
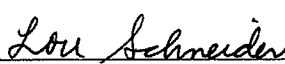
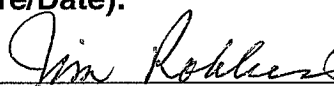



**Title: DIGESTION PROCEDURES FOR ICP: TOTAL METALS
 AND TOTAL RECOVERABLE METALS IN AQUEOUS SAMPLES
 (METHODS 3005A, 3010A, AND 200.7)**

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP contains the digestion procedure used for the preparation aqueous liquids (water), surface and groundwaters, wastewaters, and SPLP leachate samples for metals determination by ICP. This SOP is based on the guidance provided in SW-846 methods 3005A (Total Recoverable Metals) and 3010A (Total Metals) and EPA method 200.7.
- 1.2 The elements to be prepared by this SOP are listed in sections 8.2.1 and 8.2.2.

2.0 SUMMARY OF METHOD AND DEFINITIONS

- 2.1 Total metals: A 5mL aliquot of a well-mixed sample diluted to 50mL with DI water is transferred to a digestion tube. The sample is refluxed with nitric acid at approximately 95°C. After the sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume of 50mL with DI water.
- 2.2 Total recoverable metals: A 50mL aliquot of a well-mixed sample is transferred to a digestion tube. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95°C. After sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume of 50mL with DI water.
- 2.3 Samples filtered through a 0.45µm filter for the determination of dissolved metals do not require digestion if the sample:
- has a low COD(<20mg/L);
 - has a turbidity <1 NTU ;
 - is colorless with no significant odor ; and
 - is of one liquid phase and free of suspended particulates or precipitates after acidification (40 CFR Part 136 Table 1B-note 4)
- 2.4 Definitions
- 2.4.1 **Digestate** - The digested sample
- 2.4.2 **ICP** - Inductively Coupled (argon) Plasma
- 2.4.3 **HCl** -Hydrochloric Acid
- 2.4.4 **HNO3** – Nitric Acid
- 2.4.5 **RL Standard**- Reporting Limit Standard, the lowest calibration standard or the equivalent of the lowest standard. Also referred to as the practical quantitation limit (PQL) standard.
- 2.4.6 **PQL**- Practical Quantitation Limit, same as RL
- 2.4.7 **MDL**- Method Detection Limit, the concentration that can be reported with 99% confidence that the result is greater than zero.
- 2.4.8 **QAM**- Quality Assurance Manual
- 2.4.9 **MSDS**- Material Safety Data Sheet

2.4.10 **DI Water-** Deionized Water

2.4.11 **Dissolved Sample-** A sample that has been filtered through a 0.45µm filter prior to HNO₃ acidification of the sample.

2.4.12 **TALS LIMS-** TestAmerica LIMS System

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 The samples are digested in strong acid solutions and contain acid concentrations of 10-20% by volume. The analyst must wear protective clothing such as a lab coat. 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.

3.1.3 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.

3.1.4 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.

3.1.5 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.

3.1.6 Each analyst should be familiar with the Material Safety Data Sheets (MSDS) for each reagent and standard used in this procedure. These sheets denote the type of hazard that each reagent poses and the safe handling instructions for these compounds.

3.1.7 Care must be taken when handling the digestion tubes. Before handling a digestion tube that has been in use, check the temperature to make sure that it is not hot. Make sure that the digestion tubes are placed on a stable platform during and after the digestion. Vibrations from the hood or an unstable platform can cause the digestion tubes to move and possibly to fall and splatter an analyst with a hot acid solution. Hot acids can cause severe skin burns and destroy unprotected clothing.

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.

Chemical Hazard List

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 The analyst should be cautioned that this digestion procedure may not be sufficiently vigorous enough to destroy some metal complexes. Precipitation will cause a lowering of the silver concentration and therefore an inaccurate analysis.

5.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- 5.1 Liquid samples are collected in 500mL plastic or glass containers. The samples are preserved with 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. The lab preservation time and the digestion start time must be documented to demonstrate the 24-hour waiting period. Samples must be digested and analyzed within 6 months of collection.
- 5.2 SPLP leachates are extracted in accordance with SOP TL-ME-017: *Synthetic Precipitation Leaching Procedure (SPLP): Semivolatile Organic Compounds and Metals*. The SPLP leachate extract samples are preserved in a 250mL plastic container with HNO_3 to a pH <2. 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.
- 5.3 Samples for dissolved metals should be filtered in the field before HNO_3 acid is added to the sample. If the sample is to be filtered in the lab, no HNO_3 acid preservative is added to the sample until the sample is filtered. 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 filtration, preservation, and digestion.

5.4 Sample pH Check

- 5.4.1 The pH of all preserved liquid samples must be checked and documented upon arrival in the lab. If the pH is not within the proper range, additional HNO_3 acid is added to the sample to bring the pH below 2.
- 5.4.2 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 this in the LIMS prep batch and transfer the sample to the storage area.
- 5.4.3 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. Document the pH adjustment in the "Notes" section of the LIMS 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 Digestion Block capable of maintaining a sample temperature of $95^\circ\text{C} \pm 5^\circ\text{C}$. The temperature of the digestion block must be monitored and recorded onto the digestion log with each batch of samples that are digested. The temperature is measured in a digestion tube containing DI water.
- 6.2 Graduated disposable digestion tubes- For total recoverable metals digestion.
- 6.3 Class A volumetric flasks for making standards.
- 6.4 Graduated cylinders-50mL
- 6.5 Pipettes- Pipettes must be calibrated in accordance with SOP TL-AN-030: *Pipet and Volumetric Container Calibration Check*.
- 6.6 Disposable Transfer Pipets
- 6.7 Dedicated Use Thermometer- Thermometers must be calibrated in accordance with SOP TL-AN-055: *Laboratory Thermometer Calibration*.

7.0 REAGENTS

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

- 7.1 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*.

7.2 Nitric Acid (HNO₃)-reagent grade.

7.3 Hydrochloric Acid (HCl)-reagent grade.

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 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$V_i = \frac{C_f \otimes V_f}{C_i}$$

where

V_i = volume of stock standard needed to prepare the spiking solution (mL)

C_i = concentration of stock solution (ug/mL)

C_f = concentration of spiking solution to prepare (ug/mL)

V_f = volume of spiking solution to prepare (mL)

8.2 Preparation of the ICP Spiking Standards 1 and 2

8.2.1 ICP Spike Sample Standard 1 is a premade standard purchased from SPEX. The concentrations of the analytes in this standard are as follows:

Element	Concentration(mg/L)
Alumium (Al)	200
Antimony (Sb)	50
Arsenic (As)	200
Barium (Ba)	200
Beryllium (Be)	5
Cadmium (Cd)	5
Chromium (Cr)	20
Cobalt (Co)	50
Copper (Cu)	25
Iron (Fe)	100
Lead (Pb)	50
Manganese (Mn)	50
Nickel (Ni)	50
Selenium (Se)	200
Silver (Ag)	5
Thallium (Tl)	200
Vanadium (V)	50
Zinc (Zn)	50

8.2.2 Preparation of the ICP Spike Standard 2

Element	<i>Ci(mg/L)</i>	<i>Vi(mL)</i>	<i>Vf(mL)</i>	<i>Cf(mg/L)</i>
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

8.2.2.1 Add 1mL of nitric acid and 5mL of hydrochloric acid to a 100mL volumetric flask containing about 50mL of DI water.

8.2.2.2 Add the appropriate volume of each element stock standard to the flask, dilute to volume with reagent water, and mix thoroughly.

8.2.2.3 Transfer the spiking solution to a labeled storage container.

9.0 SAMPLE PREPARATION

Unless otherwise requested, aqueous liquids, surface and groundwaters, wastewaters, and SPLP leachate will be prepared using the total recoverable metals procedure given in section 9.1.

Samples filtered for the determination of dissolved metals do not require digestion if the sample:

- has a low COD(<20mg/L);
- has a turbidity <1 NTU ;
- is colorless with no significant odor ; and
- is of one liquid phase and free of suspended particulates or precipitates after acidification (40 CFR Part 136 Table 1B-note 4)

9.1 Total Recoverable Metals: This digestion procedure is used for the preparation of aqueous liquids, surface and groundwaters, wastewaters, and SPLP leachate for total recoverable metal determination by ICP.

9.1.1 Transfer 50mL aliquots of DI water to each of three labeled disposable digestion tubes to serve as the method blank (MB), lab control standard (LCS), and lab control standard duplicate (LCSD). If SPLP samples are going to be digested, a SPLP extraction fluid blank, SPLP extraction fluid LCS, and a SPLP extraction fluid LCSD need to be digested in addition to the DI MB, DI LCS, and DI LCSD.

9.1.2 Transfer 50mL well-mixed aliquots of each sample to a disposable digestion tube. Choose one sample per batch and add three additional 50mL well-mixed aliquots of sample to each of three labeled disposable digestion tube to serve as the sample duplicate (DUP), sample matrix spike (MS), and sample matrix spike duplicate (MSD). If insufficient sample is available to perform the DUP, MS, or MSD, an NCM is generated in accordance with SOP TL-CA-085: *Nonconformance and Corrective Action Procedures*.

9.1.3 Add 0.50mL of each spiking solution prepared in section 8.2.1 and 8.2.2 to each of the digestion tubes labeled LCS, LCSD, MS, and MSD.

9.1.4 Add 2.5mL of concentrated HCl and 1.0mL of concentrated HNO₃ to each sample, MB, LCS, LCSD, DUP, MS, MSD. Place the batch into the preheated 95 °C ± 5 °C digestion block. Digest and evaporate the sample until the volume is approximately 15mL-20mL, which typically takes 5 hours. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

9.1.5 Dilute the sample digestate to 50mL with DI water.

NOTE: The digestate may be evaporated and diluted to a volume of 12.5mL if sample concentration is required to meet lower reporting limits. The pre-concentration must be batch specific and is called a X4 concentration.

9.1.6 Record the following information in the LIMS prep batch:

Enter into batch information:

- Batch start date/time (digestion start)
- Batch end date/time (digestion endt)
- Method #
- Analyst's name
- Digestion tube lot #
- Digestion block #
- Fume hood #
- Nitric acid lot #
- Hydrochloric acid lot #
- Digestion block temperature
- Thermometer ID #

Sample list tab:

- LIMS sample ID (client sample ID will autopopulate)

Worksheet tab:

- Initial pH
- Initial amount (volume, mL)
- Final amount (volume, mL)
- Notes

Reagents tab:

- Spike ID #(s)
- Spike amount(s) added, mL

9.1.7 The digests are now ready for analysis by SOP TL-ME-070: *ICP Analysis (Methods 200.7 and 6010B)*.

9.2 Total Metals: This digestion procedure is used for total metals determination by ICP. This procedure is rarely used, but may be required on a project-specific basis.

9.2.1 Transfer three 5mL of extraction fluid diluted to 50mL with DI water to each of three labeled digestion tubes to serve as the method blank (MB), lab control standard (LCS), and lab control standard duplicate (LCSD).

9.2.2 Transfer a 5mL well-mixed aliquot diluted to 50mL with DI water of each sample to a clean digestion tube. Choose one sample per batch and add an additional 5mL well-mixed aliquot (diluted to 50 mL) of sample to a labeled digestion tube to serve as the sample duplicate (DUP). Sample matrix spike (MS) and sample matrix spike duplicate (MSD) are performed only if requested by the client. If insufficient sample is available to perform the DUP, an NCM is generated in accordance with SOP TL-CA-085: *Nonconformance and Corrective Action Procedures*.

- 9.2.3 Add 0.50mL of each ICP spiking solution from section 8.2.1 and 8.2.2 to each of the digestion tubes labeled LCS, LCSD, MS, and MSD.
- 9.2.4 Add 1.5mL of concentrated HNO_3 to each sample, MB, LCS, LCSD, DUP, MS, and MSD. Place the batch into the preheated $95^\circ\text{C} \pm 5^\circ\text{C}$ digestion block. Digest and evaporate the sample until the volume is approximately 10mL, which typically takes 5 hours. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.
- 9.2.5 Remove the digestion tubes from the digestion block and cool the beakers to room temperature. Add another 1.5mL portion of concentrated HNO_3 to each sample, MB, LCS, LCSD, DUP, MS, MSD. Place the batch into the preheated $95^\circ\text{C} \pm 5^\circ\text{C}$ digestion block. Digest and evaporate the sample until the volume is approximately 10mL. Continue heating the samples and adding additional 1.5mL portions of concentrated HNO_3 until the digestate is light in color or does not change in appearance after subsequent additions of HNO_3 . If a sample requires more than 6mL of HNO_3 to digest, contact the digestion lab supervisor for guidance.
- 9.2.6 Add 2.5mL of concentrated HCl to each sample, MB, LCS, LCSD, DUP, MS, MSD. Place the batch into the preheated $95^\circ\text{C} \pm 5^\circ\text{C}$ digestion block for 15 minutes.
- 9.2.7 Wash down the inside of the digestion tube with DI water and dilute the sample digestate to 50mL with DI water.
- 9.2.8 Record information in the LIMS prep batch as listed in Section 9.1.6.
- 9.2.9 The digests are now ready for analysis by SOP TL-ME-070: *ICP Analysis (Methods 200.7 and 6010B)*.

10.0 ANALYTICAL PROCEDURES

No items for this SOP.

11.0 DATA ANALYSIS AND CALCULATIONS

No items for this SOP.

12.0 QUALITY CONTROL AND QUALITY ASSURANCE

- 12.1 Analytical batching must be in accordance with SOP TL-AN-002: *Analytical Batching and Evaluation of QC Data*.
- 12.2 Initial and on-going demonstration of capability must be performed by the analyst in accordance with SOP TL-CA-092: *Evaluation of DOCs*.
- 12.3 The method detection limit must be determined initially, when conditions change that might affect the instrument sensitivity, or every three years, in accordance with SOP TL-CA-090: *Determination of the Method Detection Limit (MDL)*.
- 12.4 Data assessment, acceptance criteria, and corrective actions are determined by SOP TL-ME-070.

13.0 PREVENTATIVE MAINTENANCE AND TROUBLESHOOTING

- 13.1 All glassware should be cleaned in accordance with SOP TL-AN-060: *Glassware Cleaning Procedure*.
- 13.2 The temperature of the digestion block must be monitored daily. If the temperature required for sample preparation cannot be maintained the digestion block must be removed from service and repaired or replaced.

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).)
Broken Mercury Thermometers or Spilled Mercury	RCRA Hazardous Waste (Spilled mercury is cleaned up with a mercury clean-up kit and added to the mercury waste satellite accumulation container located in the General lab. Broken thermometers are placed inside a baggie and added to the satellite accumulation container. When full or annually, the container is taken to the waste disposal area to await shipment to a Mercury Recovery Facility.)

⌘ These waste streams may be combined if necessary.

→ These waste streams may be combined if necessary.

15.0 REFERENCES

- 15.1 TestAmerica Corporate Environmental Health and Safety Manual, 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).
- 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

No items for this SOP.

17.0 REVISION HISTORY

Revision 10: 06/06/2008

- 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, removed references to LQM, replaced with references to TL-QAM, *TestAmerica Tallahassee Quality Assurance Manual* and TALS LIMS
- 1.1: Added method descriptions to 3005A and 3010A
- 1.1, 2.1: Deleted reference to TCLP
- 1.2: Deleted references to RL and MDL (found in analytical SOP); renumbered 1.3 as 1.2
- 2.2: Deleted "diluted to 50mL" in first sentence
- 5.1: Added new allowance for lab preservation and associated requirement for 24-hour waiting period prior to digestion
- 5.2: Deleted reference to TCLP, added correct reference to SPLP SOP, added hold time from collection to SPLP extraction
- 6.3: Deleted (Teflon tubes for TCLP digestion)
- 9.0: Deleted reference to TCLP digestion
- 9.1.2, 9.2.2: Deleted reference to noting "INSUFFICIENT SAMPLE...." on digestion log
- 9.2: Deleted reference to TCLP, added "This procedure is rarely used, but may be required on a project-specific basis."
- 9.2.1, 9.2.2, 9.2.3: Deleted "Teflon"
- 9.2.1: Deleted reference to TCLP extraction fluids
- 9.2.3: Changed "TCLP spiking solution to "ICP spiking solution", change "8.3.1 and 8.3.2" to "8.2.1 and 8.2.2"
- 9.2.9: Corrected SOP name