Title: ACID DIGESTION OF SOILS [SW-846 3050B FOR ICP, ICP/MS]

Approvals (Signature/Date):

10/19/12	10/19/12
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This SOP was previously identified as SOP No. ST-IP-0002 Rev. 15

Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the preparation of soil samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma Atomic Emission/Mass Spectrometry (ICP/MS).

1.2 This procedure is based on SW-846 Method 3050B.

1.3 Additional metals may be processed by this method, assuming that performance criteria of the determinative method are met.

1.4 This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludge, waste and sediment.

1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory's Information Management System.

2.0 SUMMARY OF METHOD

2.1 A representative 0.5 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP, ICP/MS analysis. The digestates are then diluted to 50ml/50g.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Total Metals: The concentration determined on an unfiltered sample following digestion.

4.0 INTERFERENCES

4.1 Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc). Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.3 Specific analytical interferences are discussed in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual 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.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Hydrogen peroxide (H2O2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H2O2 to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn.

5.3 PRIMARY MATERIALS USED

5.3.1 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**	**Exposure Limit (2)**	**Signs and Symptoms of exposure**
Hydrochloric Acid	Corrosive Poison	5ppm (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.
Hydrofluoric Acid	Poison Corrosive Dehydrator	3 ppm-TWA	Severely corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
Nitric Acid	Corrosive Oxidizer Poison	2ppm (TWA) 4ppm (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.
Hydrogen Peroxide	Oxidizer Corrosive	1ppm (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 preven violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit			
TWA – Time Weighted Average			

STEL – Short Term Exposure Limit
Ceiling – At no time should this exposure limit be exceeded.

6.0 EQUIPMENT AND SUPPLIES

6.1 Hot block, capable of maintaining a temperature of 90°C - 95°C.

6.2 Thermometer, temperature range of 0-250°C.

6.3 Laboratory fume hood.

6.4 Hot block digestion vessels

6.5 Watch glasses, ribbed or equivalent

6.6 Vacuum filters – 0.45 µm

6.7 Analytical balance capable weighing to the nearest 0.001g.

6.8 Pipettes or reagent dispensers, micro pipettes –0.05 -10mL

6.9 3mm glass silica beads

6.10 Centrifuge tubes

6.11 Vacuum box for Eichrom columns

6.12 250 ml beakers

6.12.1 Glassware used for TC-99 must be cleaned prior to use following procedures outlined in SOP ST-RC-5006 “Decontamination of Lab Glassware"

6.13 Teflon tape

6.14 disposable plastic funnels for Eichrom columns

6.15 vacuum pump

6.16 hot plate capable of 250° C

6.17 centrifuge

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI water: Obtained by the use of a Milli-Q System

7.3 Rhenium tracer standard

7.4 Matrix Spike Standard, NIST traceable

7.5 ERA soil laboratory control samples (LCS) or LCS aqueous standard, NIST traceable

7.6 Nitric acid (HNO_3), concentrated, trace metal grade

7.7 Hydrochloric acid (HCl), concentrated, trace metal grade

7.8 30% Hydrogen peroxide (H_2O_2), Ultrex Grade.

7.9 Nitric Acid (HNO_3), concentrated, 16 N.

7.9.1 Nitric Acid (2 N HNO_3)

7.9.1.1. Add 125 ml of concentrated HNO_3 (16 N) to 800 ml of DI water. Cool and dilute to one liter with DI water.

7.9.2 Nitric Acid (1 N HNO_3)

7.9.2.1. Add 62.5 ml of concentrated HNO_3 (16 N) to 900 ml of DI water. Cool and dilute to one liter with deionized water.

7.9.3 Nitric Acid (0.1 N HNO_3)

7.9.3.1. Add 62.5 ml of concentrated HNO3 (16 N) to 900 ml DI water. Cool and dilute to one liter with DI water.

7.9.4 Nitric Acid (0.01 N HNO_3)

7.9.4.1. Add 0.65 ml of concentrated HNO_3 (16 N) to 900 ml of DI water. Dilute to one liter with DI water. Option: dilute 100 ml of 0.1 N HNO_3, to one liter with DI water.

7.10 Nitric Acid/Hydrofluoric Acid (0.01 N HNO_3,, in 3 N HF)

7.10.1 Add 0.65 ml of concentrated HNO_3 (16N) to 800 ml of DI water, then add 103 ml of concentrated HF (29 N). Cool and dilute to one liter with DI water. Option: dilute 100 ml of 0.1 N HNO_3 to 750 ml with DI water, then add 103 ml of concentrated HF (29 N). Cool and dilute to one liter with DI water.

7.11 Pre-packaged, 2 ml, Eichrom TEVA Column, 100-150 micron particle size

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection

procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples are to be collected in plastic or glass containers.

8.3 All soils must be refrigerated to $4^{o}C \pm 2^{o}C$.

8.4 The analytical holding time for metals is 6 months.

9.0 QUALITY CONTROL

9.1 Batch

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and a Matrix Spike/ Matrix Spike Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1. Matrix Spike (MS) and Sample Duplicate (SD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 Method Blank

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Soil analyses, the method blank is comprised glass beads.

9.3 Laboratory Control Sample

9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Soil analyses, the LCS is a purchased Standard Reference Material

9.3.3.1. If requested, the laboratory may perform an LCS consisting of glass beads fortified with target analytes

9.3.3.2. Boron and Silicon are not performed using glass beads due to contamination issues. The LCS standard for these analytes is preserved with hydrofluoric acid which breaks down the glass beads causing contamination.

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 Sample Duplicate

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.6 Procedural Variations/ Nonconformance and Corrective Action

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Hot block/Hot plate must be checked daily when in use.

10.1.1 Temperature is documented on digestion log.

10.1.2 A calibrated thermometer is suspended in sand in a digestion vessel of water and brought to the proper temperature.

10.1.2.1. See SOP ST-QA-0005 for information regarding calibration of thermometers.

10.2 Instrument calibration is discussed in in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

11.0 PROCEDURE

NOTE: For DOE Antimony soil prep, see section 11.23.

11.1 Label digestion vessel with sample ID or QC identifier.

11.2 Homogenize the sample by mixing thoroughly. See SOP ST-QA-0038 for details.

11.3 Weigh a 0.5 g +/- 0.004g portion of sample and the batch QC and record the weight on the digestion log.

11.3.1 Larger sample sizes (typically 2 g) may be used if needed to meet the reporting limits.

11.3.2 Measure 0.5 g of glass beads into a digestion vessel for the method blank.

11.3.3 Spike MS/MSD with spiking mix applicable to the requested analysis.

11.3.3.1. Document spiking volumes and standard numbers on the bench sheet.

11.3.3.2. Reagent volumes are adjusted to reflect the sample volume used.

11.4 Add 2.5mL 1:1 HNO_3, mix each vessel.

11.5 Place digestion vessels in hot block at 90°C - 95°C and heat for 10 minutes.

11.5.1 **Do not allow the sample to boil or go dry during the digestion.** Allowing so may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric media.

11.6 Take samples out and cool.

11.7 Add 1.25ml HNO_3.

11.8 Return vessels to hot block.

11.9 Place watch glasses on digestion vessels

11.10 Reflux at 90°C - 95°C for 30 minutes.

11.11 Add DI water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.11.1 If brown fumes are observed, additional 1.25 mL aliquots of concentrated nitric acid until no more fumes are evolved.

11.12 Remove vessels from block and allow the samples to cool.

11.13 Add 1 mL of DI water and 2 mL of 30 % H_2O_2.

11.13.1 Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. If this occurs, add a minimal amount of DI water until sample settles, to ensure no loss of sample. If any amount of sample is lost, a redigestion must be done.

11.14 Return covered vessel to hot block and heat sample until effervescence subsides.

11.15 While in block, continue adding 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

11.4.17.1 NOTE: Do not add more than a total of 5 mL of 30 % H_2O_2.

11.16 Continue heating for 1 hour.

11.17 Remove vessel from hot block and allow to cool.

11.18 Add 2.5 mL HCL and reflux, on hot block, for an additional 15 minutes without boiling.

11.19 Remove vessel from hot block and allow to cool.

11.20 Cool the samples, Wash down the vessel wall with DI water to dissolve any precipitation to a final volume 50 ml.

11.21 Particles in the digestate should then be removed by allowing the sample to settle.

11.21.1 Alternatively if the digestate needs to be run immediately or if the digestate, due to its physical appearance, is determined to need more than settling to remove particles, the sample is filtered using a vacuum pump filter. The sample is filtered then the digestate is filtered, washing down the walls of the vessel and rinsing the entire sample out of the vessel, and then digestate is brought up to 50mL.

11.22 Sample preparation is complete. Store digestates in designated cabinet, and the digestion log is given to the analyst.

11.23 Hot Acid Prep for Antimony

11.23.1 Weigh out 0.5 g of sample and QC.

11.23.1.1. Weigh out 0.5g glass beads for blank and LCS.

11.23.2 Spike the MS/MSD, if requested, with the appropriate spiking mix.

11.23.3 Add 2.5 ml of HNO_3 and 2.5 ml of HCL.

11.23.4 Place digestion vessels in the hot block at 90-95°C and heat for 15 minutes.

11.23.5 Filter through vacuum filter while samples are still hot.

11.23.6 Rinse filter with 1.2 ml of hot HCL.

11.23.7 Rinse filter with 5 ml of hot DI water 4 times for a total of 20 ml.

11.23.8 Rinse the sample debris off the filter paper and place back into original digestion vessel.

11.23.9 Add 2.5 ml of HCL.

11.23.10 Return vessels to hot block.

11.23.11 Place watch glasses on the digestion vessels.

11.23.12 Reflux at 90-95°C for 20 minutes.

11.23.13 Filter while still hot through vacuum filter, adding original filtrate.

11.23.14 Rinse 3 times with DI water.

11.23.15 Bring samples to a final volume of 50 ml with DI water.

11.24 Preparing solid samples for Tc99 by ICPMS:

11.24.1 Soils - Extraction for Soil Matrices No Muffling (samples low in organics)

11.24.2 Initiate sample preparation worksheet.

11.24.3 Weigh 5 grams of sample into a 250 ml labeled beaker.

11.24.4 Use 5 grams sand for method blank and LCS.

11.24.5 Record sample weight.

11.24.6 Add 0.5mL TC99 Cal in to LCS, MS and MSD.

11.24.7 Add 0.5mL of 50ppm Re Tracer into all samples plus QC.

11.24.8 Add 45 ml of 1 N nitric acid to each beaker.

11.24.9 Heat on hot plate for 30 minutes at 250 degrees.

11.24.10 Remove samples from hot plate and allow cooling for 2-5 minutes.

11.24.11 Transfer the soil residue to a 50 ml centrifuge tube, with DI water. Cap centrifuge tube.

11.24.12 Centrifuge the soil solution at 2000 rpm, for 5 minutes.

11.24.13 Decant the soil leachate solution into beaker.

11.24.14 If using original beaker, ensure beaker is clean of any solid remains.

11.24.15 Add approximately 5 ml of 30 % Hydrogen peroxide, stir and heat for 30 minutes at 250 degrees.

11.24.16 The solution should lighten from dark brown to light yellow or clear. If the solution remains dark, add another 5ml of the Hydrogen peroxide solution and heat another 30 minutes.

11.24.17 Repeat step 11.1.13.1 one additional if solution is still dark.

11.24.18 After 3 additions of peroxide, if solution is still dark, write the NCM noting color and procedure with sample preparation.

11.24.19 Add approximately 100 ml DI water.

11.24.20 Add another 5ml 30% peroxide (H2O2) and heat until the sample ceases to effervesce.

11.24.21 Cool to room temperature.

11.24.21.1. Set up filtering box with labeled 50mL digestion tubes

11.24.22 Set up the vacuum apparatus for the number of samples to be analyzed.

11.24.23 Place Eichrom TEVA column directly in vacuum apparatus. Press the top frit down against the resin. Insert a clean funnel into the top of the Teva column. Use Teflon tape to make a tight seal.

11.24.24 Hook the vacuum apparatus up to a vacuum pump.

11.24.25 Add approximately 5 ml of 0.1 N nitric acid to each TEVA column.

11.24.26 Turn on pump.

11.24.27 When the 0.1 N nitric acid runs through the column, add the sample until all of it has run through the column.

11.24.28 Add 25 ml of 0.01 N nitric acid to each column, allow the rinse to run through the resin.

11.24.29 When the rinse finishes going through the column, add 20 ml of 3 N HF – 0.01 N nitric acid solution, and allow it to run through column.

11.24.30 Immediately after the HF solution runs through the column add 20 ml of 0.01 N nitric acid and allow it to run through the column.

11.24.31 Turn off the vacuum pump and transfer columns/funnels to filtering box over corresponding digestion tubes.

11.24.32 Add 10mL of 8M nitric acid to each sample

11.24.33 Allow all 10mL to gravity filter through the column into the centrifuge tube.

11.24.34 Bring digestion vessel up to a final volume of 50mL with DI

11.24.35 Samples are ready to be analyzed by ICPMS

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis QAM.

12.2 Specific calculations are included in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment, acceptance criteria and corrective actions are included in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

13.2 Data assessment does not pertain to this sample preparation procedure.

13.3 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036.

14.0 METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

14.1 Method performance data, reporting limits, and QC acceptance limits, are given in the associated analytical SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 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. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

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

16.2.2 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 3050B.

17.2 Eichrom Technologies, Inc., Analytical Procedures, Procedure TCS01, "Technetium-99 in Soil", April 26, 2001.

17.3 DOE Methods Compendium RP550 Technitium-99 Analysis using Extraction Chromatography

17.4 TestAmerica St. Louis Quality Assurance Manual (QAM), current revision

17.5 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions

17.6 Associated SOPs, current revisions

17.6.1 ST-PM-0002, Sample Receipt and Chain of Custody

17.6.2 ST-QA-0002, Standard and Reagent Preparation

17.6.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes

17.6.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.6.5 ST-QA-0016, IDL/MDL Determination

17.6.6 ST-QA-0036, Non-conformance Memorandum (NCM) Process

17.6.7 ST-QA-0038, Procedure for Compositing and Subsampling

17.6.8 ST-IP-0004, Labware Preparation for Inorganic and Trace Metal Analysis

17.6.9 ST-RC-5006 Decontamination of Laboratory Glassware
17.6.10 ST-MT-0001, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry
17.6.11 ST-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method for Trace Element Analysis

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCED METHOD

18.1 TestAmerica St. Louis uses a 0.5 sample aliquot size instead of the 1g aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.

18.2 Additionally, sample reagent volumes needed for digestion have been reduced to reflect the reduction in sample volume.

18.3 Acid strength has been reduced to allow for ICP/MS analysis.
18.3.1 TestAmerica St. Louis conducted a study of varying acid strengths to establish a successful medium between digestion efficiency and instrument tolerance.

18.4 The final acid concentration in the digestate is 10% acid.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Glass beads added to equipment (Section 6)
19.2 Balance capability and pipette volumes changed in section 6.
19.3 Additional information added to LCS composition in section 9.3.
19.4 Plunger removed from section 11.21 due to zinc contamination.
19.5 Blank & LCS composition updated in section 11.23.
19.6 Grammatical corrections.
19.7 Rev. 13; updated the Hydrogen peroxide from reagent grade to ultrex grade in section 7.0.
19.8 Rev. 13; copied 9.3.3.2 and added it to the end of section 9.2.
19.9 Rev. 13; Updated the use of glass beads in section 9.2 and cause of contamination issues in section 9.3.3.2.
19.10 Rev. 13: Adjusted reagent volumes used to reflect sample volume used in section 11.3.3.2.
19.11 Rev 14:
19.11.1 Removed filter step from section 2.1
19.11.2 Added HF acid to safety table in Section 5
19.11.3 Added TC-99 soil prep equipment to Section 6
19.11.4 Added TC-99 soil prep reagents and standards to Section 7.
19.11.5 Added soil prep for TC-99, section 11.24
19.11.6 Added Eichrom method references to Section 17.
19.12 Rev 15:
19.12.1 Annual Review, No Changes.
19.13 Rev 16:
19.13.1 Removed Technetium 99 tracer prep procedure from section 7.0 replaced Tc-99m with rhenium as tracer.
19.13.2 Updated solid sample preparation for Technetium 99 by ICPMS in 11.24.
19.13.3 Removed references to Q'tims – old laboratory LIMs system.