Officer reviews the PE, CRMs, or audit sample results for possible discrepancies. If a failure occurs, then the Laboratory Manager, will investigate the analytical run with chemists and determine which corrective action is appropriate. This information will be reported back to the QA Officer via e-mail for approval on the agreed upon corrective action.

15.0 IC OPERATION AND MAINTENANCE

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.

15.1 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. Any maintenance performed by the service engineer or laboratory analysts is recorded on an instrument log book maintained for each instrument.

15.2 Instrument Operation

Daily Instrument Setup and Analysis Procedures:

- Fill eluent reservoirs daily and check at the end of the day to ensure there is enough eluent to complete the analysis.
- Wipe up any spilled eluent.
- Ensure waste containers have sufficient capacity for overnight operation and empty them as needed.

Typically, instruments are started on the first working day of the week and continue throughout the week without a shutdown. If the system is not started:

- Turn on pump, determine if the pump back-pressure is stable.
 - Verify that no leaks are present, if there are leaks assess the situation and let the ions laboratory manager know if the leaks cannot be stopped.
 - If no leaks are found, prime the pump for at least 5 minutes.
- When the pump back pressure has achieved stability, turn on the suppressor.
- Allow the instrument to warm up for about an hour prior to beginning an analysis.
- The calibration and quality control standards are poured and loaded into designated racks on the AS-AP autosamplers and are replaced on the first working day of the week. The lowest calibration standards 1 and 2 and lowest QC standard are all replaced mid-week. Throughout the rest of the week, vials are filled and replaced as needed.

- Replace the DI water in the AS-AP rinse bottle on the first working day of the week.
- Load the analytical queue in the system.
- Start the analysis after system has equilibrated for at least an hour if it is the first working day of the instrument or as soon as the queue is loaded into the instrument and calibration standards and QC solutions have been replaced or checked to ensure that adequate volume remains for the duration of the analysis.
- Record the pump back pressure and the background conductivity on the Level 0 reviewer sheet and in the instrument logbook.
- Systems may be set up for a delayed start when an analysis begun the previous day is still running but will finish before the next workday.
- In such cases, pump back pressure and background conductivity can be obtained by reviewing the audit trail for the first DI water blank sample analyzed.
- These values should be recorded on the on the level 0 reviewer worksheet and in the instrument log book.

16.0 PREVENTATIVE MAINTENANCE

Regular preventative maintenance is essential to ensure the long-term operation of ion chromatography instruments. These instruments undergo annual servicing by vendor service engineers. For instruments no longer eligible for service contracts, maintenance is performed by the laboratory manager and analysts that have demonstrated the ability under the laboratory managers supervision. Preventative maintenance includes the following tasks:

- Pump Maintenance: Replacement of check valve and seals.
- Six-port Valve Maintenance: Replacement of rotors and stators.
- AS-AP Autosampler Maintenance: Routine disinfection with a 5-10% H₂O₂ solution.

A decline in peak areas and failing QC criteria for ammonium and potassium typically indicate biofilm buildup in the AS-AP autosamplers. All analysts are trained to identify and address this issue as part of their on-going training. Maintenance activities are documented in logbooks maintained for each instrument.

17.0 DI WATER POLLISHER SYSTEM OPERATION

The laboratory operates two DI water polisher systems that provide secondary filtration of the building's deionized water supply, which is managed by RTI facilities. These systems must produce DI water with a minimum resistance of 17.9 megaohms (MΩ). Analysts record this reading daily and must notify the Ions Laboratory Manager or Analytical Sciences Chemistry Manager if the resistance falls below 17.9 MΩ and stop use of water from the systems until the system has been serviced.

18.0 WASTE MANAGEMENT

Laboratories are urged to protect air, water, and land by minimizing releases from hood and bench operations, complying with any sewer and discharge permits and regulations, and by complying with all solid and hazardous waste regulation. More information about waste management is presented in *The Waste Management Manual for Laboratory Personnel*, which is available from the ACS.

19.0 REFERENCES

1. *RTI SOP 100-EQP-020: Gravimetric Calibration Verification and Maintenance of Liquid Dispensing Devices*
2. *RTI SOP 100-EQP-007: Refrigerator and Freezer Monitoring, Maintenance and Operation with Storage Condition Definitions*
3. *RTI SOP 100-EQP-009: Calibration of Temperature Measuring Devices*
4. *RTI SOP 100-EQP-004: Calibration, Use and Maintenance of Balances*
5. *RTI SOP: Filter Extraction via SimPrep Autodilution System*
6. *40 CFR Part 136, Appendix B: Definition and Procedure for the Determination of the Method Detection Limit (Revision 2)*

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Sample Receipt Form

RTI Project Number: _____ Date Received: _____

Number of Samples: _____

Description: _____

Sample Storage Location: Johnson 2nd Floor, Freezer # _____

Batch #: _____

Sample Condition:

____ All samples were received in good condition.

____ The following discrepancies were found (see attached sheet if necessary):

____ The following actions were taken to resolve the discrepancies see attached sheet if necessary):

Acknowledgment of Receipt	
Sample Custodian	Date

Figure 1. Example of the sample receipt form.

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EXTRACTION WORKSHEET	RESEARCH TRIANGLE INSTITUTE POST OFFICE BOX 12194 RESEARCH TRIANGLE PARK, NC 27709-2194	EXTRACTION DATE: _____ SAMPLE SET #: _____ PROJECT: NPS IMPROVE
TITLE: Extraction of Nylon Filters in Deionized Water TRAY #: _____ General Instructions Initial and date each task as soon as it is completed; sign the bottom of the page when all entries have been completed and have reviewer sign bottom of page. Data entries should be performed using indelible, black-ink ballpoint pens. Be sure to record scientific observations and deviations from this procedure on this document.		
Reagents	Supplier	Lot Number
Deionized (DI) Water	Milli-Q IQ7000	NA
Cl ⁻ (1000 PPM Stock Solution)	NSI Lab Solutions	
NO ₃ ⁻ (1000 ppm Stock Solution)	NSI Lab Solutions	
NO ₂ ⁻ (1000 ppm Stock Solution)	NSI Lab Solutions	
SO ₄ ⁻² (1000 ppm Stock Solution)	NSI Lab Solutions	
LCS spiking solution: 60 ppm - SO ₄ ⁻² 30 ppm - NO ₃ ⁻ 20 ppm - NO ₂ ⁻ 10 ppm - Cl ⁻	Calibration and QA Standards Logbook (Located Johnson, Lab 247): Date: _____ Pg#: _____	NA
Equipment	Manufacturer/Model	Serial Number or ID# / Location
Deionized (DI) Water System	Millipore / Milli-Q IQ7000	F25829069 / Johnson, Lab 187
SimPrep AutoDilution System #1	Teledyne / SimPrep	0122138A560 / Johnson, Lab 187
SimPrep AutoDilution System #2	Teledyne / SimPrep	0122139A560 / Johnson, Lab 187
Sonicator/Water Bath #1	Bransonic / 8510R-MT	RPA11097783F / Johnson, Lab 187
Sonicator/Water Bath #2	Bransonic / 8800	BGQ052499470D / Johnson, Lab 187
Temperature Probe #1	Fisherbrand / Traceable	230270889 / Johnson, Lab 187
Temperature Probe #2	Fisherbrand / Traceable	230561430 / Johnson, Lab 187
Adjustable Volumetric Pipette		
Adjustable Volumetric Pipette		
50mL Plastic Digestion tubes	Mold Pro / MP-108PW	n/a
Freezer ACS216-FRZ	General Electric / FP21SXARWH	DS160520 / Johnson hallway
Refrigerator/Freezer #2	Kenmore / Goldspot	VS30260205 / Johnson hallway
Refrigerator #4	Frigidaire / FRU17B2JW19	WA95000736 / Johnson hallway
Refrigerator #6	Frigidaire / FRU17B2JW19	WA95000723 / Johnson, Lab 247
Refrigerator #7	Whirlpool / WRR56X18FW02	U84903152 / Johnson hallway
Refrigerator #8	Whirlpool / WRR56X18FW02	U85102798 / Johnson, Rm 246

1

Analyst Signature: _____ Date: _____ Reviewer Signature: _____ Date: _____
 Filename: Extraction Worksheet_V9 121324 SimPrep

Figure 2. Example of the extraction worksheet (page 1 of 6).

EXTRACTION WORKSHEET	RESEARCH TRIANGLE INSTITUTE POST OFFICE BOX 12194 RESEARCH TRIANGLE PARK, NC 27709-2194	EXTRACTION DATE: _____ SAMPLE SET #: _____ PROJECT: NPS IMPROVE
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SAMPLE EXTRACTION PROCEDURE

- Remove the filter samples for extraction from the freezer:
Freezer Location _____ Freezer ID# _____
Date/Time taken out of freezer - Date: _____ Time: _____
☐ Analyst/Date _____ / _____

- **LCS Spiking Solution – (6-month expiration/store in refrigerator)** If needed, prepare the LCS Spiking Solution: Add the appropriate amount of each analyte into a 250 mL volumetric flask as indicated in Table 1 below. Add DI water to the 250 mL calibration line and shake the solution to mix.

Table 1 – Preparation of LCS Spiking Solution

Analyte	Volume of 1000 ppm stock to add (mL)	Final Volume (mL)	Final Concentration (ppm)
Chloride	2.50	250 mL	10
Nitrite	5.00	250 mL	20
Nitrate	7.50	250 mL	30
Sulfate	15.0	250 mL	60

☐ YES – LCS spiking solution prepared (note Logbook page # and Lot Id of the solutions in the comment box below)
☐ NO – LCS spiking solution not prepared – removed from refrigerator ID# _____
☐ Analyst/Date _____ / _____

COMMENTS:

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Analyst Signature: _____ Date: _____ Reviewer Signature: _____ Date: _____
Filename: Extraction Worksheet_V9 121324 SimPrep

Figure 2. Example of the extraction worksheet (page 2 of 6).

EXTRACTION WORKSHEET	RESEARCH TRIANGLE INSTITUTE POST OFFICE BOX 12194 RESEARCH TRIANGLE PARK, NC 27709-2194	EXTRACTION DATE: _____ SAMPLE SET #: _____ PROJECT: NPS IMPROVE
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- Rinse gloved hands and stainless steel tweezers with DI water and dry with a clean Kimwipe:
☐Analyst /Date _____/_____
- Transfer each filter into an appropriately labeled 50 mL plastic digestion tube and verify that the filter ID matches the tube ID. Arrange all samples, method blanks, and LCS samples in autosampler racks in the order as indicated on the "Sample Tracking and Extraction Log" document.
☐Analyst /Date _____/_____
- Method Blanks** - Prepare an appropriate number of Method Blanks as indicated on the "Sample Tracking and Extraction Log" document based on the number of samples that will be extracted by labeling a 50 mL extraction tube as "Method Blank". These tubes do not contain a filter.
Number of Method Blanks prepared: _____
☐Analyst /Date _____/_____
- LOW, MED, and HIGH LCS dilutions** - Prepare an appropriate number of LCS samples for dilution as indicated on the "Nylon Filter Sample List and Analysis Location" document based on the number of samples that will be extracted by labeling 50 mL centrifuge tube as "LCS Low", "LCS Med", and "LCS High". Add the appropriate amount of LCS spiking solution to each tube as indicated in Table 2.

Final Conc. (PPM)	Final Volume (mL)	No. of Samples for Dilution Prepared	LCS spiking Solution Aliquot (mL)	Analyst/Date
LCS low Cl ⁻ = 0.196 NO ₃ ⁻ = 0.392 NO ₂ ⁻ = 0.588 SO ₄ ²⁻ = 1.18	20.4		0.400 mL	_____/_____ _____/_____
LCS med Cl ⁻ = 0.476 NO ₃ ⁻ = 0.95 NO ₂ ⁻ = 1.43 SO ₄ ²⁻ = 2.86	21.0		1.0 mL	_____/_____ _____/_____
LCS high Cl ⁻ = 2.00 NO ₃ ⁻ = 4.00 NO ₂ ⁻ = 6.00 SO ₄ ²⁻ = 12.0	25.0		5.0 mL	_____/_____ _____/_____

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Analyst Signature: _____ Date: _____ Reviewer Signature: _____ Date: _____
Filename: Extraction Worksheet_V9 121324 SimPrep

Figure 2. Example of the extraction worksheet (page 3 of 6).

Analyst Signature: _____ Date: _____ Reviewer Signature: _____ Date: _____
 Filename: Extraction Worksheet_V9 121324_SimPrep

Figure 2. Example of the extraction worksheet (page 4 of 6).

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Set 5: ☐ Analyst /Date _____ / _____ ☐ Second Analyst /Date _____ / _____

Set 6: ☐ Analyst /Date _____ / _____ ☐ Second Analyst /Date _____ / _____

Set 7: ☐ Analyst /Date _____ / _____ ☐ Second Analyst /Date _____ / _____

Set 8: ☐ Analyst /Date _____ / _____ ☐ Second Analyst /Date _____ / _____

Set 9: ☐ Analyst /Date _____ / _____ ☐ Second Analyst /Date _____ / _____

Set 10: ☐ Analyst /Date _____ / _____ ☐ Second Analyst /Date _____ / _____

▪ **Tightly cap all tubes and place samples in a Sonicator/ice water bath (3 racks at a time). Sonicate the samples for 30 minutes. Begin sonication by adjusting the built-in timer to 30 minutes. During sonication, the water bath temperature should not exceed 10°C.**

Set 1: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 2: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 3: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 4: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 5: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 6: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 7: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 8: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

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Analyst Signature: _____ Date: _____ Reviewer Signature: _____ Date: _____
Filename: Extraction Worksheet_V9 121324 SimPrep

Figure 2. Example of the extraction worksheet (page 5 of 6).

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EXTRACTION WORKSHEET	RESEARCH TRIANGLE INSTITUTE POST OFFICE BOX 12194 RESEARCH TRIANGLE PARK, NC 27709-2194	EXTRACTION DATE: _____ SAMPLE SET #: _____ PROJECT: NPS IMPROVE
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Set 9: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

Set 10: Sonicator# _____ Pre-temp °C _____ Time samples in Sonicator: _____
Post-temp °C _____ Time samples removed from Sonicator _____

☐ Analyst/Date _____ / _____
Allow Samples to sit overnight at room temperature.

☐ Analyst/Date _____ / _____

- Place the LCS Spiking Solution in the Refrigerator – ID# _____
☐ Analyst/Date _____ / _____
- Place the samples in the refrigerator and allow them to remain there at least one additional overnight before analysis.
Refrigerator Location _____ Refrigerator ID# _____
Date/Time samples were placed in the refrigerator - Date: _____ Time: _____
☐ Analyst/Date _____ / _____

COMMENTS:

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Analyst Signature: _____ Date: _____ Reviewer Signature: _____ Date: _____
Filename: Extraction Worksheet_V9 121324 SimPrep

Figure 2. Example of the extraction worksheet (page 6 of 6).

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
8	Editorial changes, changed wording on freezer storage to 0 degrees. Changed QA data reviewer to independent data reviewer for clarification of roles.	2/18/21	2/18/2022
9	Change autodilution system from EasyPrep to SimPrep	7/11/2022	7/11/2023
10	Assign RTI SOP number, update SOP numbers.	8/7/2024	8/7/2026
11	Added in Definitions, Instrument detection limits and method detection limits, corrective action, preventative maintenance, and waste management sections to bring SOP in line with CSN SOP. General editorial changes and formatting.	3/5/2025	3/5/2026

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.