
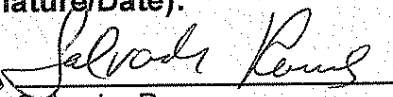
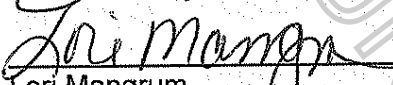
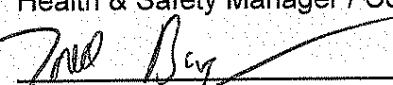
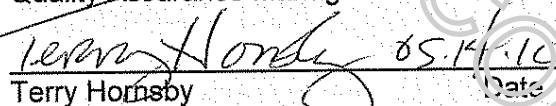
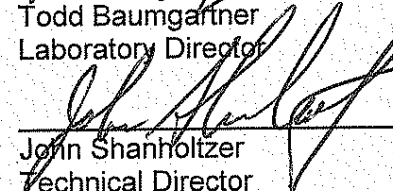


Inorganic Anions by Ion Chromatography

Method: EPA 300.0 and SW-846 9056

Approvals (Signature/Date):

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1.0 Scope and Application

This procedure can be used to determine the concentration of various anions in water, soils, the bomb combustion of solid waste, and oil samples, and leachates by ion chromatography. The method is most useful for the determination of dirty or highly colored samples that might be problematic for colorimetric determination or in some instances where a lower limit of detection is required for certain analytes. Nitrite, Nitrate, and phosphate anions should be determined by the colorimetric procedure due to the very short hold times.

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP is applicable to the determination of the concentration of anions in water and soil leachates.

Anion	Range
Fluoride	0.02 – 5.0mg/L
Chloride	0.2 – 50mg/L
Bromide	0.02 – 5.0mg/L
Nitrite	0.02 – 5.0mg/L
Nitrate	0.02 – 5.0mg/L
Sulfate	0.2 – 50mg/L

The routine target analyte lists, current Reporting Limit (RL), Method Detection Limit (MDL) and precision and accuracy limits associated with this procedure are given in the Method Limit Group (MLG) in TALS.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Tampa's most current revision of the Quality Assurance Manual (TP-QAM).

2.0 Summary of Method

- 2.1 Ion chromatography is a method for the separation of dissolved ionic species using liquid chromatography techniques, along with eluent suppression and conductivity detection. A carbonate/bicarbonate or hydroxide eluent conducts the sample through the chromatography system, which consists of a guard column, a separator column, a suppressor column, and a conductivity detector. The guard column screens out potential interference's, the separator column separates the anions, the suppressor column converts carbonate and bicarbonate ions in the eluent to carbonic acid (lowering the conductivity), and the conductivity detector detects the anions from its electrical conductivity in solution.
- 2.2 The following extraction should be used for solid materials. Add an amount of reagent water equal to ten times the weight of the dry solid material taken as a sample. This slurry is mixed for ten minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45µm membrane type filter. This can be the type that attaches directly to the end of the syringe.
- 2.3 This method is based on EPA Method 300.0 and SW-846 Method 9056.

3.0 Definitions

- 3.1 Refer to SOP TP-AN-005: *Definitions, Terms, and Acronyms* for a complete listing of applicable definitions and to the current revision of the Tampa Quality Assurance Manual (TP-QAM).
- 3.2 Anion – Is a negative charged ion

4.0 Interferences

- 4.1 Anions whose retention times are similar or substances whose peaks overlap the anion peak of interest interfere with quantitation. Sample dilution and/or a change in eluent strength can often resolve these types of problems.
- 4.2 Water, which elutes before the fluoride peak, gives a negative peak and can cause interference's. This problem can be eliminated by the addition of 1mL of 100-fold concentrated eluent to each 100mL of sample and standards.
- 4.3 Particles from samples may lodge in the system, causing blockage; thus, samples are filtered utilizing a 0.45 μ m filter prior to analysis.
- 4.4 Interference's may be caused by contamination in reagent water, reagents, glassware and other sample processing apparatuses, resulting in extraneous peaks not due to the sample matrix itself, or in elevated or noisy baselines that make identification and quantitation of the target analytes difficult.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

- Sodium Fluoride is highly toxic.
- Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Monochloro-acetic acid	Corrosive	None	Contact with skin or eyes may cause severe irritation or burns. Inhalation of vapors may cause severe irritation of the respiratory system.
Sodium fluoride	Poison	2.5 mg/m3-TWA as F	Highly Toxic. Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.
Sodium hydroxide	Corrosive	2mg/m3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 Always add acid to water to prevent violent reactions.			
2 Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- 6.1.1 Ion chromatography system, Dionex DX120 or ICS2000 or equivalent instrument with conductivity detector
- 6.1.2 Suppressor, ASRS (Auto Self-regenerating Suppressor)
- 6.1.3 Autosampler vials with caps
- 6.1.4 RFC-30: eluent free system

6.2 Supplies

- 6.2.1 Guard column(s): Dionex AG18, 4x50mm(IC#2) or AG9-HC, 4x50mm(IC#3)
- 6.2.2 Separator column(s): Dionex AS18, 4x250mm(IC#2) or AS9-HC, 4x250mm(IC#3)
- 6.2.3 Suppressor(s): ASRS 300, 4mm
- 6.2.4 Syringes, disposable, 10cc
- 6.2.5 Syringe filters, Whatman, 0.45µm pore size
- 6.2.6 Volumetric glassware, Class A

7.0 Reagents and Standards

Reagents must be tracked in accordance with TP-AN-004: *Reagent Traceability*. Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions and scanned into TALS. All preparation steps must be in accordance with the most current revision of TP-AN-004 *Standard Materials Traceability*.

- 7.1 Reagent water: lab generated deionized water
- 7.2 Eluent: Commercially purchased KOH from Dionex (IC#2), or 9mM sodium

carbonate solution

7.2.1 1.95g of anhydrous sodium carbonate to a final volume of 2L (IC#3)

-or-

7.2.2 36mL of 0.5M NaHCO₃ to a final volume of 2L.

7.3 Calibration standards

7.3.1 Prepare a mixed standard containing the analytes of interest using the guidance in the tables below. Dilute to volume to give a mixed standard containing the analytes of interest at the shown concentrations.

	Std 1 (ppm)	Std 2 (ppm)	Std 3 (ppm)	Std 4 (ppm)	Std 5 (ppm)	Std 6 (ppm)	Std 7 (ppm)
Fl	0.02	0.05	0.1	0.5	1.0	2.0	5.0
Br	0.02	0.05	0.1	0.5	1.0	2.0	5.0
Cl	0.2	0.5	1.0	5.0	10.0	20.0	50.0
SO ₄	0.2	0.5	1.0	5.0	10.0	20.0	50.0
NO ₂	0.02	0.05	0.1	0.5	1.0	2.0	5.0
NO ₃	0.02	0.05	0.1	0.5	1.0	2.0	5.0

NOTE: This table contains recommended calibration levels. Other calibration standards can be used as required as long as the calibration criteria stated in this SOP is met and the lowest standard is at or below the project RL.

7.3.2 The lowest calibration standard must be at or below the project reporting limit. The remaining standards will define the working range of the calibration.

7.4 ICV/CCV/LCS Spiking Solution (Common Anions): can use commercially purchased solutions.

7.4.1 ICV standard is prepared from second sources at the same concentration as Standard 5. F, Br, NO₂, NO₃ at 1.0ppm; Cl and SO₄ at 10ppm.

7.4.2 CCV's are prepared using same source as curve at the same concentration as Standard 5. F, Br, NO₂, NO₃ at 1.0ppm; Cl and SO₄ at 10ppm.

7.4.3 LCS's are prepared from second sources at the same concentration as Standard 5. F, Br, NO₂, NO₃ are at 1.0ppm; Cl and SO₄ are at 10ppm.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time ¹	Reference
Waters	HDPE	5mL	Cool $\leq 6^{\circ}\text{C}$	28 Days * NO ₂ & NO ₃ 48hours	40 CFR Part 136.3
Soils	Glass	3 grams	Cool $\leq 6^{\circ}\text{C}$	28 Days	N/A

¹ The holding times for each analyte are given in Table 2 of the attached SOP Summary.

Note: The extraction and analysis must be completed within the stated holding times for soils.

9.0 Quality Control

9.1 Sample QC

9.1.1 A laboratory method blank, lab control spike, lab control spike duplicate, and two matrix spikes, and matrix spike duplicates are extracted with batch. A batch can include up to 20 client samples. If no matrix spike is specified by a client request, then one per ten samples will be chosen by the analyst extracting the batch. The most current revision SOP TP-AN-006: *Analytical Batching* describes the procedures for evaluating batch-specific QC. This criteria is summarized in the SOP Summary (Table 3).

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< MDL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	Statistical Limits ²
Laboratory Control Sample (LCSD)*	1 in 20 or fewer samples	Statistical Limits ²
Matrix Spike (MS) ¹	1 in 10 or fewer samples	Statistical Limits ²
MS Duplicate (MSD) ¹	1 in 10 or fewer samples	Statistical Limits ²

* LCS Duplicate (LCSD) is performed only when there are samples for a TMDL project included in the batch.

¹ The sample selection for MS/MSD are randomly selected, unless specifically requested by a client.

² Statistical control limits are updated annually and are updated into IALS.

9.2 Instrument QC

9.2.1 Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument.

9.2.2 A calibration curve is performed daily, followed by an Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB).

9.2.2.1 The ICB serves as the method blank for water analyses.

9.2.3 A Continuing Calibration Verification (CCV) and Calibration Verification Blank (CCB) are analyzed every 10 or fewer samples.

9.2.4 The accuracy (% Recovery) and precision (% RPD) for the lab spike and matrix spikes should be checked against the limits listed in the method. The

lab spikes must meet these accuracy and precision limits. If limits are not met, investigate the cause and either reanalyze or re-extract. The matrix spike recoveries are used to evaluate the matrix effect on the analysis and are advisory.

9.2.5 Linear Calibration Range

9.2.5.1 After the calibration curve has run, the low, mid, and high points are run as samples. They are then compared to the actual values, and must agree within 10%. If they are not within the 10%, the calibration curve must be analyzed again, followed by another linear calibration range set. This must be performed initially, and every six months thereafter. Since the initial calibration is performed almost every analytical run, and the initial calibration satisfies the requirements for the LCR, the ICAL will be considered to have fulfilled the LCR requirement.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Instrument Conditions

This section provides general guidance and conditions for operation of the IC. The actual conditions used by the analyst must be documented for each analytical run either as a printout, in an analytical log, or stored on computer.

Column	AS18 or AS9-HC, 4 X 250mm
Guard	AG18 or AG9-HC, 4 X 50mm
Sample Loop	25, 50, 75, or 100 uL
Eluent	ECGII KCl, 32mm or Sodium Carbonate 9mM
SRS Current	80mA (IC#2), 45mA (IC#3)
Pressure	≈2000 psi
Suppressor	ASRS 300, 4mm
Conductivity	<1.0 μs
Run Time	10 min (IC#2), 24 min (IC#3)

10.1.1.1 Select the appropriate column for the determination and install it into the ion chromatographic system. Select the appropriate eluent for the determination as found in the Reagents and Standards section, and attach the eluent line to the eluent reservoir. Turn on the nitrogen gas supplying pressure for the sample injection system. Turn on the pump. Turn on the ASRS.

NOTE: Refer to the Instrument Manual for detailed start-up of instrument.

10.1.1.2 Check the baseline conductivity of the cell.

10.1.1.3 Allow the system to stabilize.

10.1.2 Aqueous samples require no preparation other than warming to room temperature.

10.1.3 Soil samples require no preparation other than warming to room temperature and mixing well with a metal spatula.

10.2 **Calibration**

10.2.1 Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument.

10.2.2 An initial calibration is analyzed upon instrument setup, when new columns are installed, and when calibration verification standards fail to meet the acceptance criteria.

10.2.3 Turn on the IC and make sure that the instrument is functioning within normal parameters.

10.2.4 Prepare the calibration standards for each analyte of interest. A minimum of five calibration standards and a blank must be prepared and analyzed for each target compound that is to be reported. The lowest calibration standard must be at the reporting limit and the other standards will define the working range of the detector.

10.2.5 Construct a calibration curve for each target analyte and evaluate the curve. If the correlation coefficient is greater than or equal to 0.995, the curve can be used to quantify samples. If the criteria are not met, evaluate the standards data for errors in entry, calculations, or standard concentration. Re-injection of one or more standards may be required (as long as no samples have been run after the curve) or recalibration may be necessary.

10.2.6 During the calibration process, the retention time window of each target analyte must be determined. The retention time window is calculated as three times the standard deviation of the retention times in the initial calibration. If the retention time window calculates to zero (no deviation over the calibration range), a default window of 0.1 to 0.2 minutes is used.

NOTE: The retention time window applies only to concentrations within the working range of the calibration curve.

10.3 **Sample Analysis**

10.3.1 Aqueous

10.3.1.1 Filter the sample through a 0.45um filter using a disposable syringe.

10.3.1.2 ICB/MB/LCS/LCSD

10.3.1.2.1 Transfer 5.0mL of reagent water into a vial for the ICB and MB.

10.3.1.2.2 For the LCS/LCSD, transfer 5.0mL of the second source same concentrations as Standard 5 into vials.

10.3.1.3 MS/MSD transfer 2.5mL of LCS/LCSD solution into vial, then 2.5mL of filtered sample into vial – mix well.

10.3.1.4 Add the ICB to autosampler vial. The QC samples are ready for analysis.

10.3.2 Soils

10.3.2.1 Mix the sample thoroughly with a stainless steel spatula. Discard any leaves, rocks or sticks.

10.3.2.1.1 See TP-AN-070: *Sample Compositing* if a sample composite must be performed or if the sample is difficult to homogenize. Notify the supervisor if the sample presents any unusual challenges.

10.3.2.2 Add an amount of reagent water equal to ten times the weight of the dry solid material taken as a sample. This slurry is mixed for ten minutes using a magnetic stirring device.

10.3.2.3 Filter the resulting slurry before injecting using a 0.45µm membrane type filter. This can be the type that attaches directly to the end of the syringe. Care should be taken to show that good recovery and identification of peaks is obtained with the user's matrix through the use of fortified samples.

10.3.2.4 The method blank and the LCS are prepared using 50mL of reagent water and assuming a sample weight of 5g of Ottawa Sand. The MS/MSD are prepared using one of the samples in the batch.

10.3.2.5 Add 5mL of the LCS/MS spiking solution to the LCS, MS, and MSD. The true concentrations of the LCS and MS, using 5.0g of sample, are:

Stock	Stock Conc. (*) (mg/kg)
Fluoride	40.0
Chloride	80.0
Bromide	40.0
Nitrite	20.0
Nitrate	20.0
Phosphate	20.0
Sulfate	80.0

(*) The theoretical concentration of the MS/MSD will depend on the weight of sample and percent solids of the sample. See TestAmerica Tampa SOP TP-AN-006: *Analytical Batching* for the calculation of the theoretical concentration of the MS and MSD.

10.3.2.6 Add 50mL of reagent water to each sample and QC item.

10.3.2.7 Mix the samples and QC and let settle for 2 hours.

10.3.2.8 When ready to analyze, remove a portion of the supernatant liquid and filter through a 0.45µm filter and collect the filtrate in an autosampler tube. The sample should be analyzed as soon as possible after filtration.

10.3.2.9 Filter 5.0mL of the sample into a small plastic container or autosampler vial.

10.3.3 The concentration of the sample is determined from the calibration curve. If the response of the sample exceeds that highest standard in the calibration curve, the sample is diluted and reanalyzed. The computer can calculate the dilution if put into the schedule or by the analyst if not scheduled.

10.3.4 Oils and Solid Wastes –Bomb Combustion

10.3.4.1 For the preparation of oils and wastes refer to TP-GE-065: *Bomb Preparation Method for Solid Waste* SW-846 Method 5050.

11.0 Calculations / Data Reduction

11.1 The dilution factor (DF) is calculated:

$$DF = \frac{FV}{V}$$

Where:

FV = final volume of the dilution

V = volume of sample used to prepare the dilution

11.2 Anion concentrations in liquid samples are calculated as shown:

$$\text{Anion concentration, mg/L} = \frac{\text{mg}}{\text{L}}(\text{curve}) \otimes DF$$

11.3 Anion concentrations in soil and sediment are calculated from their extract concentrations as shown:

$$\text{Anion concentration, mg/kg dw} =$$

$$\frac{\text{mg}}{\text{L}} \text{ in extract} \otimes \frac{\text{Volume of extract (L)} \otimes 1000 \text{ g/kg}}{\text{Sample wt. (g)} \otimes \text{dry wt. fraction}}$$

11.4 Accuracy

ICV / CCV, LCS % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$

11.5 Precision (RPD)

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

11.6 Data Analysis

11.6.1 Anions are identified by comparing the retention time of the peaks in the samples with the retention times of standards analyzed under the same conditions. If a peak falls within the retention time window established for the target analyte, the peak is identified as the target compound. The peak area in the sample must fall within the linear working range of the detector to be identified as the target. A sample must be diluted to bring the area within the linear range before the peak is identified as the target.

11.6.2 If a peak is suspected of being a target analyte and does not fall within the absolute retention time window, the sample or sample extract should be spiked. Spike with the target analyte at a concentration that will result in a peak that is about 50-100% higher than the peak in the sample chromatogram.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL) - The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined accordance with 40 CFR, Chapter 1, Part 136, Appendix B and with reference to the laboratory's MDL procedure in the Quality Assurance Manual. An MDL reflects a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities - Each analyst must perform an Initial Demonstration of Capability (IDOC) in accordance with the procedure outlined in the Quality Assurance Manual. The evaluation of the IDOC data should be completed prior to the analysis of samples. A Continuing Demonstration of Capability (CDOC) must be performed annually or whenever there is a significant change in the instrument parameters or the associated method.

- 12.3 Training Requirements – Each analyst's required employee training (such as orientation to the laboratory's policies and procedures and in-house method training) are outlined in the Quality Assurance Manual.

13.0 Data Assessment And Acceptance Criteria For Quality Control Measures

- 13.1 The following represent data assessment for samples and acceptance criteria for QC measures. Corrective actions for any failure in QC measures are shown in Section 14.

13.2 QC sample acceptance criteria

- 13.2.1 **Method Blank.** No target analytes may be present in the method blank above the method detection limit.

- 13.2.2 **Laboratory Control Sample (LCS).** (90 – 110% Recovery) The analyte must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

- 13.2.3 **Matrix Spike/Matrix Spike Duplicate (MS/MSD).** The analyte should be within established control limits for accuracy (%Recovery) and precision (%RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCS/LCSD. No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgment in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline. Check with supervisor, Lab Manager and or Project Manager on reporting out of control limits QC.

13.3 Sample result evaluation

- 13.3.1 **Dilutions:** If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit in the upper half of the calibration range. The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

14.0 Corrective Actions For Out Of Control Data

- 14.1 **Method Blank.** The samples in the batch associated to the defective method blank are evaluated. If the analyte found in the method blank is confirmed to not be present in one or more of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements. If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared. If the concentration in the method blank exceeded 10% of concentration found in one or more samples, the

prescribed corrective action is to re-analyze all affected samples. If the concentration in the method blank was less than 10% of the concentration found in one or more samples, the sample can be reported by qualifying the affected analytes. A nonconformance memo (NCM) is written and discussed with the laboratory supervisor and Project Management for evaluation against project requirements.

- 14.2 **Laboratory control sample.** If the analyte is out of control for accuracy, the associated samples are evaluated. If the recovery is biased high and the associated samples have no positive results for that analyte, a non-impact situation ensues. A nonconformance memo (NCM) is written to notify project management of the situation for evaluation against project requirements. If the recovery is biased high and the associated samples have positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s). If the recovery is biased low and the samples have no positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s). If the analyte is out of control for precision (RPD), the associated samples are evaluated. All decisions about corrective action(s) are made in consultation with the project manager. If there are no positive results in one or more samples, a non-impact situation ensues for those samples. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1 If there are positive results for one or more analytes, the likelihood of poor reproducibility increases and corrective action must be evaluated. A nonconformance memo is written and discussed with the laboratory supervisor and Project Management of the situation for a project decision on whether the affected sample(s) should be reanalyzed.

15.0 Contingencies For Handling Out-Of-Control Or Unacceptable Data

- 15.1 **Method blanks.** If there is insufficient sample to perform re-analysis; the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with an "I" qualifier and appropriate comments in the narrative.
- 15.2 **LCS/LCSD.** If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation. An NCM will have to be filled out stating this problem and or a solution.
- 15.3 **Insufficient sample.** If there is insufficient sample to repeat the analysis, the situation is discussed with the project manager for consultation with the client and documentation is provided in an NCM.

16.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Employees must abide by the policies in the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

17.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TP-HAZ-001 *Waste Management*).

The following waste streams are produced when this method is carried out.

- Aqueous acidic/alkaline material. All waste is to be segregated and put into the waste container to be disposed of by the hazardous waste coordinator.

18.0 References / Cross-References

18.1 Method EPA 300.0 Determination of Inorganic Anions by Ion Chromatography; Revision 2.1, Methods for the Determination of Inorganic Substances in Environmental Samples, August 1993.

18.2 Test Methods for Evaluating Solid Waste, Third Edition; U.S. EPA Office of Solid Waste SW-846 Update IVB Method 9056A Rev 1, November 2000

18.3 TestAmerica's *Environmental Health & Safety Manual* CW-E-M-001, Most current revision.

18.4 Tampa's *Quality Assurance Manual* (TP-QAM), most current revision

18.5 TestAmerica Tampa SOP's:

18.5.1 TP-HAZ-001: *Waste Management*

18.5.2 TP-AN-004: *Standard Materials and Reagent Traceability*

18.5.3 TP-AN-005: *Definitions, Terms, and Acronym*

18.5.4 TP-AN-006: *Analytical Batching*

18.5.5 TP-AN-016: *Glassware Cleaning Procedures*

18.5.6 TP-AN-070: *Sample Compositing*

18.5.7 TP-GE-065: *Bomb Preparation Method for Solid Waste SW-846 Method 5050*

18.5.8 TP-CA-090: *Procedures for the Determination of Method Detection Limit (MDL)*

18.5.9 TP-HAZ-001: *Waste Management*

19.0 Method Modifications:

This section is not applicable to this SOP.

20.0 Attachments

20.1 Table 1 – Method Summary Ion Chromatography

20.2 Table 2 – SOP Summary

21.0 Revision History

- Revision 1, dated 02 Aug 2006
 - Various paragraphs re-worded, Safety Section up dated, edited format according to NELAC criteria, ICS 2000 added as a second instrument.
- Revision 2, dated 25 March 2008
 - Format changed to TestAmerica SOP format. Not released
 - Updated safety section
 - Added revision history section
- Revision 3, dated 15 November 2009
 - Updated Scope and application section
 - Updated sections 12-20
 - Corrected revision history numbering
- Revision 4, dated 01 June 2010
 - Minor revision to Section 7: removed standard level tables
 - Added section 9.2.5 Linear Calibration Range

Table 1 - Method Summary Ion Chromatography

Analyte	Matrix	Preservation	Hold Time
Bromide, Chloride, Fluoride, Sulfate,	Water and Soils	Store at 4°C(*)	28 Days
Nitrate, Nitrite	Water	Store at 4°C(*)	48 Hours
Nitrate, Nitrite	Soils	Store at 4°C(*)	28 Days: 48 hours after extracted

(*) 4°C is target temperature; acceptance limits are less than 6°C with no frozen samples

Table 2 -- SOP Summary

QC Check		Frequency	Acceptance Criteria	Corrective Action
Initial Calibration -minimum 5 point curve with lowest point at RL		Prior to sample analysis and when CCV fails; at least every 6 months	Correlation Coefficient or Coefficient of Determination of 0.995 or greater or %RSD<=15%; Analysis of ICV to +/- 10% of true value	-Evaluate ion chromatogram and integrations. -Check calculations -Reanalyze standard(s) -Remake standard(s) and reanalyze
Initial Calibration Blank (ICB)		After analysis of ICV	Less than RL in TALS	-Evaluate ion chromatogram and integrations. -Check calculations -Reanalyze -Remake and reanalyze -Recalibrate
Continuing Calibration Verification (CCV) -mid-range cal std or QCS		After analysis of initial calibration standards, and after every ten samples and at the end of the sequence	+/-10% of true value (90-110% recovery)	-Evaluate ion chromatogram and integrations. -Check calculations -Reanalyze standard -Remake and reanalyze standard -Reanalyze associated samples -Recalibrate and reanalyze associated samples
Continuing Calibration Blank (CCB)		After analysis of initial calibration standards, and after every ten samples and at the end of the sequence	Less than RL in TALS	-Evaluate ion chromatogram and integrations. -Check calculations -Reanalyze -Remake and reanalyze -Reanalyze associated samples -Recalibrate and reanalyze associated samples
Lab Control Sample (LCS/ LCSD)		Per batch * See note below	Within TALS Limits	-Evaluate ion chromatogram and integrations. -Check calculations -Reanalyze -Remake and reanalyze -Reanalyze associated samples -Recalibrate and reanalyze associated samples
MS/MSD		One Per 10 samples	Within TALS limits	-Evaluate ion chromatogram and integrations. -Check calculations -If lab control and blank OK, matrix interference can be assumed -Reanalyze -Remake and reanalyze

* **NOTE:** A laboratory control sample duplicate (LCSD) is performed only when there are samples for a TMDL project included in the batch.

Table 2 -- SOP Summary (Continued)

QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Demonstration of Performance	Per analyst	-Prepare cal standards and demonstrate linearity -Analyze QCS (90 - 100%) -perform annual MDL study	-Evaluate ion chromatogram and integrations. Check calculations -Reanalyze for analytes that do not meet criteria.
Method Detection Limit (MDL)	Every 6 months	See Tampa's SOP TP-CA-090	See Tampa's SOP TP-CA-090
Linear Dynamic Range	Every 6 months	All analytes within 10% of expected value	Calibration range lowered to meet LDR results
Performance Evaluation (PE) Sample -WSWP	Quarterly	Within acceptance limits	-Evaluate data and initiate non-conformance reports
Retention Time Window	Each analysis	Define a reasonable window for each analyte or retention time range and apply to each analysis	-Evaluate instrument performance, column, and integration system.
Dilution/Fortification	When high concentration of adjacent peak makes identification difficult	Peak in dilution and/or fortified sample within established window.	-Dilute or fortify (spike) sample at higher dilution factor or concentration.