
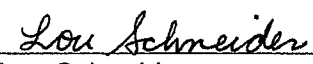


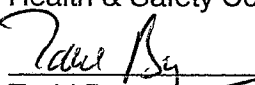
**Title:** **ICP ANALYSIS**  
**(METHODS 200.7 AND 6010B)**

**Approvals (Signature/Date):**

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## 1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes the procedures to determine the concentration of various elements by inductively coupled plasma (ICP) atomic emission spectroscopy. This method contains the analytical procedures for the determination of metals in aqueous liquids (water), surface and groundwaters, wastewaters, SPLP leachates, soils, sediments, waste samples, biological tissues, and oils after digestion. This SOP is based on the guidance provided in SW-846 method 6010B and EPA method 200.7. Prior to analysis liquid samples must be digested in accordance with SOP TL-ME-050: *Digestion Procedures for ICP: Total Metals and Total Recoverable Metals in Aqueous Samples (Methods 3005A, 3010A, and 200.7)*. Prior to analysis solid samples must be digested in accordance with SOP TL-ME-051: *Digestion Procedures for ICP: Total Metals in Soils, Sediments, Wastes, Tissues, and Oils (Method 3050B)*.
- 1.2 The elements and the characteristic wavelength to be analyzed by this SOP are listed in Table 1.
- 1.3 The reporting limit (RL) for each element is listed in document TL-QA-001, *TestAmerica Tallahassee Method/Analyte List*, current revision. The method detection limit (MDL) for each element is listed in the current revision of document TL-QA-002, *TestAmerica Tallahassee Quality Control Limit Summary*. The RLs and MDLs are also listed in Work Instruction WME003, current revision.

## 2.0 SUMMARY OF METHOD AND DEFINITIONS

- 2.1 Sample digestates are aspirated and nebulized into a spray chamber. 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.
- 2.2 Definitions
- 2.2.1 **ICP** -Inductively Coupled (Argon) Plasma; sometimes referred to as an "ICAP"
- 2.2.2 **Analytical Spike** - Addition of a known concentration of analytes to an aliquot of sample after the preparation steps have been performed; also called a post digestion spike
- 2.2.3 **SPLP**- Synthetic Precipitation Leaching Procedure
- 2.2.4 **RL Standard**- Reporting Limit Standard, the lowest calibration standard or the equivalent of the lowest standard; published in LQM or project-specific quality assurance plan (QAPP); also referred to as the practical quantitation limit (PQL).
- 2.2.5 **PQL**- Practical Quantitation Limit, same as RL Standard
- 2.2.6 **MDL**- Method Detection Limit, the concentration that can be reported with 99% confidence that the result is greater than zero; published in LQM
- 2.2.7 **LQM**- Laboratory Quality Manual
- 2.2.8 **MSDS**- Material Safety Data Sheets
- 2.2.9 **DI Water**- Deionized Water

2.2.10 **Regulatory Threshold Limit-** The concentration of analyte in the sample at which the sample is deemed hazardous.

### 3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.

#### 3.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

3.1.1 Use good common sense when working in the lab. Do not perform any procedures that you do not understand or that will put you or others in potentially dangerous situations.

3.1.2 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.

3.1.3 Each reagent, standard, and sample must be treated as a potential health hazard. Lab coats, gloves, safety glasses, and other protective equipment should be used when preparing and using the reagents, standards, and samples.

3.1.4 The Material Safety Data Sheets (MSDS) for each reagent and standard are located in each laboratory. These sheets denote the type of hazard that each reagent poses, the safe handling instructions for these compounds, and first aid instructions.

#### 3.2 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 (1)	Hazards	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

## 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 use of an internal standard.

## 5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

### 5.1 Aqueous Samples

- 5.1.1 Liquid samples are collected in 500mL plastic or glass containers. The samples are preserved with  $\text{HNO}_3$  to a pH <2. Samples may be delivered to the lab unpreserved and the lab personnel may add the preservative; however, if this is done, the samples MUST be allowed to sit for 24 hours before the digestion is begun. This is to allow any metals which have adhered to the bottle to be redissolved into the water sample. If samples come in with inadequate preservation (e.g. pH 3 or 4), lab staff must add sufficient acid to reduce the pH to <2. The lab preservation time and the digestion start time must be documented in the LIMS prep batch to demonstrate the 24-hour waiting period. Upon lab preservation, an NCM must be generated using the NCM type "preservation – Metals 24 hrs wait time". The NCM must contain the initial pH, amount of acid added, acid lot #, bottle lot#, time preservative was added, and the time when samples may be digested. When commencing digestion, transfer the information to the LIMS prep batch. Check all pHs prior to digestion; the initial and final pH will be recorded as <2 in the prep batch.

Samples must be digested and analyzed within 6 months of collection.

- 5.1.2 SPLP leachate extracts are extracted in accordance with SOP TL-ME-017: *Synthetic Precipitation Leaching Procedure (SPLP): Semivolatile Organic Compounds and Metals*. The SPLP leachate samples are preserved in a 250mL plastic container with  $\text{HNO}_3$  to a pH <2.

Note: The SPLP leachate aliquots designated for matrix spikes must be spiked prior to addition of the acid preservative.

The SPLP extraction must be performed within 6 months of sample collection. The analysis of the leachate must be completed within 6 months of the leaching procedure.

- 5.1.3 Samples for dissolved metals should be filtered in the field before  $\text{HNO}_3$  acid is added to the sample. If the sample is to be filtered in the lab, no  $\text{HNO}_3$  acid preservative is added to the sample until the sample is filtered. Ideally, the samples are to be filtered and preserved upon receipt. If this is not possible, the sample is stored at 4 °C (less than 6 °C, but not frozen) until filtration and preservation. A lab blank must be subjected and documented to all processes to which the associated samples are subjected, specifically predigestion, filtration, and preservation. Lab-filtered samples are filtered into preserved bottles and the acid and bottle lot numbers are recorded in the LIMS prep batch. Lab-filtered samples for dissolved metals do not have to sit for 24 hours prior to digestion.

Filtered samples must be digested and analyzed within 6 months of collection

## 5.2 Solid Samples

5.2.1 Soils, sediments, waste samples, biological tissues, and oils samples are routinely collected in 500mL plastic containers. The sample is iced at the time of collection and is stored in the lab at room temperature until the time of digestion and analysis. The sample must be digested and analyzed within 6 months of collection.

## 5.3 Sample pH Check

5.3.1 The pH of all preserved liquid samples must be checked and documented prior to digestion and documented in the LIMS prep batch. If the pH is not within the proper range, additional HNO<sub>3</sub> acid is added to the sample to bring the pH below 2:

5.3.2 With a disposable transfer pipet, transfer a few drops of the sample onto a narrow range pH paper and note the color change. If the pH <2, record in the LIMS prep batch, and transfer the sample to the storage area.

5.3.3 If the pH is greater than 2, generate an NCM in accordance with SOP TL-CA-085: *Nonconformance and Corrective Action Procedures*, to get approval to adjust the pH. If the Project Manager approves the pH adjustment, move the sample under a hood. Add 1:1 nitric acid to the sample in 0.50mL aliquots, checking the sample pH after each addition, until the pH <2. The volume of 1:1 nitric acid added to the sample should not exceed 1% of the total volume of the sample. For a 500-mL sample, the maximum volume of 1:1 nitric is 5mL. If more acid is required, contact the supervisor for further guidance. Record all pH adjustments in the "Notes" section of the LIMS prep batch Worksheet tab.

NOTE: Samples that are not at pH <2 upon arrival in the lab may contain cyanide or sulfide or may be highly buffered. Working under a hood minimizes the hazard that may be caused by the evolution of hydrogen cyanide or hydrogen sulfide upon acidification of the sample. Be aware that acid/base neutralization reaction may be violent and evolve a good deal of heat.

## 6.0 MATERIALS AND APPARATUS

6.1 Thermo Jarrell Ash TJA ICAP61E-trace inductively coupled plasma emission spectrometer with data system, or equivalent

6.2 Argon gas supply and appropriate fittings

6.3 Cooling water supply

6.4 Peristaltic pump

6.5 Class A volumetric glassware for making standards

6.6 Pipettes- Pipettes must be calibrated in accordance with SOP TL-AN-030: *Pipet and Volumetric Container Calibration Check*.

6.7 Disposable Transfer Pipets

6.8 Test tubes to fit auto sampler TJA AS300, or other equivalent autosampler

6.9 Tygon tubing for inducing sample into ICP

## 7.0 REAGENTS

All reagents must be tracked in accordance with SOP TL-AN-041: *Standard Materials and Reagent Traceability*.

7.1 Nitric acid (HNO<sub>3</sub>)-reagent grade.

7.2 Hydrochloric acid (HCl)-reagent grade.

7.3 Deionized Water-lab generated deionized water. ASTM Type I or Type II. The conductivity must be checked daily in accordance with SOP TL-AN-035: *Conductivity Checks for Laboratory Deionized Water*.

## 8.0 STANDARDS

All standards must be tracked in accordance with SOP TL-AN-041: *Standard Materials and Reagent Traceability*. All standards must be prepped in accordance with SOP TL-AN-043: *Standard Preparation*.

8.1 Calibration and spike solutions are prepared from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions.

8.2 Concentrations for the calibration standards are given in Table 1. Appendix B contains examples for the preparation of the initial calibration and calibration verification standards for both 6010 and 200.7. If the laboratory uses "recipes" other than those listed in Appendix A, the recipe must be documented in the standard material traceability logbook. All standards must have been prepared in 5% hydrochloric acid and 1% 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.

8.3 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<sub>s</sub> = volume of stock standard (mL)

C<sub>s</sub> = concentration of stock standard (mg/L)

V<sub>lc</sub> = volume of linearity check standard to prepare (mL)

C<sub>lc</sub> = 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 5% by volume and the nitric acid concentration at 1% by volume.

## 9.0 SAMPLE PREPARATION

- 9.1 Liquid samples must be digested in accordance with SOP TL-ME-050: *Digestion Procedures for ICP: Total Metals and Total Recoverable Metals in Liquid Samples (Methods 3005A, 3010A, and 200.)*. Solid samples must be digested in accordance with SOP TL-ME-051: *Digestion Procedures for ICP: Total Metals in Soils, Sediments, Wastes, Biological Tissues, and Oils (Method 3050B)*.

## 10.0 ANALYTICAL PROCEDURES

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

### 10.1 Initial Calibration/Standardization

NOTE: The following instructions are for the TJA ICAP61E instrument. For other instruments, follow manufacturer's procedure.

- 10.1.1 Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about a half an hour for the instrument to warm up. If optics were turned off, allow 2 hours warm up time.
- 10.1.2 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.
- 10.1.3 Analyze the calibration standards and calibrate the ICP.
- 10.1.4 The highest concentration calibration standard is reanalyzed after the instrument is standardized as an "unknown". The results for the re-analysis of the highest concentration calibration standard must be within  $\pm 5\%$  of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.
- 10.1.5 The QC Check standards (ICV) and the Calibration Blank (ICB) are analyzed as a check on the instrument calibration.
- 10.1.5.1(EPA Method 6010) The results for the target compounds in the initial calibration verification (ICV) must be within the  $\pm 10\%$  of the true value.
- 10.1.5.2(EPA Method 200.7) The results for the target compounds in the initial calibration verification (ICV) must be within the  $\pm 5.0\%$  of the true value. **When performing Method 200.7, note that this solution should be prepared fresh weekly.**
- 10.1.5.3(EPA 6010/200.7) The results for the target compounds in the initial calibration blank (ICB) must be evaluated to be less than the upper 3 times standard deviation, but greater than the lower 3 times standard deviation of the calibration blank.
- 10.1.6 The RL Trace Check Solution is analyzed to demonstrate that the ICP is capable of detecting the target compounds at or near the reporting limit (RL). The determined concentration must within  $\pm 50\%$  of the true concentration.
- 10.1.7 The ICP Interference Check Sample A (ICSA) is analyzed. The concentrations of the target analytes in the ICSA must fall within the established control limits of 1.5 times the standard deviation of the mean value. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

10.1.8 The ICP Interference Check Sample AB (ICSAB) is analyzed. The concentrations of the target analytes in the ICSAB must fall within the established control limits of 80-120 %.

## 10.2 Continuing Calibration Verification (CCV)

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

10.2.1.1 (EPA Method 200.7-NPDES) The results for the target compounds in the continuing calibration verification (CCV) must be within  $\pm 10.0$  % of the true value.

10.2.1.2 (EPA 6010/200.7) The results for the target compounds in the continuing calibration blank (CCB) must be evaluated to be less than the upper 3 times standard deviation, but greater than the lower 3 times standard deviation of the calibration blank.

10.2.2 ICP Interference Check Solution and the RL check solution are analyzed at the beginning and end of each analytical sequence.

## 10.3 Sample Analysis

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

10.3.2 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)
REANALYSIS OF HIGH CONCENTRATION CALIBRATION STANDARD AS A SAMPLE
RL TRACE CHECK SOLUTION
ICP INTERFERENCE CHECK SOLUTION A (ICSA)
ICP INTERFERENCE CHECK SOLUTION AB (ICSAB)
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
CONTINUING CALIBRATION VERIFICATION (CCV)
CONTINUING CALIBRATION BLANK (CCB)

The analytical sequence must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.3 Determine the concentration of the samples and QC items using the procedures of Section 11.



- 10.3.4 If the concentration of a sample is above the linear range of the ICP, the sample digestate must be diluted and reanalyzed. Calculate the amount of sample digestate needed to prepare the desired dilution per 11.2.3

NOTE: Dilutions must be prepared in deionized water containing 5% hydrochloric acid and 1% nitric acid by volume. Some samples may require multiple dilutions.

- 10.3.5 The dilution factor is calculated per 11.2.4.

#### 10.4 Dilution QC Check

A dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

- 10.4.1 Select a sample digestate that contains one or more target analytes at a concentrations greater than 10X the reporting limit.
- 10.4.2 Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.
- 10.4.3 Compare the results of the diluted and un-diluted aliquots of sample digestate.
- 10.4.4 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 10.5).

If the concentration of the analyte in the sample is not at least 50 times the instrument detection limit, evaluate the post-digestion spike.

#### 10.5 Post-digestion Spike QC Check

A post-digestion spike is 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 10.4, above.

- 10.5.1 Transfer 10mL of a digestate to a suitable vial.
- 10.5.2 Spike the sample with 0.10mL of ICP Spike Sample Standard I and 0.10mL of ICP Spike Standard II. The theoretical concentration of the post digestion spike is the same as the LCS/LCSD/MS/MSD if the volume of spiking solution is discounted.
- 10.5.3 Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed).
- 10.5.4 Calculate the percent recovery of the post digestion spike per 11.2.3.
- 10.5.5 Evaluate the recovery using the following decision matrix. Limits for 6010B post digestion spikes are 75-125% recovery. Limits for 200.7 post spikes are 85-115 % recovery.

Result of Post Digestion Spikes	Action
Within 75-125% limits for 6010B Within 85-115% limits for 200.7	None
>125% recovery for 6010B >115% recovery for 200.7	Repeat analysis. Remake spiking solutions, re-spike, and reanalyze. Reanalyze un-spiked sample
<75% recovery but >50% recovery for 6010B <85% recovery but >50% recovery for 200.7	1) Dilute and re-spike. Elevate RL accordingly (for all associated samples). 2) Spike and evaluate all associated samples. 3) Qualify all associated samples
<50% recovery for 6010B and 200.7	Dilute digestate and repeat spike. <b><i>Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples)</i></b> 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.

- 10.5.6 The post digestion spike 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.

## 11.0 DATA ANALYSIS AND CALCULATIONS

### 11.1 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.

- 11.1.1 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

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

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

where

RL<sub>qap</sub> = reporting limit from STL LQM (ug/L)

F = final volume of the sample digestate (L)

V = volume of sample digested (L)

DF = dilution factor

The LQM Reporting Limits assumes:

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

## 11.2 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.

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

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

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.)

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

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

where

RL<sub>qap</sub> = 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)

The LQM Reporting Limits assumes:

F = 0.100L (100mL)

DF = 1

W = 0.0010kg (1.0g)

solids = 1.0

11.2.3 The percent recovery of the post digestion spike is calculated as follows:

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

where

C<sub>ps</sub> = concentration of post digestion spike (ug/L)

C<sub>s</sub> = concentration of un-spiked sample (ug/L)

C<sub>2</sub> = theoretical concentration of spike (ug/L)  
(See 10.2.5.2)

11.2.3 The amount of sample digestate needed to prepare the desired dilution is determined as follows:

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

where

V<sub>f<sub>vol</sub></sub> = final volume of diluted sample (mL)

V<sub>digest</sub> = volume of sample digestate used to make the dilution (mL)

11.2.4 The dilution factor is calculated as follows:

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

where

V<sub>f<sub>vol</sub></sub> = final volume of diluted sample extract (mL)

V<sub>digest</sub> = 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.

## 12.0 QUALITY CONTROL AND QUALITY ASSURANCE

- 12.1 SOP TL-AN-002: *Analytical Batching and Evaluation of QC Data* and the SOP Summary provide guidance on evaluating QC and sample data. This guidance, including corrective actions, is summarized in Appendix A. SOP TL-AN-002 contains the equations for the evaluation of the QC samples for accuracy and precision as well as corrective actions.
- 12.2 A batch blank must be processed for each batch of 20 or fewer samples that are subjected to Mercury digestion. If a batch consists of samples for SPLP, or are required to be lab filtered, an extraction fluid blank for each extraction or filtered blank must be taken through the entire digestion and analytical procedure.
- 12.3 Initial and on-going demonstration of capability must be performed by the analyst in accordance with SOP TL-CA-092: *Evaluation of DOCs*. IDOC and CDOC must be  $\pm 5\%$  of the stated value for 200.7
- 12.4 The method detection limit (MDL) must be determined initially, whenever changes occur that may affect instrument sensitivity, or every 3 years, in accordance with SOP TL-CA-090: *Determination of the Method Detection Limit (MDL)*.

- 12.5 The MDL must be verified annually by analysis of a verification standard at 1-2 times the calculated MDL. The acceptance criteria are that each element be detected without any manipulation of instrument sensitivity. If an element cannot be detected, a new MDL study must be performed.
- 12.6 The instrument detection limit must be determined annually in accordance with SOP TL-CA-091: *Determination of the Instrument Detection Limit (IDL)*.
- 12.7 Data that does not meet the acceptance criteria may be conditionally reported with the use of data flags on the final report or through the use of a case narrative attached to the final report.
  - 12.7.1 The linear range of the ICP must be determined at least every 6 months. 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. 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.
    - 12.7.1.1 Profile and calibrate the ICP as described in Section 10.1.
    - 12.7.1.2 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.
    - 12.7.1.3 Analyze the standards following the analytical sequence described in Section 10.3. Verify the calibration after every 10 analyses.
    - 12.7.1.4 Compare the concentration of the linear range standard with its true concentration.

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

where

C<sub>cal</sub> = concentration determined from analysis

C<sub>true</sub> = 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.

- 12.6 Interelement correction factors (IEC) for all elements must be determined every 6 months. Use the manufacturer's guidance for determination of the IECs. The IECs must be verified at the beginning of each analytical sequence.
- 12.8 Work Instructions which may be posted for analyst reference are referenced in Appendix C.

## 13.0 PREVENTATIVE MAINTENANCE AND TROUBLESHOOTING

ICP PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Sample and Internal Standard Pump Tubing	X							Rotate every other day; replace as needed.
Miscellaneous Pump Tubing					X			Replace.
Nebulizer							X	Clean.
Filters			X					Inspect monthly, clean or replace as needed.
Spray Chamber							X	Clean.
Quartz Torch			X					Clean.
Argon wetting system		X						Fill with water.

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

## 14.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Excess samples, digests, reagents, and standards must be disposed in accordance with SOP TL-CA-070: *Waste Management*.

### 14.1 Waste Streams Produced by the Method

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

WASTE STREAMS PRODUCED BY THE METHOD	
Waste Stream Description	Category (Preferred Treatment)
Acid Digests	RCRA Hazardous Waste (Acid digests from RCRA Hazardous samples are labeled as hazardous waste and stored in a satellite accumulation vessel until the lab accumulation of hazardous materials is picked up by an outside vendor.)
Acid Digests and Rinses	Non-RCRA Hazardous Waste (Acid digests from Non-RCRA Hazardous samples and acid rinses are accumulated in a satellite vessel, neutralized with sodium bicarbonate, and disposed of in the sewer system (down the drain with water running).)
Water Samples Preserved with Acids or Bases	RCRA Hazardous Waste (Excess preserved water samples designated as RCRA Hazardous Waste are labeled as hazardous waste and stored until the lab accumulation of hazardous materials is picked up by an outside vendor.)
Water Samples Preserved with Acids or Bases	Non-RCRA Hazardous Waste (Excess preserved water samples designated as Non-RCRA Hazardous Waste are accumulated in a satellite vessel, neutralized with sodium bicarbonate, and disposed of in the sewer system (down the drain with water running).)
Water Samples not Preserved with Acids or Bases	Non-RCRA Hazardous Waste (Excess unpreserved water samples are discarded to the sewer system (down the drain with water running).)
Unused Standards	RCRA Hazardous Waste (Unused standards are labeled and stored until the lab accumulation of hazardous materials is picked up by an outside vendor.)

## 15.0 REFERENCES

- 15.1 TestAmerica Corporate Environmental Health and Safety Manual (CSM), current revision
- 15.2 TL-QAM, *TestAmerica Tallahassee Quality Assurance Manual* (QAM), current revision
- 15.3 *Test Methods for Evaluating Solid Waste*; Third Edition, SW-846; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC, November 1992 (including Update III), Method 6010B.
- 15.4 Method 200.7 (NPDES): *Methods for Chemical Analysis of Water and Wastes*; USEPA Office of Research and Development, Cincinnati, Ohio, March 1983; 40 CFR Part 136.

## 16.0 TABLES, DIAGRAMS, and FLOWCHARTS

TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Trace 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	100	6.0/5.0	0.20	1000	2.0	200
Antimony (Sb)	206.838	5.0	2.0/2.0	0.020	100	0.50	50
Arsenic (As)	189.042 193.696	5.0	2.0/2.0	0.0080	30	2.0	200
Barium (Ba)	493.409	5.0	1.0/1.0	0.010	50	2.0	200
Beryllium (Be)	313.042	5.0	2.0/2.0	0.0040	15	0.050	5.0
Cadmium (Cd)	226.502 228.802	5.0	2.0/2.0	0.0050	40	0.050	5.0
Calcium (Ca)	317.933 315.887	25	2.0/2.0	0.50	400	5.0	500
Chromium (Cr)	267.716	5.0	2.0/2.0	0.010	75	0.20	20
Cobalt (Co)	228.616	5.0	2.0/2.0	0.010	75	0.50	50
Copper (Cu)	324.754	5.0	2.0/2.0	0.010	40	0.25	25
Iron (Fe)	259.940 271.441	10	2.0/2.0	0.050	200	1.0	100
Lead (Pb)	220.353	5.0	2.0/2.0	0.0050	100	0.50	50
Magnesium (Mg)	279.079	100	2.0/2.0	0.50	500	5.0	500



TABLE 1

Element	Wavelength (nm)	Calibration Conc. (mg/L)	ICV/CCV Conc. (mg/L)	RL Trace 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	2.0/2.0	0.010	10	0.50	50
Molybdenum (Mo)	202.030	5.0	2.0/2.0	0.010	20	0.50	50
Nickel (Ni)	231.604	5.0	2.0/2.0	0.020	10	0.50	50
Potassium (K)	766.491	25	10/10	1.0	40	5.0	500
Selenium (Se)	196.026	5.0	2.0/2.0	0.010	50	2.0	200
Silver (Ag)	328.068	5.0	2.0/2.0	0.010	1.5	0.050	5.0
Sodium (Na)	588.995 330.231	50	25/31	0.50	100	5.0	500
Thallium (Tl)	189.042 190.801 377.572	5.0	2.0/2.0	0.010	20	2.0	200
Tin (Sn)	189.989	5.0	2.0/2.0	0.050	10	1.0	100
Vanadium (V)	292.402	5.0	2.0/2.0	0.010	20	0.50	50
Zinc (Zn)	213.856 206.200+	5.0	2.0/2.0	0.020	10	0.50	50

\*For guidance only-instrument sensitivity will vary.

## APPENDIX A SOP SUMMARY

### METHOD SUMMARY - ICP ANALYSIS

#### ANALYTICAL SEQUENCE

Ignite Plasma	Follow instrument manufacturer's guidelines and allow instrument to stabilize for at least 30 minutes.
Profile Instrument	Follow manufacturer's guidelines.
Initial Calibration	Calibrate with a blank and a high standard.
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. Verify calibration by reanalyzing calibration standard as a sample.
Interference Check Solutions	At the beginning of an analysis run, verify the inter-element and background corrections by analyzing the interferent check solutions (ICSA & ICSAB).
RL Trace check solution	At the beginning 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..
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.

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration	Daily	1 std. and 1 blank	
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	6010B = within $\pm 10\%$ of TV 200.7 = within $\pm 5\%$ of TV	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	6010B = within $\pm 10\%$ of TV 200.7 = within $\pm 10\%$ of TV TMDL = within $\pm 5\%$ of TV	Terminate the analysis, fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	The result for the target compound must be less than the upper 3 times standard deviation, but greater than the lower 3 times standard deviation of the calibration blank.	Evaluate down to the $\pm 3$ times standard deviation, if the result is greater than or equal to RL terminate the analysis, correct the problem and reanalyze the previous 10 samples
Highest Standard	Immediately after every calibration	Recoveries within $\pm 5\%$ of expected values	New initial calibration
Interference check standards (ICSA/ICSAB)	At the beginning of an analysis run	ICSA: Results must fall within the established control limits of 1.5 times the standard deviation of the mean value. ICSAB: Recovery must fall within 80-120 %	Terminate the analysis, correct the problem, and recalibrate.
Lab Control Standard and Lab Control Standard Duplicate (LCS/LCSD)	One set per batch of twenty samples or less	6010B: 80-120% 200.7: 85-115%	Redigest and reanalyze batch
Preparation blank – 6010B	One per batch of twenty samples or less	$ \text{result}  < \text{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	$ \text{result}  < \text{RL}$ or result $< 10\%$ of the analyte level in the sample	Redigest and reanalyze batch
MS/MSD – 6010B MS/MSD – 200.7	One set per batch of twenty samples or less.	6010B: 75-125% 200.7: 70-130%	Flag and report data
Serial Dilution (1/5 Dilution)	One per batch of twenty samples or less	$\pm 10\%$	

QC Item	Frequency	Criteria	Corrective Action
Post Digestion Spike	One per batch of twenty samples or less	6010B-75-125% 200.7-85-115%	
RL Trace Check Solution (CRDL)	At the beginning of an analysis run	6010B = within $\pm$ 50% of TV 200.7 = within $\pm$ 50% of TV TMDL = within $\pm$ 30% of TV	Stop the analysis, fix the problem and reanalyze the affected samples.

## APPENDIX B EXAMPLES OF STANDARD PREPARATION

### GENERAL INSTRUCTIONS

All calibration standards must contain 5% hydrochloric acid and 1% 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	5.0	1.0
200	10	2.0
500	25	5.0
1000	50	10

For example, to prepare 500mL of a standard:

- Add 100mL to 200mL of reagent water to a clean 500mL volumetric flask.
- Add 5.0mL of concentrated nitric acid ( $\text{HNO}_3$ ) and 25mL of hydrochloric acid ( $\text{HCl}$ ) 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.

### SINGLE POINT CALIBRATION STANDARDS FOR 6010

#### Calibration STANDARD 1-Calibration Blank (ICB, CCB)

Add 500mL to 600mL of reagent water to a clean 1-L volumetric flask. Add 10mL of concentrated nitric acid ( $\text{HNO}_3$ ) and 50mL of hydrochloric acid ( $\text{HCl}$ ) to the volumetric flask. Dilute to a final volume of 1.0-L with reagent water. Store the standard at room temperature. Other volumes may be prepared at the discretion of the lab. The nitric acid concentration must be 1% by volume and the hydrochloric acid concentration must be 5% by volume.

#### Calibration STANDARD 4

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (ml)	Conc. of Cal Std (mg/L)
Silver(Ag)	1000	1.5	1000	1.5
Arsenic(As)	1000	2.5	1000	2.5
Lead(Pb)	1000	5.0	1000	5.0
Selenium(Se)	1000	5.0	1000	5.0
Thallium(Tl)	1000	5.0	1000	5.0
Cadmium(Cd)	1000	5.0	1000	5.0
Copper(Cu)	1000	5.0	1000	5.0
Nickel(Ni)	1000	5.0	1000	5.0
Tin(Sn)	1000	5.0	1000	5.0
Vanadium(V)	1000	5.0	1000	5.0
Zinc(Zn)	1000	5.0	1000	5.0

## Calibration STANDARD 2

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Beryllium(Be)	1000	5.0	1000	5.0
Barium(Ba)	1000	5.0	1000	5.0
Cobalt(Co)	1000	5.0	1000	5.0
Chromium(Cr)	1000	5.0	1000	5.0
Manganese(Mn)	1000	5.0	1000	5.0
Molybdenum(Mo)	1000	5.0	1000	5.0
Antimony(Sb)	1000	5.0	1000	5.0
Potassium	10000	2.5	1000	25
Sodium	10000	5.0	1000	50

## Calibration STANDARD 3

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of Cal Std (mg/L)
Aluminum(Al)	10000	10	1000	100
Magnesium(Mg)	10000	10	1000	100
Calcium(Ca)	10000	2.5	1000	25
Iron(Fe)	10000	1.0	1000	10

## Continuing Calibration Verification (CCV) Solution

Element/Stock	Conc. of Stock Std, mg/L	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
SPEX QC19	100 (1)	10	500	2.0 (1)
SPEX QC 7	100 (1)	5.0	500	1.0 (1)
Tin(Sn)	1000	1.0	500	2.0
Aluminum (Al)	10000	0.20	500	5.0 (2)
Sodium (Na)	10000	1.5	500	31 (2)

- (1) SPEX QC19 and SPEX QC7 are solutions containing multiple elements. The concentrations are given on the certificate of analysis.
- (2) These concentrations include the contribution from SPEX QC-7 and SPEX QC19.

## Initial Calibration Verification (ICV) Solution

Element/Stock	Conc. of Stock Std, mg/L	mL of Stock Std	Final Volume (mL)	Conc. of CCV Std (mg/L)
SPEX QC19	100 (1)	2.0	100	2.0 (1)
SPEX QC 7	100 (1)	1.0	100	1.0 (1)
Tin(Sn)	1000	0.20	100	2.0
Aluminum (Al)	10000	0.050	100	6.0 (2)
Sodium (Na)	10000	0.25	100	25 (2)

- (1) SPEX QC19 and SPEX QC7 are solutions containing multiple elements. The concentrations are given on the certificate of analysis.
- (2) These concentrations include the contribution from SPEX QC-7 and SPEX QC19.

## Reporting Limit (RL) Trace Check Standard

### Preparation of RL/PQL Stock -ICP

Element	Conc. of Stock Std	mL of Stock Std	Final Volume (mL)	Conc. of RL/PQL Stock -ICP (mg/L)
Silver (Ag)	1000	0.050	100	0.50
Arsenic (As)	1000	0.080	100	0.80
Cadmium (Cd)	1000	0.050	100	0.50
Copper (Cu)	1000	0.10	100	1.0
Nickel (Ni)	1000	0.20	100	2.0
Lead (Pb)	1000	0.050	100	0.50
Selenium (Se)	1000	0.10	100	1.0
Thallium (Tl)	1000	0.10	100	1.0
Aluminum (Al)	10000	0.20	100	20
Barium (Ba)	1000	0.10	100	1.0
Beryllium (Be)	1000	0.040	100	0.40
Calcium (Ca)	10000	0.50	100	50
Cobalt (Co)	1000	0.10	100	1.0
Chromium (Cr)	1000	0.10	100	1.0
Iron (Fe)	10000	0.050	100	5.0
Magnesium (Mg)	10000	0.50	100	50
Manganese (Mn)	1000	0.10	100	1.0
Molybdenum (Mo)	1000	0.10	100	1.0
Potassium	10000	1.0	100	100
Sodium (Na)	10000	0.50	100	50
Antimony (Sb)	1000	0.20	100	2.0
Tin (Sn)	1000	0.50	100	5.0
Vanadium (V)	1000	0.10	100	1.0
Zinc (Zn)	1000	0.20	100	2.0

### Preparation of the RL Trace Check Solution-ICP

RL/PQL Stock	Conc. of RL/PQL Stock -ICP (mg/L)	mL of RL/PQL Stock	Final Volume (mL)	Conc. of RL Trace Check Solution-ICP (mg/L)
Stock -ICP	as above	1.0	100	1/100 of concentrations listed above

## ICP Interference Check Solutions

### Preparation of ICP Interference Check Solution A (ICSA)

Element	Conc. Of Stock(mg/L)	mL of Stock Std	Final Volume(mL)	Conc. (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25	500	500
Magnesium (Mg)	10000	25	500	500
Iron (Fe)	10000	10	500	200

### Preparation of ICP Interference Check Solution AB (ICSAB)

Element	Conc. of Stock(mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std (mg/L)
Aluminum (Al)	10000	25	500	500
Calcium (Ca)	10000	25	500	500
Magnesium (Mg)	10000	25	500	500
Iron (Fe)	10000	10	500	200
Silver (Ag)	1000	0.10	500	0.20
Arsenic (As)	1000	0.050	500	0.10
Barium (Ba)	1000	0.25	500	0.50
Beryllium (Be)	1000	0.25	500	0.50
Cadmium (Cd)	1000	0.50	500	1.0
Cobalt (Co)	1000	0.25	500	0.50
Chromium (Cr)	1000	0.25	500	0.50
Copper (Cu)	1000	0.25	500	0.50
Manganese (Mn)	1000	0.25	500	0.50
Nickel (Ni)	1000	0.50	500	1.0
Lead (Pb)	1000	0.025	500	0.050
Antimony (Sb)	1000	0.30	500	0.60
Selenium (Se)	1000	0.025	500	0.050
Thallium (Tl)	1000	0.050	500	0.10
Vanadium (V)	1000	0.25	500	0.50
Zinc (Zn)	1000	0.50	500	1.0
Molybdenum (Mo)	1000	0.50	500	1.0
Tin (Sn)	1000	0.50	500	1.0



## ICP Matrix Spiking Solutions

ICP Matrix Spiking Solution 1 is a solution purchased from SPEX. The certificate of analysis will list the concentrations of the analytes. Store this solution at room temperature. Prepare this solution every six months or sooner if needed or required.

### Preparation of the ICP Matrix Spiking Solution 2

Element	Conc. of Stock (mg/L)	mL of Stock	Final Volume (mL)	Conc. of Std. (mg/L)
Calcium (Ca)	10000	5.0	100	500
Magnesium (Mg)	10000	5.0	100	500
Molybdenum (Mo)	1000	5.0	100	50
Potassium (K)	10000	5.0	100	500
Sodium (Na)	10000	5.0	100	500
Tin (Sn)	1000	10	100	100

## APPENDIX C WORK INSTRUCTIONS

Work Instructions may be posted for easy reference by the analyst. The following WIs are included by reference and are found in the QA forms folder on Talsvr05:

WGE002: 04.24.08:2 NOTE: For all BRA Samples

WME001: 01.18.10:3 CONTROL LIMITS FOR ICP

WME002: 04.30.09:3 CALIBRATION STANDARDS

WME003: 01.18.10:2 LINEAR RANGES, RLs, MDLs, AND SPIKING SOLUTIONS FOR ICP

Work Instruction may be edited and assigned a new date and revision number without revising the SOP. Current WI revision numbers must be included in each biannual SOP revision.

## 17.0 REVISION HISTORY

Revision 10: 02/11/2008

- Changed all STL references to TestAmerica
- Incorporated new logo, cover page, and naming convention
- Added Section 17.0, REVISION HISTORY
- Revised SOP references to reflect new naming convention
- Added references to documents TL-QA-001 and TL-QA-002, removed references to LQM, replaced with references to TL-QAM, *TestAmerica Tallahassee Quality Assurance Manual*
- 2.2.2, 5.1.2, 12.2: deleted reference to TCLP, renumbered remainder of section
- 12.5: added annual MDL verification standard requirement, renumbered remainder of section
- 12.8: edited text: work instructions no longer included in SOP, only referenced
- Appendix C: deleted actual work instructions, added location

Revision 11: 06/13/2008

- 2.2.10: Deleted reference to TCLP, changed "TCLP leachate" to "sample"
- 5.1: Added information regarding receipt of unpreserved samples, lab preservation, and 24-hour waiting period
- 5.1.2: Corrected SOP name
- 5.3.1: Changed "upon arrival at the lab" to "prior to digestion and documented in the LIMS prep batch"
- 5.3.2: Changed "in the sample logbook" to "in the Notes section of the LIMS prep batch Worksheet tab"
- 6.1, 6.8: Added "or equivalent"
- 10.1: Added NOTE regarding other instruments
- Appendix A QC Checks table: Revised CCV and CRDL limits to match Work Instruction WME001:04.24.08:2
- Appendix B Standard Preparation: Revised table entries for Arsenic (Std. 4) and Iron (Std. 3) to match Work Instruction WME002:04.24.08:2
- Appendix C Work Instructions: Updated revision numbers for WGE002, WME001, WME002, and WME003

Revision 12: 10/08/2009

- 5.1.1: Added requirements for procedure and documentation of preservation for unpreserved or inadequately preserved samples
- 5.1.2: Added requirement for SPLP leachates to be matrix spiked prior to preservation
- 5.1.3: Added documentation requirements for lab-filtered and preserved samples for dissolved metals
- 5.2.1: Changed storage requirements for soil samples from 4 °C to room temperature
- 12.7.1: Changed frequency requirement for linear range study to every 6 months
- 12.6: Changed frequency requirement for IECs to every 6 months

Revision 13: 01/20/2010

- 10.3.2, 12.6, Appendix A Sequence and QC Table: Deleted ending analysis of RL Trace, ICSA, and IC SAB (only required at beginning of run)
- Appendix C: Updated work instruction references, added WME004