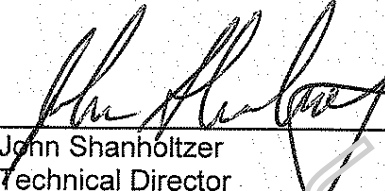
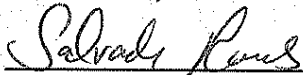




Title: Elements by ICP

Method: EPA 200.7, EPA 6010B

Approvals (Signature/Date):	
 John Shanholtzer Technical Director	 Salvador Ramos Health & Safety Manager / Coordinator Department Manager, Metals
 Lori Mangrum Quality Assurance Manager	 Keith Blanchard Laboratory Director
6/9/09 Date	6/4/09 Date
6/9/09 Date	6/15/09 Date

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures to determine the concentration of various elements by inductively coupled plasma (ICP) atomic emission spectroscopy according to EPA Method 200.7 and SW-846 Method 6010B.

This method contains the analytical procedures for the determination of metals in surface and ground water, wastewater, soil, sediment, leachate (EP, TCLP, or SPLP), and waste samples after digestion.

Table 1 lists the elements that may be determined by ICP and the characteristic wavelength used for each element.

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 (MLGs) in LIMS.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 20 in the Quality Assurance Manual.

2.0 Summary of Method

Prior to analysis by ICP, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber where a stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

3.0 Definitions

3.1 Preparation Batch: The set of samples extracted/distilled/or digested at the same time to a maximum of 20 samples (this includes the MB/LCS/MS/MSD).

3.2 Analytical Batch: The set of samples that were analyzed on the instrument that comprises the opening calibration and ends with a passing CCV/CCB.

3.3 Laboratory Control Standard (LCS)--A solution prepared in the laboratory by adding known quantities of analyte to reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy.

- 3.4 Initial Calibration Verification Check Sample (ICV) -- A sample containing analytes of interest at known concentrations (true values) of analytes; this sample is run immediately after the calibration. The ICV is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.
- 3.5 Continuing Calibration Verification Check Sample (CCV) -- A sample of reagent water containing analytes of interest at known concentrations (true values) of analytes. This test ensures that the laboratory is meeting performance criteria during the time the samples are analyzed. This test is run after every 10 samples.
- 3.6 Initial Calibration Blank (ICB) -- A volume of reagent water in the same matrix as the calibration standards, but without the analyte run after the ICV.
- 3.7 Continuing Calibration Blank (CCB) -- A volume of reagent water in the same matrix as the calibration standards, but without the analyte and is run after the CCV.
- 3.8 Matrix Spike (MS) -- An environmental sample to which analytes of interest at known concentrations have been added. This is performed to distinguish if the matrix of an unknown environmental sample has any positive or negative effect on the analytical results. The concentration of analytes in the parent sample must be determined first and the results of the parent sample becomes the background correction for the MS.
- 3.9 Matrix Spike Duplicate (MSD) -- Is an aliquot of the same environmental sample as that of the Matrix Spike to which known quantities of analytes are added in the laboratory. The MS and MSD are treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures. One MS and MSD sample is run per every 10 samples for method 200.7. For method 6010B one MS and one MSD is run for every 20 samples.
- 3.10 Analytical Spike or Post-Digestion Spike - addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed. Refer to SOP TP-AN-005: Definitions, Terms, and Acronyms and to the current revision of the Tampa's Quality Assurance Manual (TP-QAM) for a complete listing of applicable definitions.
- 3.11 The interference check solution (ICS) -- sometimes called Spectral Interference Check (SIC) is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest, particularly those with known interferences at 0.5 to 1 mg/L. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

4.0 Interferences

- 4.1 Spectral interferences are caused by (1) the overlap of a spectral line from another element, (2) unresolved overlap of molecular band spectra, (3) background contribution from continuous phenomena, and (4) stray light from the line emissions of highly concentrated elements.
- 4.1.1 Spectral overlap may be compensated for by the use of inter-element correction factors.
 - 4.1.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.
- 4.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample digestate, by using a peristaltic pump, or by using the method of standards additions (MSA), or use of an internal standard.
- 4.3 Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

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

- 5.1.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- 5.1.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.
- 5.1.3 The samples are digested in strong acid solutions and contain acid concentrations of 4% by volume. The analyst must wear protective clothing such as a lab coat or apron. The acids used in this procedure will destroy unprotected clothing. The analyst must wear proper eye protection such as lab glasses or face shield. Acid can be splashed into the eyes from many sources. Gloves must be worn to protect hands from acid burns.
- 5.1.4 The acid digestion procedures must be performed under a properly functioning fume hood. The acid fumes from the digestion can cause mild to severe respiratory problems if breathed.
- 5.1.5 Each digestion lab must have acid spill kits. These kits must be located in a highly accessible area of the lab. Each digestion lab must be equipped with a properly working shower.

- 5.1.6 The standards and reagents used to prepare the standards in this method should be treated as potential hazards. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation. Lab coats, gloves, and other protective equipment should be used when preparing and using the standards and reagents.
- 5.1.7 Care must be taken when handling the digestion cups. Before handling a vessel that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion vessels are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the beakers to move and possibly to fall and splatter. On analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.
- 5.1.8 All work must be stopped in the event of a known or potential compromise to the health and safety of a Tampa associate. The situation must be reported **immediately** to a laboratory supervisor.
- 5.1.9 Always carry bulk concentrated acid bottles in appropriate impact proof containers.
- 5.1.10 Acid / peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.
- 5.1.11 Discard cracked or dirty digestion cups to prevent injury and or contamination. Dispose of the cracked or dirty vessels in the proper receptacle.
- 5.1.12 Any and all accidents and spills must be reported to the lab supervisor or EH&S coordinator.

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.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
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

- Thermo Jarrell Ash TJA ICAP61E-trace or other suitable inductively coupled plasma emission spectrometer with data system
- Autosampler
- Argon gas supply and appropriate fittings
- Peristaltic Pump
- Cooling Water Supply

6.2 Supplies

- Volumetric Flasks (Class A): 100 mLs; 200 mLs; 500 mLs; 1000 mLs
- Eppendorf Pipettes, varying volumes
- Pipettes, various sizes
- Digestion vessels: Disposable 50-ml digestion cups with volumetric gradations. Cups must come with analysis report per case of digestion cups. The Lot number must entered in the digestion sheet and the analysis report must be saved for future reference.

7.0 Reagents and Standards

Reagents are tracked in accordance with Tampa's most current revision of SOP TP-AN-004: *Standard Materials and Reagent Traceability*.

7.1 Reagents

- 7.1.1 Reagent water-lab generated deionized water, ASTM Type I or Type II. The conductivity monitored in accordance with Tampa's most current revision of SOP TP-AN-009 *Conductivity Checks For Laboratory Deionized Water*.
- 7.1.2 Nitric acid (HNO₃)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.
- 7.1.3 Hydrochloric acid (HCl)-reagent grade. The assay sheet of each lot of acid received into the lab must be reviewed to make sure that the quality of the acid is sufficient for trace analysis of metals.

7.2 Standards

- 7.2.1 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. All preparation steps must be in accordance with Tampa's most current revision of SOP TP-AN-004: *Standard Materials and Reagent Traceability*, it contains guidance for the preparation of standards.
- 7.2.2 Recommended concentrations for the calibration standards are given in Table 1. Appendix A contains examples for the preparation of the initial calibration and calibration verification standards for both 6010 and 200.7. If the laboratory uses preparations other than those listed in Appendix A, the preparation must be documented in the standard material traceability logbook, the standards and reagent module in LIMS or as a controlled posting. All standards **must** be prepared in 4% hydrochloric acid and 4% nitric acid by volume.

NOTE: Standards must be prepared every six months "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

7.3 Preparation of the Linearity Check Solutions

The linearity check solutions are prepared individually according to the following equation:

$$V_s = \frac{V_{lc} \otimes C_{lc}}{C_s}$$

where

V_s = volume of stock standard (mL)

C_s = concentration of stock standard (mg/L)

V_{lc} = volume of linearity check standard to prepare (mL)

C_{lc} = concentration of linearity check standard to prepare (mg/L)

The linearity check solutions are prepared at the concentrations specified in Table 1. Prepare sufficient volume to perform the linearity check, maintaining the hydrochloric acid concentration at 4% by volume and the nitric acid concentration at 4% by volume.

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	Reference
Waters ²	HDPE	250 mLs 500 mLs	HNO ₃ , pH < 2;	180 Days	40 CFR Part 136.3
Soils	Glass or HDPE	3 grams	Cool 0-6°C	180 Days	N/A
Leachates (TCLP, SPLP, EP Tox)	HDPE	250 mLs	HNO ₃ , pH < 2	180 Days	N/A
Wastes	HDPE	250 mL or 3 grams	Cool 0-6°C	180 Days	N/A

¹ Inclusive of digestion and analysis.

² Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

9.0 Quality Control

9.1 Tampa's most current revision of SOP TP-AN-006 *Analytical Batching and Evaluation of Data* provide guidance on evaluating QC and sample data, including recommended corrective actions.

9.2 The method detection limit (MDL) is determined annually in accordance with Tampa's most current revision of SOP TP-CA-090 *Procedure for the Determination of Method Detection Limit*. The concentrations of the IDL and MDL solutions are given in Appendix B of this SOP.

9.3 Determination of the Instrument Detection Limit (IDL)-The difference between the MDL and the IDL is the *digestion step*. The MDL samples are prepared and digested prior to analysis. The IDL is defined as three times the standard deviation of seven replicate analyses analyzed over three non-consecutive days. The concentrations of the IDL and MDL solutions are given in Appendix B of this SOP. See Tampa's most current revision of SOP TP-CA-091 *Determination of Instrument Detection Limit* for the procedures for the determination of the IDL.

9.4 The linear range of the ICP must be determined at least annually. The procedure for the determination is given in Section 9.8.8 of this SOP. If any calibration regression fit, other than linear, is utilized for the calibration of the ICP (i.e., Curvilinear or Full Fit), the upper limit of the linear range is the concentration of the High Standard

9.5 Interelement correction factors (IEC) for all elements must be determined annually. Use the manufacturer's guidance for determination of the IECs. The IECs must be

verified at the beginning of each analytical sequence.

9.6 The SOP Summary in Appendix A includes the acceptance criteria for QC, including recommended corrective actions. The analytical sequence, including standardization and calibration verification, is included in the SOP Summary in Appendix A.

9.7 Procedure

9.7.1 Sample Preparation

The sample preparation and digestion procedures are listed in the following SOPs:

MATRIX	SOP
Aqueous and leachate samples	TP-ME-007; TP-ME-004
Soils and Sediments	TP-ME-009
Wastes and oils	TP-ME-009

9.7.2 Calibration

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. Preparation of all reference materials used for calibration must be documented.

9.7.2.1 Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If optics were turned off, allow 2 hours warm up time.

9.7.2.2 Run the "Automatic Profile" program. The "automatic profile" of the instrument should be checked twice a day to compensate for changes in air pressure, humidity, and temperature. If the environment of the instrument is such that daily changes in the instrument profile are extreme, the instrument should be "profiled" every few hours.

9.7.2.3 Analyze the calibration standards and calibrate the ICP. ***If using a multi-point calibration, use the Calibration/Analysis and Curvefit programs to calibrate the instrument.***

9.7.2.4 The QC Check standards (ICV) and the Calibration Blank (ICB) are analyzed as a check on the instrument calibration.

9.7.2.4.1 (EPA Method 6010) The results for the target compounds in the initial calibration verification (ICV) must be within +/- 10 % of the true value.

9.7.2.4.2 (EPA Method 200.7) The results for the target compounds in the initial calibration verification (ICV) must be within +/-5.0 % of the true value for 200.7. A fresh solution should be prepared quarterly (every 3 months).

9.7.2.4.3 (EPA 6010/200.7) The results for the target compounds in the initial calibration blank (ICB) must be less than the RL.

9.7.2.5 The ICP Interference Check Sample is analyzed. The concentrations of the target analytes must be within 20% of the true concentrations. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

9.7.2.6 Continuing Calibration Verification (CCV)

9.7.2.6.1 The calibration of the ICP must be verified after every 10 samples by the analysis of the QC Check Solutions (CCV) and the Calibration Blank (CCB).

9.7.2.6.2 (EPA Method 6010/200.7-DW) The results for the target compounds in the continuing calibration verification (CCV) must be within +/- 10 % of the true value.

9.7.2.7 (EPA 6010/200.7) The results for the target compounds in the continuing calibration blank (CCB) must be less than the Reporting Limit (RL).

9.7.2.8 ICP Interference Check Solution is analyzed at the beginning and end of each analytical sequence.

9.8

Sample Analysis

9.8.1 The instrument is calibrated and standardized according section 9.7 which is outlined above prior to sample analysis.

9.8.2 The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

9.8.3 The samples are analyzed in a sequence as follows:

INSTRUMENT WARM-UP
PROFILE
INITIAL CALIBRATION (STANDARDIZATION/CALIBRATION OF THE ICP)
INITIAL CALIBRATION VERIFICATION (ICV)
INITIAL CALIBRATION BLANK (ICB)
DETECTION LIMIT CHECK SOLUTION (CRI/RL)
REANALYSIS OF HIGH CONCENTRATION CALIBRATION STANDARD AS A SAMPLE
ICP INTERFERENCE CHECK SOLUTION A (ICSA)
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)
10 SAMPLES
CCV
CCB
10 SAMPLES
CCV
CCB

10 SAMPLES
CCV
CCB

The analytical sequence must end with the analysis of the detection limit check standard, CCV, CCB, and ICSA. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

9.8.4 Determine the concentration of the samples and QC items using the procedures of Section 9.

9.8.4.1 If the concentration of a sample is above the linear range of the ICP, the sample digestate must be diluted and reanalyzed.

9.8.4.2 The amount of sample digestate needed to prepare the desired dilution is determined from the following equation:

$$V_{digest} = \frac{V_{f_{vol}}}{DF}$$

where

$V_{f_{vol}}$ = final volume of diluted sample (mL)

V_{digest} = volume of sample digestate used to make the dilution (mL)

9.8.4.3 The dilution factor is calculated as follows:

$$DF = \frac{V_{f_{vol}}}{V_{digest}}$$

where

$V_{f_{vol}}$ = final volume of diluted sample extract (mL)

V_{digest} = volume of sample extract used to make the dilution (mL)

NOTE: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE:

A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$DF = \frac{100mL}{1.0mL} = 100$$

Dilutions must be prepared in reagent water containing 4% hydrochloric acid and 4% nitric acid by volume.

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

9.8.5 Dilution QC Check

- 9.8.5.1 A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present. This is required for CLP work and for new or unusual matrices
- 9.8.5.2 Select a sample digestate that contains one or more target analytes at a concentrations greater than 10X the reporting limit.
- 9.8.5.3 Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.
- 9.8.5.4 Compare the results of the diluted and un-diluted aliquots of sample digestate.
- 9.8.5.5 If the results of the dilution are within $\pm 10\%$ of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than $\pm 10\%$, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike (see section 9.8.6). ***If the concentration of the analyte in the sample is not at least 50 times the instrument detection limit, evaluate the post-digestion spike.***

9.8.6 Post-digestion Spike QC Check

A post-digestion spike may be performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. This should be the same sample selected for dilution in section 9.8.5, above. This is required for CLP work and for new or unusual matrices.

- 9.8.6.1 Spike the sample with 0.1 mL of QC7; 0.1mL of QC23, and 0.018mL Na into 9.78 mls of digestate. The theoretical concentration of the post digestion spike is the same as the LCS or MS if the volume of spiking solution is discounted.
- 9.8.6.2 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).
- 9.8.6.3 Calculate the percent recovery of the post digestion spike:

$$\%REC = \frac{C_{ps} - C_s}{C_2} \times 100$$

Where

C_{ps} = concentration of post digestion spike (ug/L)
C_s = concentration of un-spiked sample (ug/L)
C₂ = theoretical concentration of spike (ug/L)

- 9.8.6.4 Evaluate the recovery using the following decision matrix. Limits for post digestion spikes are 75-125% recovery.

Result of Post Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% recovery but >50% recovery	1) Dilute and re-spike. Elevate RL accordingly (for all associated samples). 2) Spike and evaluate all associated samples. 3) Spike and evaluate all associated samples by single point MSA 4) Qualify all associated samples
<50% recovery	Dilute digestate and repeat spike. <i>Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples)</i> If recoveries are not 75-125%, analyze all associated samples by single point MSA. Note – high level of target analytes may inhibit spike recovery. Consult the supervisor in events where high levels of targets appear to be interfering

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

9.8.6.5 The post digestion spike and the method of standard additions must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgement when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyzed to determine the extent of the matrix interferences.

9.8.7 Single Point Method of Standard Additions

Two identical aliquots of the sample digest, V_x , are taken. One aliquot is spiked with a solution of known concentration, C_s . The second aliquot is analyzed un-spiked (the small volume of standard added to the spiked sample should be disregarded). The concentrations of both aliquots are measured and the sample concentration, C_x , is calculated:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

where

S_1 = absorbance or concentration of the spiked aliquot

S_2 = absorbance or concentration of the un-spiked aliquot

V_s = Volume of spike solution

Example: Sample concentration (S_2): 523 ug/L.
 Spike solution concentration (C_s): 50,000 ug/L
 Volume of spike solution (V_s): 0.10mL
 Volume of sample aliquots (V_x): 10mL
 Spiked sample concentration (S_1): 951 ug/L

$$C_x = [(523) \cdot (0.10) \cdot (50,000)] / [(951 - 523) \cdot 10] = [2,615,000] / [4280] = 611 \text{ ug/L}$$

9.8.8 Determination of Linear Range of the ICP

9.8.8.1 The linear range is described as the highest point where one can get a true recovery that is within 5 % of the true value. In other words, one point inside the range, one point at the end of the range and one point outside the range (so far outside that it fails).

9.8.8.2 The linear range must be determined a minimum of once per year. Divisions performing CLP analyses are required to determine and document the linear range quarterly. Documentation of the linear range study must be kept on hand and be available for inspection. A summary of the linear range study must be available to the bench analyst.

9.8.8.3 Profile and calibrate the ICP as described in Section 9.7.

9.8.8.4 Prepare individual standards at concentrations that are expected to define the linear range of the instrument. Use the concentrations in Table 1 for guidance. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids.

9.8.8.5 Analyze the standards following the analytical sequence described in Section 14.3.2. Verify the calibration after every 10 analyses.

9.8.8.6 Compare the concentration of the linear range standard with its true concentration.

$$\text{PercentDifference} = \left| \frac{C_{cal} - C_{true}}{C_{true}} \right| \otimes 100$$

where

C_{cal} = concentration determined from analysis

C_{true} = true concentration of the standard

If the percent difference is less than or equal to 5%, the linear range is confirmed at that concentration. If the percent difference is greater than 5%, repeat the analysis with a lower concentration.

The linear range may be extended by analyzing higher standards and evaluating the results against the 5% difference criterion. The linear range of the ICP for an analyte is the highest standard of that analyte that meets this criterion.

10.0 Calculations / Data Reduction

10.1 Aqueous and Leachate Samples

Aqueous samples are routinely reported in mg/L while the ICP is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

10.1.1 The concentration of the target analyte in liquid samples is calculated as follows:

$$\text{Concentration (mg/L)} = \text{ug/L (from printout)} \otimes \frac{F}{V} \otimes DF \otimes \frac{1\text{mg}}{1000\text{ug}}$$

where

F = final volume of the sample digestate (L)-usually 50mL (0.050L)

V = volume of sample digested (L)

DF = dilution factor

10.1.2 The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

$$\text{Concentration (mg/L)} = \text{RLqap} \otimes \frac{F}{V} \otimes DF \otimes \frac{1\text{mg}}{1000\text{ug}}$$

where

RLqap = reporting limit from TA LQM (ug/L)

F = final volume of the sample digestate (L)

V = volume of sample digested (L)

DF = dilution factor

NOTE: The LIMS Reporting Limits assumes:

F = 50mL, V = 50mL, and DF = 1

10.2 Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

10.2.1 The concentration of the target analyte in soil and solid samples is calculated as follows:

$$\text{Concentration}(\text{mg/kg, dw}) = \text{ug/L}(\text{from printout}) \otimes \frac{F}{W \otimes \text{solids}} \otimes DF \otimes \frac{1\text{mg}}{1000\text{ug}}$$

where

F = final volume of the sample digestate (L)

W = volume of sample digested (kg)

DF = dilution factor

solids = decimal equivalent of the percent solids (percent solids/100)

(for example, if the percent solids is 85%, the decimal equivalent is 0.85; if the %solids is 100%, the decimal equivalent is 1.0.)

10.2.2. The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$\text{Concentration}(\text{mg/kg, dw}) = RL_{qap} \otimes \frac{0.0010\text{kg}}{W \otimes \text{solids}} \otimes \frac{F}{0.100\text{L}} \times DF$$

where

RL(qap) = reporting limit from LQM

W = weight of sample digested (kg)

F = final volume of the sample digestate (L)

V = volume of sample digested (L)

DF = dilution factor

solids = decimal equivalent of the percent solids (percent solids/100)

NOTE: The LIMS Reporting Limits assumes:

F = 0.100L (100mL), DF = 1, W = 0.0010kg (1.0g), and solids = 1.0

10.3 Accuracy

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

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

10.4 Precision (RPD)

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

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

11.0 Method Performance

11.1 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 Section 20 of 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.

11.2 Demonstration of Capabilities - Each analyst must perform an Initial Demonstration of Capability (IDOC) in accordance with the procedure outlined in Section 20 of 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.

11.3 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 Section 18 of the Quality Assurance Manual.

11.4 The SOP Summary in Appendix A includes the acceptance criteria for QC, including recommended corrective actions. The analytical sequence, including standardization and calibration verification, is also included in the SOP Summary in Appendix A.

12.0 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 Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.0 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 Tampa's current revision of SOP TP-HAZ-001 *Waste Management*). The following waste streams are produced when this method is carried out.

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

- Acidic waste containing nitric acid generated by the extraction: Acid waste is to be neutralized and poured down the drain.

14.0 Data Assessment And Acceptance Criteria For Quality Control Measures

14.1 See Attachments for all quality control acceptance criteria.

15.0 Corrective Actions For Out Of Control Data

15.1 See Attachments for corrective action procedures.

16.0 Contingencies For Handling Out Of Control Data

16.1 When QC results, unknown positives, or sample matrix present the analyst with questionable data, the spike results, sample matrix, and reported positives in the batch are all to be considered. Acceptability may be determined by citing historical data on a case by case basis. The project manager must be notified of any method anomalies, and can then advise the client on the usability of the data and any other necessary actions.

17.0 References / Cross-References

17.1 *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development, Cincinnati, OHIO, March 1983.

17.2 *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response; Washington, D.C., November 1986.

17.3 *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.

17.4 SW-846 Method 60105 Inductively Coupled Plasma-Atomic Emission Spectrometry Revision 3 November 2000.

17.5 Tampa's Quality Assurance Manual (TP-QAM), current revision.

17.6 Corporate Environmental Health and Safety Manual (CW-E-M-001), current revision.

17.7 TestAmerica Tampa SOP's:

17.7.1 TP-CA-090 *Procedure for the Determination of Method Detection Limit.*

17.7.2 TP-CA-091 *Determination of Instrument Detection Limit*

17.7.3 TP-HAZ-001 *Waste Management*

17.7.4 TP-AN-004: *Standard Materials and Reagent Traceability.*

17.7.5 TP-AN-005: *Definitions, Terms, and Acronyms*

17.7.6 TP-AN-006 *Analytical Batching and Evaluation of Data*

17.7.7 TP-AN-009 *Conductivity Checks For Laboratory Deionized Water.*

17.7.8 TP-ME-004 *TCLP Non-Volatiles Extraction (EPA 1311)*

17.7.9 TP-ME-007 *Digestion Procedure for ICP: Total Metals and Total Recoverable Metals in Liquid Samples (EPA 3005/3010)*

17.7.10 TP-ME-009 *Digestion Procedure For ICP: Total Metals In Soils, Sediments, Wastes, and Tissue Samples (EPA 3050)*

18.0 Method Modifications:

Not applicable to this SOP.

19.0 Attachments

Attachment 1: Troubleshooting and Preventive Maintenance
Table 1: Analyte List, Wavelengths, Standard & QC Concentrations
Appendix A: SOP Summary
Appendix B: Examples of Standard Preparations
Appendix C: Sample Flow Chart

20.0 Revision History

- Revision 3, dated 15 May 2009
 - Updated to TestAmerica format, removed all STL logo's, nomenclature, etc.
 - Updated references, added TestAmerica SOP's to reference section
- Revision 2, dated 28 June 2008
 - Added a definition for Preparation Batch, Fixed definition for Analytical batch, Removed Dup definition and added MSD definition, Edited CCV definition, Edited definition of CCB, 6.9 edited definition of MS, 8.3 Changed the percent acid added from 10% to 4%, 8.7.3-8.7.6 deleted due to it being repetitiveness and redundancy, 12.5 changed line to reflect that the IEC std is not run at the end of the analytical run, 13.5- changed percent recovery of PQL from 50% to 30% as per method, 13.5.2 Changed frequency of ICV makeup from daily to quarterly, 13.6 changed paragraph to reflect analytical run, 13.4-13.7 Changed the paragraph order to reflect analytical run, 13.10 changed line to reflect the fact that the RL std is not analyzed at the end of the run, 14.3.2 made changes to reflect analytical run, 14.5.2 Corrected amount of spike to add to the post digestion spike, 22.0 Changed frequency of replacement of peristaltic pump tubing from daily to as needed, Table 1 changed values for ICV/CCV/RL; made changes to appendix C flowchart to reflect analytical run, 21.4 Added SW-846 Method 6010B Inductively Coupled Plasma-Atomic Emission Spectrometry Revision 3 November 2000.

Attachment 1.

TROUBLESHOOTING AND PREVENTIVE MAINTENANCE

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	S A	A	AN	
ICAP								
Pump Tubing							x	Change.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch							X	Clean and realign.

D = daily W = Weekly M = monthly Q = Quarterly SA = semi-annually A = annually AN = as needed

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Aluminum (Al)	308.215	50	1.0/25	0.20	800	1.0	100
Antimony (Sb)	206.838	2.0	1.0/1.0	0.006	10	1.0	100
Arsenic (As)	189.042 193.696	2.0	1.0/1.0	0.008	25	1.0	100
Barium (Ba)	493.409	5.0	1.0/2.5	0.010	10	1.0	100
Beryllium (Be)	313.042	2.0	1.0/1.0	0.0050	10	1.0	100
Boron (B)	249.678	2.0	1.0/1.0	0.050	100	1.0	100
Cadmium (Cd)	226.502 228.802	2.0	1.0/1.0	0.0050	10	1.0	100
Calcium (Ca)	317.933 315.887	50	1.0/25.0	0.50	800	40.0	4000
Chromium (Cr)	267.716	5.0	1.0/2.5	0.010	25	1.0	100
Cobalt (Co)	228.616	5.0	1.0/2.5	0.010	25	1.0	100
Copper (Cu)	324.754	5.0	1.0/2.5	0.020	50	1.0	100
Iron (Fe)	259.940 271.441	50	1.0/2.50	0.050	800	10.0	1000
Lead (Pb)	220.353	2.0	1.0/1.0	0.0030	5	1.0	100
Magnesium (Mg)	279.079	50	1.0/25.0	0.50	1000	1.0	100

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Std. Conc. (mg/L)	Linear Range Std. Conc. (mg/L)*	MATRIX SPIKE CONC. (mg/L)	
						Water (mg/L)	Soil (mg/kg)
Manganese (Mn)	257.610	5.0	1.0/2.5	0.010	50	1.0	100
Molybdenum (Mo)	202.030	5.0	1.0/2.5	0.010	50	1.0	100
Nickel (Ni)	231.604	5.0	1.0/2.5	0.040	10	1.0	100
Potassium (K)	766.491	50	25.0/25.0	1.0	50	10.0	1000
Selenium (Se)	196.026	2.0	1.0/1.0	0.005	25	1.0	100
Silver (Ag)	328.068	2.0	1.0/1.0	0.010	5.0	1.0	100
Sodium (Na)	588.995 330.231	10	10.0/5.0	0.50	20	10.0	1000
Strontium (Sr)	421.552	2.0	1.0/1.0	0.010	100	1.0	100
Thallium (Tl)	189.042 190.801 377.572	2.0	1.0/1.0	0.010	30	1.0	100
Tin (Sn)	189.989	5.0	1.0/2.5	0.050	50	1.0	100
Titanium (Ti)	334.941	5.0	1.0/2.5	0.010	10	1.0	100
Vanadium (V)	292.402	5.0	1.0/2.5	0.010	50	1.0	100
Zinc (Zn)	213.856 206.200+	5.0	1.0/2.5	0.020	20	1.0	100

*For guidance only-instrument sensitivity will vary.

APPENDIX A**SOP SUMMARY****METHOD SUMMARY - ICP ANALYSIS****HOLD/STORAGE**

Container	Minimum 250mL plastic bottle with plastic or Teflon-lined lid
Preservative	HNO ₃ to pH <2 in the field. If dissolved metals are required, filter the samples before preservation.
Storage	Liquids preserved to pH <2 may be stored at room temperature until preparation. Solid samples must be stored at 4C (less than 6C but not frozen) until preparation.
Hold Time	Samples must be analyzed within six months from the time of collection.

SAMPLE PREPARATION

Samples should be prepared with the appropriate matrix-specific procedure.

ANALYTICAL SEQUENCE

Ignite Plasma	Follow instrument manufacturer's guidelines and allow instrument to stabilize for at least 60 minutes.
Profile Instrument	Follow manufacturer's guidelines.
Initial Calibration	Calibrate with a blank and a high standard or a blank and three standards. Verify calibration by reanalyzing highest concentration standard for each element.
Initial Calibration Verification (ICV/ICB)	Analyze an initial calibration verification solution at the beginning of the run. ICV solution must come from a source other than the calibration standard source. Analyze a calibration blank after the ICV.
Continuing Calibration Verification (CCV/CCB)	Analyze a standard with concentrations at or near mid-range levels of the calibration. The CCV should be analyzed every 10 samples and at the end of the analysis run. Analyze a continuing calibration blank after every CCV.
Interference Check Solutions	At the beginning and the end of an analysis run, verify the inter-element and background corrections by analyzing the interferent check solutions (ICSA).
Detection limit check solution	At the beginning and the end of an analysis run and verify the accuracy at the RL by analyzing a solution at the RL.
Serial Dilution	Perform serial dilution (1/5) on a representative sample from each batch. This is required for CLP work and for new or unusual matrices.
Post Digestion Spike Recovery.	To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). The post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration in the sample is not at least 50 times the instrument detection limit. This is required for CLP work and for new or unusual matrices.

UNCONTROLLED

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration: Multi-point- minimum 3 stds and 1 blank	Daily	Correlation ≥ 0.995	Recalibrate
Highest Standard	Immediately after every calibration	Recoveries within $\pm 10\%$ of expected values	New initial calibration
Initial Calibration Verification Standard (ICV), second source	At the beginning of the analysis	SW846 = within $\pm 10\%$ 200.7 = within $\pm 5\%$	Recalibrate
Continuing Calibration Verification Standard (CCV) and (LLCCV)	At the beginning and end of the analysis, and every 10 samples (not required for LLCCV, unless low levels expected)	SW846 = within $\pm 10\%$ (CCV) SW846 = within $\pm 20\%$ (LLCCV) 200.7-NPDES - within $\pm 5\%$ 200.7-Drinking Water - within $\pm 10\%$	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the RL/CRDL	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA)	At the beginning and end of an analysis run	Determined values must be within $\pm 20\%$ of the true values. Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Lab control sample	One per batch of twenty samples or less	6010B: Tampa's TP-QAM 200.7: 85-115%	Redigest and reanalyze batch
Preparation blank - SW846	One per batch of twenty samples or less	result < RL or result < 5% of the analyte level in the sample.	Redigest and reanalyze batch
Preparation blank - 200.7	One per batch of twenty samples or less	result < RL or result < 10% of the analyte level in the sample	Redigest and reanalyze batch
MS/MSD	One set per batch of twenty samples or less: SW846 MS every ten samples: 200.7	SW846 = within $\pm 25\%$ SW846 = Dupes within $\pm 20\%$ 200.7 = within $\pm 30\%$	Flag and report data
Serial Dilution (1/5 Dilution)	One per batch of twenty samples or less	See section 14.4	
Post Digestion Spike	One per batch of twenty samples or less	See section 14.5	

APPENDIX B

EXAMPLES OF STANDARD PREPARATION

GENERAL INSTRUCTIONS

All calibration standards must contain 4% hydrochloric acid and 4% nitric acid by volume. The following table lists the volume of each acid needed to prepare the desired final volume of standard.

Final Volume of Standard (mL)	Volume of Hydrochloric acid (mL)	Volume of Nitric Acid (mL)
100	4.0	4.0
200	8.0	8.0
500	20.0	20.0
1000	40.0	40.0

For example, to prepare 500mL of a standard:

- Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask.
- Add 20.0mL of concentrated nitric acid (HNO_3) and 20mL of hydrochloric acid (HCl) to the volumetric flask.
- Add the volumes of the stock standards given in the table to the volumetric flask.
- Dilute to a final volume of 500mL with reagent water. Store the standard at room temperature.

CALIBRATION

The ICP must be calibrated with a minimum of high standard and a blank. The following standards may be used for this purpose. With the Thermo Jarrell Ash software the Calibration Analysis and Curve-fit programs must be used to be successful with the calibration of the ICP instruments.

High Standard.

Element/Stock	Conc. of Stock Std (mg/L)	mL of Stock Std	Final Volume (mL)
ICPA CAL #1	(1)	10	500
ICP TPA #2	(1)	2.5	
ICP CAL #4	(1)	2.5	
ICP TPA #5	(1)	2.0	
Silver (Ag)	1000	1.0	
Antimony (Sb)	1000	1.0	
Element	Conc. of Cal Std (mg/L)		
Aluminum (Al)	50		
Antimony (Sb)	2.0		
Arsenic (As)	2.0		
Boron (B)	2.0		
Barium (Ba)	5.0		
Beryllium (Be)	2.0		
Cadmium (Cd)	2.0		
Calcium (Ca)	50		
Cobalt (Co)	5.0		
Chromium (Cr)	5.0		
Copper (Cu)	5.0		
Iron (Fe)	50		
Lead (Pb)	2.0		
Magnesium (Mg)	50		
Manganese (Mn)	5.0		
Molybdenum (Mo)	5.0		
Nickel (Ni)	5.0		
Potassium (K)	50		
Selenium (Se)	2.0		
Silver (Ag)	2.0		
Sodium (Na)	10		
Strontium (Sr)	2.0		
Thallium (Tl)	2.0		
Tin (Sn)	5.0		
Titanium (Ti)	5.0		
Vanadium (V)	5.0		
Zinc (Zn)	5.0		

(1) The solutions containing multiple elements. The concentrations are given on the certificate of analysis.

Initial Calibration Verification (ICV) Solution

Element/Stock	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of ICV Std (mg/L)
SPEX QC 23	(1)	2.0	200	(2)
SPEX QC 7	(1)	2.0		(2)
Potassium (K)	10000	0.30		50(3)
Sodium (Na)	10000	0.18		20(3)

- (1) SPEX QC23 and SPEX QC7 are solutions containing multiple elements. The concentrations are given on the certificate of analysis.
- (2) The final concentrations of the various elements are the same as listed in Table 1. The SPEX QC solutions are diluted by a factor of 100 from the concentration listed on the certificate of analysis.
- (3) These concentrations include the contribution from SPEX Solutions QC-7

Continuing Calibration Verification (CCV) Standard

Element	mL of High Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
Aluminum (Al)	200	400	25
Antimony (Sb)			1.0
Arsenic (As)			1.0
Boron (B)			1.0
Barium (Ba)			2.5
Beryllium (Be)			1.0
Cadmium (Cd)			1.0
Calcium (Ca)			25
Cobalt (Co)			2.5
Chromium (Cr)			2.5
Copper (Cu)			2.5
Iron (Fe)			25
Lead (Pb)			1.0
Magnesium (Mg)			2.5
Manganese (Mn)			2.5
Molybdenum (Mo)			2.5
Nickel (Ni)			2.5
Potassium (K)			25
Selenium (Se)			1.0
Silver (Ag)			1.0
Sodium (Na)			5.0
Strontium (Sr)			1.0
Thallium (Tl)			1.0
Tin (Sn)			2.5
Titanium (Ti)			2.5
Vanadium (V)			2.5
Zinc (Zn)			2.5

Reporting Limit (RL) Check Standard

Preparation of RL/PQL Check Standard (EXAXOL Custom Stock)

Element	Conc. of Stock Std (Mg/L)	mL of Stock Std	Final Volume (mL)	Conc. of Check Std (ug/L)
Silver (Ag)	4.0	0.50	500	4.0
Arsenic (As)	10			10
Cadmium (Cd)	4.0			4.0
Copper (Cu)	10			10
Nickel (Ni)	8.0			8.0
Lead (Pb)	5.0			5.0
Selenium (Se)	10			10
Thallium (Tl)	10			10
Aluminum (Al)	200			200
Boron (B)	50			50
Barium (Ba)	10			10
Beryllium (Be)	2.0			2.0
Calcium (Ca)	500			500
Cobalt (Co)	10			10
Chromium (Cr)	10			10
Iron (Fe)	100			100
Magnesium (Mg)	80			80
Manganese (Mn)	4.0			4.0
Molybdenum (Mo)	15			15
Sodium (Na)	500			500
Antimony (Sb)	10			10
Tin (Sn)	50			50
Vanadium (V)	10			10
Zinc (Zn)	20			20
Potassium (K)	1000			1000
Strontium (Sr)	5.0			5.0
Titanium (Ti)	10			10

ICP Interference Check Solutions

Preparation of ICP Interference Check Solution (Elements Custom Stock)

Element	Conc. Of Stock (mg/L)	mL of Stock Std	Final Volume (mL)	Conc. (mg/L)
Aluminum (Al)	5000	50	500	500
Calcium (Ca)	5000			500
Magnesium (Mg)	5000			500
Iron (Fe)	2000			200

ICP Matrix Spiking Solutions

ICP Matrix Spiking Solutions are solutions purchased from Elements. The certificate of analysis will list the concentrations of the analytes. Store this solution at room temperature.

Preparation of ICP Matrix Spike Solution

Stock	mL of Stock Std	Final Volume(mL)
23 Element QC Stock 100 ppm	0.5	50
7 Element QC Stock 100/1000(K) ppm	0.5	
Sodium (Na) 10,000 ppm	0.045	

APPENDIX C SAMPLE FLOW CHART

