
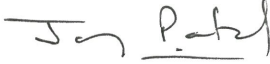
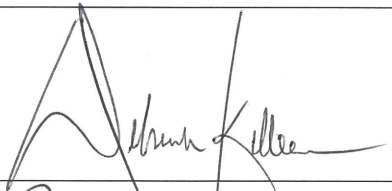



STANDARD OPERATING PROCEDURE APPROVAL AND CHANGE FORM

Scientific, Engineering, Response and Analytical Services 2890 Woodbridge Avenue Building 209 Annex Edison New Jersey 08837-3679	
STANDARD OPERATING PROCEDURE	
Title: DIGESTION AND ANALYSIS OF METALS BY INDUCTIVELY COUPLED PLASMA/ ATOMIC EMISSION SPECTROMETRY (ICP-AES) (EPA/SW-846 Methods 3015A/3051A/6010C)	
Approval Date: 12/12/2014	
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The top row of this table shows the most recent changes to the controlled document. For previous revision history information, archived versions of this document are maintained by the SERAS QA/QC Officer on the SERAS local area network (LAN).

History	Effective Date
Supercedes: SOP #1811, Revision 4.0, 1/10/12	12/15/14
Section 1.0 Deleted methods # 3050B	12/15/14
Section 2.0 Deleted paragraph: For solid samples digested by the hot block method, a representative 1 to 2 gram (wet weight) sample is digested with repeated additions of HNO ₃ and hydrogen peroxide (H ₂ O ₂). Hydrochloric acid (HCl) is added to the digestate and the sample is refluxed. The digestate is then diluted to a final volume of 100 mL with deionized (DI) water.	12/15/14
Section 5.0 Deleted: Hot block digestion system or hot plate, temperature-controlled	12/15/14
Section 6.0 Deleted - Working Soil (Hot Block digestion) LLQC Standards – Spike reagent blank with 1.0 mL of the appropriate Intermediate Soil (Hot Block) LLQC Standard and digest (Hot Block) with other samples.	12/15/14
Deleted entire section 7.3.1, Hot Block Digestion System (SW-846 Method 3050B)	12/15/14
Section 12.0 References: Deleted “U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 1996. <i>Test Methods for Evaluating Solid Waste</i> , SW-846, 3rd ed., Method 3050B, Revision 2.0.”	12/15/14
Section 13.0 Appendices - Deleted Appendix F - iCAP6500 Soil (Hot Block Digestion) LLQC Standard Preparation	12/15/14
Appendix A, Table 1. Deleted footnotes related to hot block digestion method 3050	12/15/14
Appendix C, Table 1.2. Delete table for “Intermediate LLICV/CCV Standard Preparation for Soil (Hot Block)	12/15/14



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 - D - Procedure for Determination of Total Percent (%) Solids
 - E - iCAP6500 Water LLQC Standard Preparation
 - F - iCAP6500 Soil (Microwave 3051A Digestion) LLQC Standard Preparation

SUPERCEDES: SOP #1811; Revision 4.0; 01/10/12; U.S. EPA Contract EP-W-09-031



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1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) outlines the digestion and analysis procedures for metals in water and solid matrices using inductively coupled plasma/atomic emission spectrometry (ICP/AES). This method is based on United States Environmental Protection Agency (EPA) Methods SW-846 3015A./3051A/6010C and those requirements set forth in the latest approved version of The NELAC Institute (TNO) standard. A list of target analyte list (TAL) compounds routinely analyzed by the Environmental Response Team/Scientific Engineering Response and Analytical Services (ERT/SERAS) Laboratory and their corresponding reporting limits (RLs) are provided in Table 1, Appendix A. These RLs are dependent upon the ICP instrumentation and operating parameters used (i.e., plasma power, sample aspiration method, and support gas flow rate), sample preparation method, and the sample matrix. Typical calibration standard concentrations are listed in Table 2, Appendix A. The analyst should be familiar with ICP spectroscopic techniques for correction of spectral, chemical, and physical interferences which can adversely affect the analysis and data integrity.

This method may not be changed without the expressed approval of the Analytical Support Leader and the Quality Assurance/Quality Control (QA/QC) Officer. Only those versions issued through the SERAS document control system may be used. Modifications made to the procedure due to interferences in the samples or for any other reason must be documented in the case narrative and on a nonconformance memo.

2.0 METHOD SUMMARY

For aqueous samples, a representative 45-milliliter (mL) sample is digested with 5 mL of concentrated nitric acid (HNO₃) in a Teflon digestion vessel using microwave heating. After the digestion process, the sample is cooled, and then filtered or allowed to settle in a clean sample bottle prior to analysis.

For solid samples digested by the microwave method, a representative 0.5 g sample is digested with HNO₃ and HCl in a microwave digestion system. After digestion is completed, the sample is cooled and diluted to a final volume of 50 mL with DI water.

Simultaneous or sequential, multi-element determination of metals in solution may be achieved using ICP spectroscopic techniques. The method makes multi-elemental determinations using simultaneous emission spectra by optical spectrometry. Sample solutions are nebulized and the resultant aerosol is transported to the plasma torch where the spectrum of analyte-specific atomic-line emissions is produced. The spectrum is dispersed by a diffraction grating and the intensities of the individual emission lines are monitored by photomultiplier tubes or a solid-state sensor. Background correction is required for trace element determination and is measured at wavelengths adjacent to the analyte lines during analysis. The positions selected for background intensity measurement are determined by the complexity of the spectrum adjacent to the analyte lines. These positions must be free of spectral interference and must reflect the same change in background intensity as that occurring at the analyte lines. The possibility of additional matrix-dependent interelement effects should be recognized and appropriate corrections made.



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3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Sample holding times, suggested collection amount, preservative, and type of containers are as follows:

Measurement Parameter	Matrix	Volume/Weight Required	Type of Containers	Preservative	Holding Time
<u>Metals</u> (except hexavalent chromium and mercury):					
Dissolved	Aqueous	1000 mL	P, G	Filter on site HNO ₃ to pH < 2	6 months
Suspended	Aqueous	1000 mL	P, G	Filter on site HNO ₃ to pH < 2	6 months
Total	Aqueous	1000 mL	P, G	HNO ₃ to pH < 2	6 months
Total	Solids	200 g	G	None	6 months

P - plastic, G - glass, g - grams, mL - milliliters

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

During the microwave process, water samples that contain organics will result in higher vessel pressures that have the potential to cause venting of the digestion vessels. This can result in loss of analytes and/or sample. A smaller volume of sample diluted to 45 mL may be used but the dilution must be taken into account during the final calculation of analyte concentration.

Solid samples may contain diverse matrix types. Spiked samples and other relevant quality control (QC) samples may aid in determining if there are any interferences.

Spectral interferences are the most common type of interference in ICP spectroscopy. They are caused by: 1) interelement effects due to overlap of an emission line from another element or stray light from the line emission of high concentration elements, and 2) background contribution from continuum or recombination events. Inter-element corrections (IECs) compensate for overlap or stray light spectral interferences. Background emission and stray light are compensated by background correction measurements adjacent to the analyte lines.

Physical interferences are associated with the sample nebulization or transport process. Changes in viscosity and surface tension may cause significant inaccuracies, especially for samples containing high dissolved solids or high acid concentrations. When physical interferences are present, they can be reduced by diluting the sample, by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer.

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant for ICP spectroscopic techniques. If observed, they can be minimized by careful selection of instrument operating parameters, buffering the sample, and matrix matching. Chemical interferences are highly dependent upon the specific element and sample matrix type.



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ICP analyzers may have hydroxyl band interference for aluminum measurement. Due to this interference, the Reporting Limit (RL) for aluminum is typically elevated to 100 micrograms per liter ($\mu\text{g/L}$) or higher depending on the sample matrix type.

5.0 EQUIPMENT/APPARATUS

- Thermo iCAP6500 Duo Inductively Coupled Plasma Spectrometer simultaneous emission spectrometer with interelement and background correction capabilities, computer-controlled with iTEVA Security software and including printer, monitor and personal computer
- CEM Microwave Digestion System, MARS or MARSExpress, with temperature control and rotating turntable, well ventilated with corrosion-resistant cavity
- Electronic Variable Volume Autopipettors, BIOHIT 500 to 5000 microliters (μL) and BIOHIT 100 to 1000 μL or equivalent
- Microwave digestion vessels for water, soil, sediment, sludge, and oil samples, Teflon, capable of holding ~75 mL, designed for temperatures up to 260 degrees Centigrade ($^{\circ}\text{C}$) with self-regulating pressure control
- Digestion vessels for soil samples, capable of holding ~50 mL
- Watch glasses or vapor recovery device
- Glass dispensers, 2-liter (L), 1-L, or 1-gallon, checked quarterly for accuracy
- Graduated Cylinders, Class A, 50 mL
- Volumetric flasks, Class A, assorted volumes or equivalent container (verified to be accurate)
- Balance, top-loading, capable of reading to 0.01 g, for weighing digestion vessels before and after digestion, and weighing soil samples, capable of reading to 0.001 g
- Funnels, plastic or equivalent
- Henke SASS plastic syringes or equivalent
- Corning SCFA 0.45 microns (μm) filters or equivalent

6.0 REAGENTS

- DI water, Type I Water (American Society for Testing and Materials [ASTM] D1193), for the preparation of all reagents and calibration standards and as dilution water
- Nitric acid, concentrated, trace metal grade
- Nitric Acid, 10 percent (%) volume to volume (v/v), for the preparation of working standards, also



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to be used for the Initial Calibration Blank/Continuing Calibration Blank (ICB/CCB)

- Hydrochloric Acid, concentrated, trace metal grade
- Hydrogen Peroxide, 30%, reagent or trace grade
- Argon Plasma Support Gas, commercially available, in pressurized cylinders or from the gas outlet of a liquid argon Dewar
- Primary Stock Calibration Standards, 1000 milligrams per liter (mg/L), commercially available, accompanied by a certificate of analysis, for all elements except aluminum (Al), calcium (Ca), iron (Fe), magnesium (Mg), potassium (K), and sodium (Na).
- Primary 2nd Source Standards, 1000 milligrams per liter (mg/L), commercially available, accompanied by a certificate of analysis, for boron (B), strontium (Sr), and tin (Sn).
- Primary Stock Calibration Standards, 10,000 mg/L, commercially available, accompanied by a certificate of analysis, for Al, Ca, Fe, Mg, K, and Na.

NOTE: Stock standard solutions may also be prepared from high purity metals, oxides, or non-hygroscopic reagent-grade salts using Type I water and trace metal grade HNO₃. Sulfuric or phosphoric acids should be avoided as they produce an adverse effect on many elements.

- Primary Working Calibration Standard - Prepare 200 mL of this working calibration standard in 10% nitric acid using the volumes listed in Table 2.
- Stock Initial Calibration Verification/Continuing Calibration Verification (ICV/CCV) Standard 1, 100 mg/L containing all analytes (except boron (B), strontium (Sr), tin (Sn)), 2nd source, commercially available, accompanied by a certificate of analysis
- Stock ICV/CCV Standard 2, 5000 mg/L of Al, Ca, Fe, Mg, K, and Na, 2nd source, commercially available, accompanied by a certificate of analysis
- Working Mid-Range ICV/CCV containing all analytes at 2 mg/L except Al, Ca, Fe, Mg, K, Na at 20 mg/L - Add 10 mL of the stock 100 mg/L ICV/CCV, 1 mL stock 1000 mg/L B, 1 mL stock 1000 mg/L Sr, 1 mL stock 1000 mg/L Sn, and 1.8 mL of stock 5000 mg/L ICV/CCV to 500 mL using 10% nitric acid in a Class A volumetric flask.
- Intermediate Water Low Level Initial Calibration Verification/Low Level Continuing Calibration Verification (LLICV/LLCCV) Standard – Add the volumes of Primary Stock Calibration Standards specified in Table 1-1, Attachment 1 for the iCAP6500.
- Intermediate Soil LLICV/LLCCV Standard – Add the volumes of Primary Stock Calibration Standards specified in Tables 1-2 and 1-3, Attachment 1 for the iCAP6500.
- Working Water LLICV/LLCCV - Dilute 0.5 mL of the Intermediate Water LLICV/LLCCV Standard to 500 mL in a Class A volumetric flask using 10% nitric acid.



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- Working Soil LLICV/LLCCV - Dilute 0.5 mL of the appropriate Intermediate Soil LLICV/LLCCV Standard to 500 mL in a Class A volumetric flask using 10% nitric acid.

NOTE: Depending on the annual Limit of Detection/Limit of Quantitation (LOD/LOQ) studies, the intermediate and/or working LLICV/LLCCV standards are subject to change.

- Working ENDCCV containing all analytes at 2.5 mg/L except Al, Ca, Fe, Mg, K, Na at 12.5 mg/L - Add 12.5 mL of the stock 100 mg/L ICV/CCV, 1.25 mL stock 1000 mg/L B, 1.25 mL stock 1000 mg/L Sr, 1.25 mL stock 1000 mg/L Sn, and 1 mL stock 5000 mg/L ICV/CCV to 500 mL using 10% nitric acid in a Class A volumetric flask.
- iCAP6500 Intermediate Water Lower Level Quality Control (LLQC) Standards – Add the volumes of Primary Stock and Intermediate Standards specified in Appendix E for the iCAP6500.
- iCAP6500 Intermediate Soil (Hot Block digestion) LLQC Standards – Add the volumes of Primary Stock and Intermediate Standards specified in Appendix F for the iCAP6500.
- iCAP6500 Intermediate Soil (Microwave 3051A digestion) LLQC Standards – Add the volumes of Primary Stock and Intermediate Standards specified in Appendix F for the iCAP6500.
- Working Water LLQC Standards - Dilute 1.0 mL of the appropriate Intermediate Water LLQC Standard to 100 mL in a Class A volumetric flask using 10% nitric acid (see Appendix E).
- Working Soil (Microwave 3051A digestion) LLQC Standards – Spike reagent blank with 0.5 mL of the appropriate Intermediate Soil (Microwave 3051A) LLQC Standard and digest (Microwave 3051A) with other samples.

NOTE: Depending on the annual Limit of Detection/Limit of Quantitation (LOD/LOQ) studies, the intermediate and/or working LLQC standards are subject to change.

- Laboratory Control Sample (LCS) and Matrix Spike/Matrix Spike Duplicate (MS/MSD) Intermediate Spiking Solution – Prepare the spiking solution in 10% nitric acid at the concentrations listed in the Spiking Solution Log in Attachment 3, Appendix C using 2nd source standards.
- Post Digestion Spike (PDS) Spiking Solution - Prepare the PDS spiking solution in 10% nitric acid at the PDS Spike solution concentrations listed in Table 3, Appendix A using the primary calibration standards.
- Soil Laboratory Control Sample (LCS), commercially available, accompanied by a certificate of analysis, or a blank matrix spiked with the target analytes from an independent source at or near mid-range of the calibration. Prepare the LCS with each analytical batch of 20 samples.
- Stock Interference Check Standard (ICS) containing interferents (INT-A1) - Al, Ca, Mg at 5000 mg/L and Fe at 2000 mg/L, commercially available, accompanied by a certificate of analysis.



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- Intermediate ICSB Standard (10 mg/L) - Add 1.0 mL each of stock 1000 mg/L of Sb, As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Sr, Tl, Sn, V and Zn standards to 100 mL using 10% nitric acid in a Class A volumetric flask.
- Working ICSA - Add 20 mL of the stock INT-A1 standard to 200 mL using 10% nitric acid in a Class A volumetric flask.
- Working ICSAB - Add 20 mL of the stock INT-A1 standard and 2 mL of the intermediate ICSB standard to 200 mL using 10% nitric acid in a Class A volumetric flask.

NOTE: The working ICSAB standard may also be prepared using commercially available ICSA and ICSB standards.

- Internal Standard Solution : iCAP6500 - Add 8 mL of stock 1000 mg/L yttrium (Y) and 5 mL of stock 10000 mg/L indium (In) to 1000 mL using 10% nitric acid in a 1-liter polyethylene bottle
NOTE: 1000 mg/L Y, 10000mg/L In, and reagent grade LiNO₃ are commercially available.

NOTE: Premixed certified standards will be stored according to the manufacturer's documented storage requirements. These standards may be kept in storage up to the manufacturer's stated expiration date. Once dilutions are made, the standards will be stored for a period not to exceed six months or the manufacturer's expiration date, whichever is less.

NOTE: All calibration standards and spiking solutions will be prepared and documented in accordance with SERAS SOP #1012, *Preparation of Standard Solutions and Reagents*.

NOTE: Stock concentrations may vary depending on the vendor; thus, the concentration of intermediate standards may also vary. Preparation instructions may differ based on the volumes prepared and the concentrations of standards available. The final working standard concentrations will remain as stated in the above section.

7.0 PROCEDURE

7.1 Glassware Cleaning Procedures

All glassware and Teflon containers should initially be cleaned using the following sequence: detergent, tap water, 1:1 nitric acid, tap water, and Type I water. If it can be documented through an active analytical quality control (QC) program using spiked samples and reagent blanks that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

7.2 Water Sample Digestion (SW-846 Method 3015A)

1. Weigh the empty digestion vessel, valve and cap to 0.01 g. Record the weight on the sample digestion log.
2. Check the pH of the aqueous sample to ensure that the pH is less than (<) 2. Record the pH on the sample digestion log. If the pH is not <2, adjust the sample pH to <2 with



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additional concentrated HNO_3 . Do not add more than 1% of the total volume of sample. Note the volume of HNO_3 added on the sample digestion log. A nonconformance memo must be written if pH is greater than or equal to 2.0 prior to adjustment.

3. Measure 45 mL of a well mixed sample in a Class A graduated cylinder. Transfer the sample to a digestion vessel and mark the digestion vessel with the sample number.
4. Measure 45 mL of DI water in a Class A graduated cylinder and transfer to a digestion vessel. Mark the digestion vessel as the method blank. One method blank must be prepared for each analytical batch not to exceed 20 samples.
5. Measure 45 mL of DI water in a Class A graduated cylinder and transfer to a digestion vessel. Mark the digestion vessel as the LCS; add 0.45 mL of the LCS and MS/MSD intermediate spiking solution. One LCS must be prepared for each analytical batch not to exceed 20 samples.
6. Measure two separate well-mixed 45-mL portions of the sample chosen for the MS/MSD in a Class A graduated cylinder. Transfer these portions of samples to separate digestion vessels, add 0.45 mL of the LCS and MS/MSD intermediate spiking solution to each and mark the digestion vessels as the MS and MSD. At least one MS/MSD pair must be digested with every 20 samples similar in matrix or per project.
7. Add 5 mL of concentrated HNO_3 to each vessel. Check to make sure that the pressure relief disks are in the caps and hand-tighten the caps on the vessels. Weigh each digestion vessel and record the weight on the sample digestion log.
8. Evenly distribute the vessels in the carousel according to the manufacturer's instructions. When fewer than the recommended number of vessels is digested, include additional vessels with the same volume of DI water and concentrated HNO_3 as the samples.
9. Program the microwave digestion unit according to the manufacturer's recommended specifications to bring the samples to 160 ± 4 °C in 10 minutes and rise slowly to between 165 and 170° C in the second 10 minutes. Power settings may be adjusted as long as they result in the same time and temperature conditions.
10. At the end of the digestion, allow the digestion vessels to cool for a short period of time in the microwave and then on the bench or in a water bath. Once the vessels are cool, weigh and record the weight of each digestion vessel assembly. If the weight of the digestate has decreased more than 10%, discard the sample.
11. Uncap and vent each vessel in a fume hood and transfer the sample to an acid-cleaned sample bottle. If the digested sample contains particulates, filter prior to analysis using the plastic syringes and the 0.45 μm filters, or equivalent.
12. Correct the concentration values from analysis by a dilution factor of 1.11 (45 mL sample + 5 mL of acid/45 mL of original sample)



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NOTE: For TCLP extract samples, take an aliquot for MS/MSD analysis and then adjust the pH of the remaining sample to pH <2 with HNO₃ prior to digestion.

7.3 Solid Sample Digestion

7.3.1 Microwave Digestion System (SW-846 Method 3051A)

1. Weigh the empty digestion vessel, valve and cap to 0.01 g. Record the weight on the sample digestion log.
2. Accurately weigh approximately 0.500 g of a well-mixed sample to the nearest 0.001 g and transfer to a digestion vessel.
3. Mark a separate digestion vessel as the method blank. One method blank must be prepared for each analytical batch not to exceed 20 samples.
4. Accurately weigh approximately 0.500 g of the LCS and transfer to a digestion vessel. Mark the digestion vessel as the LCS. One LCS must be prepared for each analytical batch not to exceed 20 samples.

NOTE: A blank spike (e.g. reagent blank spiked with target analytes from an independent source at or near mid-range of the calibration) may be used in lieu of the LCS. Add 1.0 mL of the LCS and MS/MSD intermediate spiking solution to reagent blank.

5. Weigh two separate well-mixed portions of the sample chosen for the MS/MSD and transfer these portions to separate digestion vessels. Add 1.0 mL of the LCS and MS/MSD intermediate spiking solution to each and mark the digestion vessels as the MS and MSD. At least one MS/MSD must be digested with every 20 samples of similar matrix or per project.
6. Add 9 mL of HNO₃ and 3 mL HCl to each digestion vessel in a fume hood. Alternatively, add 10 mL of HNO₃ (the addition of HCl to nitric acid is appropriate for stabilization of certain analytes such as silver, barium, and antimony and high concentrations of iron and aluminum in solution).

NOTE: The analyst should be aware of the potential for a vigorous reaction, especially with samples containing volatile or easily oxidized organic species. When digesting a matrix of this type, do not leach this type of sample as described in this method, due to the high potential for unsafe and uncontrollable reactions. Instead, these samples may be predigested in a hood for approximately 30 minutes at room temperature until the carbonate reaction subsides, with the vessel loosely capped to allow gases to escape, eliminating the hazard presented by rapid addition of thermal energy (MW power) to a reactive mixture. After predigestion, the samples may be digested according to the procedures described in this method.



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Weigh the digestion vessel, sample, digestion acids, valve and cap to 0.01 g. Record the weight on the sample digestion log.

7. Evenly distribute the vessels in the carousel according to the manufacturer's instructions. When fewer than the recommended number of vessels are digested, be sure to include additional vessels with the same volume of concentrated HNO₃ and concentrated HCl as the samples.
8. Program the microwave digestion unit according to the manufacturer's recommended specifications to bring the samples to 175 ± 5 °C in approximately 5.5 ± 0.25 minutes and remain at 175 ± 5 °C for 4.5 minutes, or for the remainder of the 10-minute digestion period.
9. At the end of the digestion, allow the vessels to cool for a minimum of 5-minutes before removing them from the microwave system. Once the vessels are cool, weigh and record the weight (to 0.01 g) of each digestion vessel assembly. If the weight loss of sample exceeds 1% of the weight of the sample and reagents, then the sample is considered compromised. For vessels with burst disks, a careful visual inspection of the disk, in addition to weighing, may identify compromised vessels. For vessels with resealing pressure relief mechanisms, an auditory or a physical sign that can indicate whether a vessel has vented is appropriate.
10. Uncap and vent each vessel in a fume hood, transfer the sample to an acid-cleaned sample bottle, and dilute to 50 mL with DI water using a Class A volumetric flask (see section 5). Remove particulates by filtering prior to analysis.
11. Correct the concentration values from analysis using the sample weight, final volume and % total solids.

7.4 Total Solids

A total solids analysis must be conducted for soil, sediment, solids, and semisolid samples to report data on a dry weight basis. Refer to Appendix D for detailed instructions.

7.5 ICP Operation

Differences between the various makes and models of satisfactory ICP spectrometers preclude the formation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for a particular instrument. Daily operation and troubleshooting procedures for the Thermo Electron iCAP6500 analyzer are in Appendix B. Element information for the iCAP6500 is in Attachment 4, Appendix C.

After igniting the plasma, the instrument should be allowed to thermally stabilize for 30 to 60 minutes prior to calibration. After the plasma has stabilized, the spectrometer optimization must be performed and the spectrometer must be calibrated according to the manufacturer's recommended procedures prior to sample analysis.



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1. Prior to analyzing samples (daily), calibrate the ICP using a blank and the working calibration standard (high standard).
2. Immediately following calibration, analyze the mid-range ICV standard. The % recovery must be within $\pm 10\%$ of its true value and the RSD for all elements must be less than 5%.
3. Immediately following the mid-range ICV, analyze the appropriate LLICV. The LLCCV may not be analyzed more than two times and the last run must be within the acceptance criterion for the run to proceed. The % recovery must be $\pm 30\%$ of its true value.
4. Analyze the ICB standard. The concentration must be $< RL$ for each element.
5. Analyze the ICSA, ICSAB, continuing calibration verification (CCV), and continuing calibration blank (CCB) standards. Refer to Section 9.0 for QC criteria.
6. Analyze the method blank, LCS and samples.
7. The CCV and CCB standards must be run after every 10 samples. OPTIONAL: The LLCCV standard may be analyzed after every 10 samples (after CCV and before CCB) if low-level sample concentrations are anticipated and the system stability at the low end of the calibration is questionable. The LLCCV may not be analyzed more than two times. Refer to Section 9.14 for the acceptance criterion. At the end of the analytical sequence, the ENDCCV, RL and CCB standards must be analyzed. Refer to Section 9.0 for QC criteria.
8. The ICSA and ICSAB standards must be run at the beginning of the sequence and when the sequence exceeds an 8-hour shift.
9. If the measured concentration is greater than the linear analytical range (Section 9.8), the sample must be diluted with the appropriate acid matrix blank and reanalyzed (Section 9.10).
10. To verify the absence of interference, follow the serial dilution analysis procedure in Section 9.9.
11. To verify that matrix effects are present for analytes that fail MS/MSD criteria (Section 9.7), follow the post digestion spike addition procedure in Section 9.18.

8.0 CALCULATIONS

8.1 Sample Concentration

For the determination of metals concentration in solution, the value in micrograms per liter ($\mu\text{g/L}$) for the iCAP6500 is read directly from the read-out system of the instrument. For aqueous samples, report concentrations as $\mu\text{g/L}$.



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If a dilution of the sample is required:

$$\text{concentration of metal in sample, } \mu\text{g/L} = A \frac{C+B}{C}$$

where:

- A = $\mu\text{g/L}$ of metal in diluted aliquot from read-out
- B = Acid blank matrix used for dilution, mL
- C = Sample aliquot, mL

For solid samples, all concentrations are calculated as mg/kg based on wet weight; therefore:

$$\text{mg metal / kg sample} = \frac{A \times V}{1000 \times W} \times DF$$

where:

- A = $\mu\text{g/L}$ of metal in processed sample from read-out
- V = Final volume of the processed sample, mL
- W = Weight of sample, grams
- DF = Dilution factor for diluted samples (1.00 with no sample dilution)

If dilution of the sample is required, the DF is given by:

$$DF = \frac{C+B}{C}$$

where:

- B = mL of acid blank matrix used for dilution
- C = mL of sample aliquot

For solid samples, report concentrations as mg/kg based on dry weight:

$$\text{mg metal / kg sample (dry basis)} = \frac{\text{mg / kg (wet basis)}}{(S/100)}$$

where:

- S = percent total solids in the sample

8.2 Laboratory Control Sample

Percent recovery (%R) must be within the specifications supplied by the vendor or within 80-120% and calculated as:



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$$\%R = \frac{(LCS - BLK)}{SA} \times 100$$

where:

LCS = LCS result, $\mu\text{g/L}$ or mg/kg

SA = Spike added or certified value, $\mu\text{g/L}$ or mg/kg

BLK = Method Blank results, $\mu\text{g/L}$ or mg/kg

8.3 Matrix Spike/Matrix Spike Duplicate Sample

Spike sample percent recovery (%R) must be within 75-125% or within control chart limits (see section 9.7) and calculated as:

$$\%R = \frac{(MS - SR)}{SA} \times 100$$

where:

MS = Spiked sample result (MS or MSD)

SR = Sample result

SA = Spike added

The Relative Percent Difference (RPD) of matrix spike and matrix spike duplicate samples should be within $\pm 20\%$ and calculated as:

$$RPD = \frac{|MS - MSD|}{(MS + MSD)/2} \times 100$$

where:

MS = matrix spike sample concentration

MSD = matrix spike duplicate sample concentration

9.0 QUALITY ASSURANCE/QUALITY CONTROL

All QC data should be maintained and available for easy reference and inspection (see the Troubleshooting sections of Appendix B for QC failure actions).

9.1 Initial Calibration Verification Standards

The calibration curve must be verified by the analysis of an ICV standard (at or near mid-range) from an independent source and a LLICV using the same source as the calibration standards at or near the lower limit of quantitation. The acceptance criteria for the mid-range ICV standard must be $\pm 10\%$ of the true value. The suggested acceptance criterion for the LLICV is $\pm 30\%$ of its true value. If the mid-range ICV is outside QC limits, the cause needs to be determined and the



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instrument recalibrated before samples are analyzed. Quantitative sample analyses should not proceed for analytes that fail the mid-range ICV. However, analyses may continue with an understanding that these results should be qualified and would be considered estimated values. These samples may also be re-analyzed for the affected analytes at a later time. The RSD for all analytes in the mid-range ICV must be <5%.

9.2 Interference Check Standards

ICP interelement corrections are verified by analyzing ICS samples at the beginning and when the analytical run exceeds an 8-hour shift. Results must be within $\pm 20\%$ of the true value for each element present in the ICS sample. The control limits for elements not present in the ICSA sample will be the respective Reporting Limits (RLs, see section 9.13) and accounting for impurities in the stock ICSA sample (based on the certificate of analysis). The ICP operator will scan the ICSA raw data printout, check to verify that all elements are in control, and sign/date the raw data printout (noting any exceptions). IEC factors must be updated every six months.

9.3 Continuing Calibration Verification

The working standard curve must be verified by analyzing the CCV standard (at or near the mid-range) after every 10 samples, and the ENDCCV at the end of the sequence. The acceptance criteria for CCV/ENDCCV results should be $\pm 10\%$ of the true value. If CCV/ENDCCV results are outside QC limits, the cause needs to be determined and the previous ten samples must be reanalyzed. Quantitative sample analyses should not proceed for analytes that fail CCV/ENDCCV verification. However, analyses may continue with an understanding that these results should be qualified and would be considered estimated values. These samples may also be re-analyzed for the affected analytes at a later time. The RSD for all analytes in the CCV/ENDCCV must be <5%.

9.4 Initial/Continuing Calibration Blanks

The ICB/CCB results must be less than the reporting limit (RL) for the sequence to continue. However, analyses may continue for analytes that fail the criteria with an understanding that these results should be qualified and would be considered estimated values.

9.5 Method Blank

A method blank must be prepared for each analytical batch of samples (not to exceed 20 samples) of the same matrix. The method blank results must be less than the RL. A method blank containing an analyte concentration $>RL$ may be used in instances when the sample concentrations are at least 10 times the method blank concentration.

9.6 Laboratory Control Sample

Aqueous and solid LCSs must be obtained from an independent source, and must be prepared with each analytical batch of samples using the same preparation method as employed for the samples with the frequency of 1 LCS for 20 samples per matrix. The LCS sample may be either a certified reference material or a blank matrix spiked with the target analytes from an independent source at or near mid-range of the calibration. LCS results for each analyte must be within the specifications



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supplied by the vendor or within 80 - 120% of the true value and calculated as in Section 8.2. If the LCS is not acceptable, then the LCS should be re-run once and, if still unacceptable, all samples in the analytical batch with the failing LCS should be re-prepared and reanalyzed

9.7 Matrix Spike/Matrix Spike Duplicate

At least one MS and one MSD sample must be digested with every 20 samples of the same matrix or with each project. MS/MSD recovery will not be calculated for analytes with concentration greater than 4-times the spike level. MS/MSD recoveries must be within 75-125% of the true value for all analytes. Alternatively, control chart limits may be used. The warning and control limits will be calculated using the last 10 sets of MS/MSD data or the last 20 data points, updated each time a batch is analyzed. Recoveries are calculated as in Section 8.3.

Control limit data will consist of the mean (\bar{x}) of the 20 data points, the standard deviation (σ), the upper and lower warning limits (UWL/LWL) and the upper and lower control limits (UCL/LCL). The UWL/LWL will be calculated using the $\bar{x} \pm 2 \sigma$. The UCL/LCL will be calculated using the $\bar{x} \pm 3 \sigma$.

In the event there is not sufficient sample available in the batch to run a MS/MSD, a LCS/laboratory control sample duplicate (LCSD) will be run. LCS/LCSD results for each analyte must be within the specifications supplied by the vendor or within 80-120% of the true value, if laboratory prepared. Recoveries will be calculated as in Section 8.2.

The Relative Percent Difference (RPD) of MS/MSD samples must be within $\pm 20\%$ and calculated as in Section 8.3.

9.8 Linear Analytical Range

One or more linear analytical range (LAR) standards must be analyzed to determine the maximum linear range of the calibration for each element. Recovery must be within 90-110 %. If the recovery is outside these limits, the maximum calibration standard concentration defines the linear range. At a minimum, LAR standards are analyzed and updated on a semi-annual basis. They may be prepared from the stock standards used to prepare calibration standards or from second source standards.

9.9 Serial Dilution

A sample (typically the sample chosen for the MS/MSD) from each project in an analytical batch is analyzed at a 5x dilution in conjunction with the samples. If the analyte concentration is sufficiently high (at least a factor of 10 above the RL after dilution), an analysis of a 1:5 dilution should agree within $\pm 10\%$ of the original determination. If not, a chemical or physical interference effect should be suspected.

If the concentration of all analytes in all samples is less than 10x the RL, the serial dilution will not be performed. NOTE: Only one serial dilution analysis for each batch and/or project will be included with the data package.



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9.10 Dilution Analysis

If the concentration of any analyte in any sample exceeds the linear range, the sample must be diluted and re-analyzed. An appropriate dilution or series of dilutions (for example, 5x, 10x, 20x) may be required depending on the concentration in the undiluted sample. Results are reported from the lowest dilution that falls within the linear range.

If chemical/physical matrix effects are suspected or for analytes that saturate the detector or Internal Standard (IS) counts are significantly high (e.g., > 10%) compared to the typical range of values based on the current calibration, samples must be diluted and re-analyzed. If the IS counts are approaching the 10% criterion, the analyst may use professional judgment to determine whether or not a sample needs to be diluted further and re-analyzed. An appropriate dilution or series of dilutions may be required depending on the concentrations or IS counts in the undiluted sample. Comparisons are first made with respect to the undiluted sample and then, within the series. Based on the analyst's professional judgment, results may be reported from the undiluted sample or from the diluted sample that has the smallest dilution factor and indicates the absence of interferences. The analyst may encounter a LCS with high IS counts; if results for all analytes are within specifications (see section 9.6), the LCS does not need to be diluted and re-analyzed. The analyst must document the reason(s) for dilution analysis and its relevance to data processing in the case narrative.

An optional approach to determine if chemical/physical matrix effects are present is to use post digestion spike (PDS) analysis.

9.11 Demonstration of Capability

Prior to running a method or any time there is a change in instrument type, personnel or test method or any time a method or analyst has not performed a method in a twelve-month period, an analyst must demonstrate acceptable performance for that method. This is known as the initial demonstration of capability (DOC). Four aliquots of a QC standard (second source) must be prepared and analyzed according to the method over one or a period of days. If the method does not specify a concentration to be used for the DOC, then the concentration must be prepared at 1-4 times the RL. Using Excel, the analyst must provide a table containing the following: Results of the 4 replicates, Mean Concentration, Mean Recovery, Standard Deviation and %RSD with the LCS acceptance limits.

On-going demonstration of capability on an annual basis may be satisfied by the analyst successfully passing a performance testing (PT) sample or a minimum of 4 consecutive LCS samples with acceptable precision and accuracy levels or another initial DOC. Results of tabulated QC samples (i.e., control chart data) that are analyst-specific accompanied by precision and accuracy criteria agreed upon by the Analytical Task Leader and the QA/QC Officer may also be used.



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9.12 Limit of Detection/Limit of Quantitation Studies

The LOD must be determined for each target analyte on every instrument that will be used for the analysis and reporting of samples. The LOD must be determined each time there is a change in the method that affects how the test is performed or when there is a change in instrumentation. The LOD must be verified annually for each matrix, method and analyte. The LOD will be run using a minimum of seven replicates of a sample prepared from the calibration source at 4 times the LOD for multiple analyte tests. Each of these 7 aliquots must be subjected to the entire analytical procedure. Calculate the mean, mean recovery, variance and standard deviation of the replicate measurements. The LOD is calculated by multiplying the standard deviation times the Students t-Value of 3.143. If more than 7 replicates are used, the Students t-Value must be adjusted accordingly.

The LOQ must be confirmed by the successful analysis of a secondary source QC sample containing each target analyte in each matrix at 1-2 times the claimed LOQ (typically the low standard). For the LOQ, a LLQC standard from a secondary source is prepared at a concentration 1 to 2 times the RL. The recoveries must fall within the recoveries established for the LCS. Alternately, a control chart with recoveries established from previously analyzed LOQ samples may be used. The LOQ must be subjected to the entire analytical procedure. The LOQ must be verified annually for each matrix, method and analyte. The LOD/LOQ must be determined at the same time.

9.13 Reporting Limit

For ICP analysis, the RL is defined as five times the statistically calculated LOD. An average LOD over a period of three to four years is calculated, multiplied by a factor of five, and compared with the current LOD study value multiplied by five. To maintain consistent RLs for the client, the average RLs are used as long as the currently calculated RLs do not exceed this average. In the event that the current RLs are greater than the average RLs, the client will be notified that the RLs will be adjusted.

9.14 Low Level Quality Control Standard

The LLQC standard is run annually as the LOQ. The LLQC may be digested with a soil or water batch and run on an "as needed" basis to verify the RL. If the LLCCV analyzed during a run to verify the RL does not meet the acceptance criterion, data <2X the RL will be qualified estimated (J). The suggested acceptance criterion for the LLQC standard is $\pm 30\%$ of its true value.

9.15 Instrument Detection Limits

IDLs are run quarterly on each ICP and are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with a minimum of seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs are useful for monitoring response changes over time.



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9.16 System Troubleshooting

Refer to Appendix B for instrument operation and troubleshooting.

9.17 Nonconformance Memo

A nonconformance memo will be generated any time the QC requirements of this SOP are not met or when an employee notices a deficiency suspected of being a nonconformance. This nonconformance memo will be included with the data package.

9.18 Post Digestion Spike (PDS) Addition

A post digestion spike (PDS) addition will be performed on the MS/MSD sample. If all MS/MSD recoveries are acceptable, results of the PDS will not be reported. If the MS/MSD recoveries are unacceptable, PDS recoveries should be calculated for all analytes with concentration less than 4-times the spike level. An analyte spike is added to a portion of the prepared sample, and should be recovered to within 80% to 120% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the Reporting Limit (RL). If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed. Add the PDS spiking solution to the sample at the levels indicated in Table 3, Appendix A; for example, 0.4 mL of PDS spike solution to 20 mL sample, or 0.2 mL PDS spike solution to 10 mL sample.

10.0 DATA VALIDATION

Data will be assessed based on the project's data quality objectives by the Data Validation and Report Writing Group using the most current revision of the SERAS SOP #1017, *Data Validation Procedure for Routine Inorganic Analysis*. However, data is considered satisfactory for submission purposes when all of the requirements listed in this procedure are met and the data package is subjected to the peer review process.

11.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for following the chemical hygiene plan and laboratory safety program regarding the safe handling of the chemicals specified in this method.

When working with potentially hazardous materials, refer to EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to SERAS SOP #3013, *SERAS Chemical Hygiene Plan*.

12.0 REFERENCES

Thermo Electron Corporation. 2005. ICAP 6000 Series ICP-OES Spectrometer Operator Manual.

Thermo Electron Corporation. 2009. ICAP 6000 Series ICP-OES Spectrometer User Guide.



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The NELAC Institute, 2009. TNI Standard, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, EL-V1-2009-ISO.

U.S. Environmental Protection Agency. 1984. Federal Register, 40 Code of Federal Regulations (CFR) Part 136, Appendix B, *Definition and Procedure of the Determination of the Method Detection Limit - Revision 1.11*, October 26, 1984.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 2007. *Test Methods for Evaluating Solid Waste, SW-846*, 3rd ed., Method 3015A, Revision 1.0.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 2007. *Test Methods for Evaluating Solid Waste, SW-846*, 3rd ed., Method 3051A, Revision 1.0.

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 2007. *Test Methods for Evaluating Solid Waste, SW-846*, 3rd ed., Method 6010C, Revision 3.0.

13.0 APPENDICES

A - Tables

B - iCAP 6500 ICP-AES (Thermo Scientific) Daily Operation Procedures

C - Attachments

1 - iCAP 6500 ICP-AES Water and Soil Intermediate LLICV/LLCCV Preparation

2 - Spiking Solutions

3 - iCAP 6500 ICP-AES Element Information

D - Procedure for Determination of Total Percent (%) Solids

E - iCAP6500 Water LLQC Standard Preparation

F - iCAP6500 Soil (Microwave 3051A Digestion) LLQC Standard Preparation



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TABLE 1. Typical ICP Detection and Reporting Limits for TAL Metals

ANALYTE	TYPICAL ANALYTICAL RANGE (mg/L)	TYPICAL INSTRUMENT DETECTION LIMIT (IDL, mg/L)	iCAP6500 ⁽¹⁾	
			TYPICAL REPORTING LIMITS	
			Soil, (mg/kg)	Water, (mg/L)
Aluminum	0.085 - 1000	0.015	10 - 20	0.1
Antimony	0.010 - 100	0.002	1.5	0.010
Arsenic	0.015 - 100	0.003	1.5	0.020
Barium	0.002 - 50	0.0001	0.3	0.002
Beryllium	0.002 - 25	0.0001	0.2	0.002
Boron	0.010 - 25	0.001	1	0.010
Cadmium	0.002 - 25	0.0001	0.2	0.002
Calcium	0.060 - 1000	0.005	6 - 10	0.06
Chromium	0.003 - 100	0.0002	0.4	0.003
Cobalt	0.002 - 100	0.0004	0.2	0.002
Copper	0.004 - 100	0.0006	0.5	0.005
Iron	0.025 - 1000	0.005 - 0.035	6 - 30	0.035 - 0.30
Lead	0.010 - 250	0.002	1	0.010
Magnesium	0.15 - 1000	0.020	20	0.16

⁽¹⁾ Thermo Electron iCAP6500 instrument: soil RL based . 0.500 gram sample diluted to final volume of 50 mL (microwave sample preparation method #3051); water RL based on sample preparation Methods #3015.



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TABLE 1. (cont) Typical ICP Detection and Reporting Limits for TAL Metals

ANALYTE	TYPICAL ANALYTICAL RANGE (mg/L)	TYPICAL INSTRUMENT DETECTION LIMIT (IDL, mg/L)	iCAP6500 ⁽¹⁾	
			TYPICAL REPORTING LIMITS	
			Soil, (mg/kg)	Water, (mg/L)
Manganese	0.002 - 100	0.0002	0.2	0.002
Molybdenum	0.005 - 50	0.001	0.5	0.005
Nickel	0.003 - 100	0.0005	0.3	0.003
Potassium	0.20 - 1000	0.025	25 - 50	0.25
Selenium	0.015 - 100	0.003	1.5	0.015
Silver	0.004 - 50	0.001	0.5	0.005
Sodium	0.20 - 50	0.20 - 1.00	30 - 100	0.20 - 1.20
Strontium	0.002 - 25	0.0002	0.20	0.002
Thallium	0.015 - 100	0.002	2	0.017
Tin	0.010 - 25	0.001	2	0.010
Vanadium	0.002 - 100	0.0005	0.3	0.002
Zinc	0.003 - 100	0.0003 - 0.004	0.70 - 3.5	0.003 - 0.020

⁽¹⁾ Thermo Electron iCAP6500 instrument: soil RL based . 0.500 gram sample diluted to final volume of 50 mL (microwave sample preparation method #3051); water RL based on sample preparation Methods #3015.



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TABLE 2. Typical ICP Calibration Standard Concentrations for Elements in Soil and Water Matrices			
Volume of 1000 mg/L Stock Cal Standard, mL ⁽¹⁾	Volume of 10000 mg/L Stock Cal Standard, mL ⁽¹⁾	Elements	Final Working Standard Concentration, mg/L ⁽²⁾
1	-	Be, Cd, Cr Co, Cu, Mn, Ni, Ag, Zn, Ba, V, Sb, As, Se, Tl, Pb, Mo, B, Sn, Sr	5
-	1	Al, Ca, Mg, Na	50
-	0.5 - 1.0 ⁽³⁾	Fe, K	25 - 50 ⁽³⁾

⁽¹⁾ Use single element stock calibration standards (see Section 6)

⁽²⁾ Final volume = 200 mL

⁽³⁾ iCAP6500 - 50 mg/L Fe and K



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TABLE 3. Post Digestion Spike (PDS) Spike Levels and Spiking Solution Concentration			
Analyte	Symbol	PDS spike Level ⁽¹⁾ µg/L	PDS Spike Solution Concentration, mg/L
Aluminum	Al	6000	300
Antimony	Sb	400	20
Arsenic	As	800	40
Barium	Ba	200	10
Beryllium	Be	100	5
Boron	B	400	20
Cadmium	Cd	100	5
Calcium	Ca	3000	150
Chromium	Cr	200	10
Cobalt	Co	100	5
Copper	Cu	200	10
Iron	Fe	3000	150
Lead	Pb	400	20

⁽¹⁾ Spike at 50-times dilution, for example, 0.4 mL PDS spike solution to 20 mL sample, or 0.2 mL to 10 mL sample.



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TABLE 3. (cont) Post Digestion Spike (PDS) Spike Levels and Spiking Solution Concentration			
Analyte	Symbol	PDS spike Level, µg/L	PDS Spike Solution Concentration, mg/L
Magnesium	Mg	10000	500
Manganese	Mn	100	5
Molybdenum	Mo	200	10
Nickel	Ni	200	10
Potassium	K	10000	500
Selenium	Se	300	15
Silver	Ag	200	10
Sodium	Na	10000	500
Strontium	Sr	100	5
Thallium	Tl	400	20
Tin	Sn	400	20
Vanadium	V	100	5
Zinc	Zn	800	40

⁽¹⁾ Spike at 50-times dilution, for example, 0.4 mL PDS spike solution to 20 mL sample, or 0.2 mL to 10 mL sample.



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APPENDIX B

Thermo Electron iCAP6500 Daily Operation Procedures

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The attached procedure summarizes the ERT/SERAS Inorganic Laboratory daily operation practice for sample analysis with the Thermal Electron iCAP6500 ICP Analyzer system. These procedures are based on the assumption that methods have already been developed and the instrument is in the standby mode. Refer to the Thermo Electron iCAP6500 Analyzer operator's manual for specific details on instrument setup, methods development, operation, and maintenance.

THERMO ELECTRON iCAP6500 ANALYZER DAILY OPERATION PROCEDURES

PURPOSE: To provide a step by step procedure for the proper setup and operation of the Thermo Electron iCAP6500 Analyzer instrument.

REFERENCE: Thermo Electron Corporation. 2005. ICAP 6000 Series ICP-OES Spectrometer Operator Manual.
Thermo Electron Corporation. 2009. ICAP 6000 Series ICP-OES Spectrometer User Guide.

Assuming that methods have already been developed, the operator is familiar with the Thermo Electron iTEVA software, and the instrument is on standby mode, the normal daily operation of the iCAP6500 is as follows.

STARTUP

1. Check that Argon pressure is 120 psig at the tank before starting plasma
2. Start computer and log onto Windows
3. Start the water chiller
4. Start iTEVA software, wait for initialization to complete (about one minute)
5. Connect tubing at pump; DO NOT apply platen pressure
6. Connect manual sample line and place it in 10% HNO₃; place the IS line in Internal Std solution
7. Start plasma (from the Plasma Control Panel) and note the x- and y-values for spectrometer optimization at plasma startup. After the pump starts, apply and adjust platen pressure on tubing. Check argon pressure: tank should be 110 psig; line at unit should be 100 psig

ANALYSIS SETUP

1. Load the Analyst program from iTEVA control center; load ICPMETALS method (note the rev number)
2. Set plasma to method conditions; check/adjust platen pressure on tubing as necessary

1. Set appropriate limit and QC check tables in the Method: Method tab, sequence automation and report preferences (unknowns, blanks) sections. Ensure that the correct operations in the correct order are included in the Initial



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Actions, Continuing Actions, and End Actions of the sequence (sequence automation) as shown below:

a. Initial Actions

<u>Operation</u>	<u>Failure Actions</u>
Calibration	None
ICVCCV	Halt
LLICVCCV-W	Halt
ICBCCB-W	Halt
INTA1	Halt
INTA1B3	Halt
ICVCCV	Halt
ICBCCB-W	Halt

b. Continuing Actions

<u>Operation</u>	<u>Failure Actions</u>	<u>Frequency</u>
ICVCCV	None	10
LLICVCCV-W (optional)	None	10
ICBCCB-W	None	10

c. End actions

<u>Operation</u>	<u>Failure Actions</u>
EndCCV2	None
LLICVCCV-W	None
ICBCCB-W	None

Note: Select the appropriate QC table for ICBCCB and LLICVCCV standards depending on the sample matrix: ICBCCB-W/LLICVCCV-W for water, ICBCCB-S/LLICVCCV-S for soil (Hot Block digestion), and ICBCCB-S3051/LLICVCCV3051 (microwave method 3051 digestion).

d. Check the Sequence Report in the Method to ensure that all operations are specified correctly prior to creating the sequence for the samples that you plan to run. Print the Sequence Report (OPTIONAL).

e. Ensure that the appropriate limit check tables for unknowns and blanks are specified in the report preferences section of the method (they are matrix dependent).

- Set results export file name in the Method, automated output section; example: results021910.txt for samples run on 19-Feb-2010
- Save the method (note the rev number)
- Go to the Sequence tab and create a new sequence for the samples that you plan to run (including anticipated dilutions); initialize/connect the autosampler



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7. Fill the 250-mL Nalgene bottle with 10% HNO₃ and place at location #108 (it is marked on the autosampler)
8. Rinse manual sample wand with DI H₂O, air dry, and then disconnect it.
9. Attach the autosampler (AS) sample line and move the AS to position #108 (see 5 above)
10. Edit sample and limit check table names in the AS table. Print the sample name table and the autosampler view graphic; note positions for standards, QC samples, and samples on the autosampler view printout
11. Save the sequence

SAMPLE ANALYSIS

1. Allow one-hour warm-up after starting the plasma before analyzing samples
2. While the unit warms up, fill and load standards and QC samples (in the stds/QC rack per item 10 of Analysis Setup above), prepare samples as necessary (e.g., filter), and load samples (per item 10 of Analysis Setup above)
3. After one-hour warm-up, start the autosession. Record the Spectrometer Optimization x- and y-values. NOTE: if you are running a manual analysis, perform a Spectrometer Optimization before calibration/analysis. From the Analysis tab, select Tools, Optimize Spectrometer; record x- and y-values.
4. The unit should run unattended; however, it is a good idea to periodically check to ensure that there are no problems. You will need to review results to determine if dilutions are necessary; record sample results as necessary.
5. Prepare dilutions as necessary and load them into appropriate spaces in the sample tray
6. After the autosession run ends, perform the shutdown procedure below

REPORTS AND SHUTDOWN

1. After the autosession ends, rinse tubing in 10% HNO₃
2. Export the method to the appropriate folder (e.g., iCAP6500_data\2010\exported_methods\)
3. Print the common report and the calibration report (Method tab, reports section)
4. Create a folder for data files (e.g., iCAP6500_data\2010\02192010\ for data files from 19-Feb-2010). Export ALL files created in Publisher to this folder.
5. From the control panel, start Publisher



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- a. Create a run log report and export it to a pdf file (runlogmmddy.pdf), for example, runlog021910.pdf for the run log for 19-Feb-2020. In the tools tab, click "reuse sample list"
- b. Close the run log report and create a simple sample report (SSR); export to the following files:
 - pdf - SSRmmddy.pdf
 - csv - SSRmmddy.csv
 - ttx - SSRmmddy.ttx
 - xls (Excel, data only) - SSRmmddy.xls
- c. Close the simple sample report and create a vertical table report (VTR); export to the following files:
 - xls (Excel, data only) - VTRmmddy.xls
 - ttx - VTRmmddy.ttx
6. Close publisher; minimize control panel and analyst.
7. Go to the "Program files\Thermo\iTEVA\export" folder. Copy the results export file (see B4 above) to same folder used for Publisher files.
8. Rinse tubing in DI water
9. Air dry the tubing: air dry the IS line first (takes longest time) then the sample line. Move the autosampler to the HOME position (NOT the Rinse position) to dry the sample line.
10. Open the Plasma Control Panel and shut the plasma off. BE SURE that all liquid is gone from the tubing before shutting the plasma off.
11. Remove platen pressure and disconnect tubing.
12. Shut chiller off (about one minute after shutting plasma off).
13. Back up all files to the USB backup drive.

TROUBLESHOOTING

(See Thermo Electron ICAP 6000 Series ICP-OES Spectrometer Operator Manual)

1. ICP will not start (see "STARTUP" above)
 - a. Check: power supplies are ON, Argon gas is ON and at correct pressure, cooling water circulator is ON, exhaust is ON, sample and IS tubing are connected, correct platten pressure on pump winding tubing, and no air leaks in Argon gas lines.



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- b. After making any necessary adjustments, try again.
 - c. If fails to start, power down, power up, recheck all settings, and try again.
 - d. If repeated attempts fail to start the ICP, call Thermo Electron Customer Support for assistance.
2. High RSD During Standardization (IS RSD > 5% and/or RSD for STD2, STD3 elements > 5%)
- a. Repeat the measurement.
 - b. Adjust platten pressure on the IS and Sample pump winding tubing and repeat.
 - c. Check and replace if necessary: IS mix kit tubing, sample line capillary tubing, sample uptake wand connector tubing, IS wand connector tubing, sample wand, and/or IS uptake wand.
 - d. Re-standardize as needed or based on professional judgment.
3. Initial QC Samples Fail
- a. Repeat the measurement.
 - b. Check for high IS RSD and high RSD for results.
 - c. Check for contamination.
 - d. Check another QC sample.
 - e. If repeated attempts fail, re-standardize the instrument and repeat measurements.
4. Sample Analysis Problems
- a. High RSD for IS (>5%), see above.
 - b. IS counts, CCV, and/or CCB results pass (but indicate drift based on professional judgment).
 - check for blockage in the IS and/or sample wand or tubing; replace as needed and re-standardize.
 - check spectrometer optimization and compare the x- and y-values to those just before calibration, reanalyze CCV and CCB. If they pass continue with analysis, otherwise re-standardize.
 - c. CCV Fails



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- repeat the measurement.
 - If the repeat fails, check for high IS RSD, high RSD for elements, IS drift, correct problems as necessary and re-standardize/re-analyze previous 10 samples.
 - If the repeat passes, run a third measurement.
 - If the third measurement passes, continue with CCB measurement
 - If it fails, check for high IS RSD, high RSD for elements, IS drift, correct problems as necessary and re-standardize/re-analyze previous 10 samples
- d. CCB Fails
- Check for carryover; if so, wait a minimum of 5 minutes.
 - repeat the measurement.
 - If the repeat fails, check for high IS RSD, IS drift; correct problems as necessary and re-standardize/re-analyze previous 10 samples.
 - If the repeat passes, run a third measurement.
 - If the third measurement passes, continue with analysis.
 - If it fails, check for high IS RSD, IS drift; correct problems as necessary and re-standardize/re-analyze previous 10 samples.
- e. RL Standard Fails
- Initial RL standard fails after calibration; see step 3 above
 - RL standard fails after 10 samples
 - check for high IS RSD, check for contamination
 - if high IS RSD, repeat the measurement
 - Check analyte concentrations in previous 10 samples
 - If all sample analyte concentrations greater than 10-times that in the RL standard, continue analysis
 - Flag samples that contain analytes with concentrations less than or equal to 10-times the RL value; these samples may be re-analyzed for the affected analytes at a later time; continue



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analysis

- f. Re-standardize as necessary based on professional judgment.



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APPENDIX C
Attachments
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ATTACHMENT 1

iCAP6500 Water and Soil Intermediate LLICV/LLCCV Preparation

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TABLE 1-1. Intermediate LLICV/LLCCV Standard Preparation for Water

iCAP6500 LLICV/LLCCV Standard Preparation Log - Water

Name: iCAP6500 Water LLICV/LLCCV Spike solution *

Final Vol(mL) 100

Number	Starting Materials			Final Standard	
	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add
1	10000	Al		85	0.85
2	1000	Sb		10	1
3	1000	As		19	1.9
4	1000	Ba		3	0.3
5	1000	Be		2	0.2
6	1000	B		10	1
7	1000	Cd		2	0.2
8	10000	Ca		60	0.6
9	1000	Cr		3	0.3
10	1000	Co		2	0.2
11	1000	Cu		8	0.8
12	10000	Fe		35	0.35
13	1000	Pb		10	1
14	10000	Mg		200	2
15	1000	Mn		2	0.2
16	1000	Mo		5	0.5
17	1000	Ni		3	0.3
18	10000	K		250	2.5
19	1000	Se		15	1.5
20	1000	Ag		8	0.8
21	10000	Na		200	2
22	1000	Sr		2	0.2
23	1000	Tl		17	1.7
24	1000	Sn		10	1
25	1000	V		2	0.2
26	1000	Zn		20	2

* RLW6500 - iCAP6500 Water LLICV/LLCCV standard (0.5 mL spike solution to 500 mL final volume)

TOTAL 23.6



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TABLE 1-2. Intermediate LLICV/LLCCV Standard Preparation for Soil (Microwave)

iCAP6500 LLICV/LLCCV Standard Preparation Log - Soil (Microwave 3051A dig)

Name: iCAP6500 Soil (3051) Reporting Level (RL) Spike solution *

Final Vol(mL) 100

Number	Starting Materials			Final Standard	
	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add
1	10000	Al		85	0.85
2	1000	Sb		10	1
3	1000	As		10	1
4	1000	Ba		3	0.3
5	1000	Be		2	0.2
6	1000	B		10	1
7	1000	Cd		2	0.2
8	10000	Ca		60	0.6
9	1000	Cr		4	0.4
10	1000	Co		2	0.2
11	1000	Cu		5	0.5
12	10000	Fe		300	3
13	1000	Pb		10	1
14	10000	Mg		200	2
15	1000	Mn		2	0.2
16	1000	Mo		5	0.5
17	1000	Ni		3	0.3
18	10000	K		500	5
19	1000	Se		15	1.5
20	1000	Ag		5	0.5
21	10000	Na		300	3
22	1000	Sr		2	0.2
23	1000	Ti		17	1.7
24	1000	Sn		20	2
25	1000	V		2	0.2
26	1000	Zn		21	2.1

* RLS3051 - iCAP6500 Soil (3051) LLICV/LLCCV standard (0.5 mL spike solution to 500 mL final volume)

TOTAL 25.15



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ATTACHMENT 3

Spiking Solutions

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SERAS INORGANIC LABORATORY

SPIKING SOLUTION LOG

SOLUTION	CONCENTRATION mg/L or ug/L	PARAMETERS
Spiking Solution #1 for LCS & MS/MSD For Water & Soil/Sed samples ANALYSIS BY ICP (2nd Source)		
	20 ppm	Ag, Be, Cd, Cr, Cu, Co, Mn, Ni, Se, Tl
	20 ppm	Zn, Ba, V, As, Sb, Pb, Mo, B, Sn, Sr
	400 ppm	Al, Ca, Fe, Mg, K, Na
Spiking Solution #2 for LCS/BS/BSD/MS/MSD For Wipe / Air / Tissue samples (2nd Source)		
	300 ppm	Al, Ca, Fe, Mg, K, Na
	10 ppm	Ag, Be, Cd, Cr, Cu, Co, Mn, Ni, Se, Tl
	10 ppm	Zn, Ba, V, As, Sb, Pb, Mo, B, Sn, Sr



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ATTACHMENT 4
iCAP6500 Element Information
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iCAP6500 Element Information

August 2010

ELEMENT	PARAMETER	Wavelength (nm) {order}	Plasma View	Internal Standard
Aluminum	Al3082	308.215 {109}	radial	Y_3710
Aluminum *	Al3961	396.152 {85}	radial	Y_3710
Antimony	Sb2068	206.833 {463}	axial	Y_2243
Arsenic	As1890	189.042 {478}	axial	Y_2243
Barium	Ba4554	455.403 {74}	radial	Y_3710
Beryllium	Be3131	313.107 {108}	radial	Y_3710
Boron	B_2089	208.959 {461}	axial	Y_2243
Cadmium	Cd2144	214.438 {457}	axial	In2306
Calcium	Ca3158	315.887 {107}	radial	Y_3710
Chromium	Cr2677	267.716 {126}	axial	Y_3600
Cobalt	Co2286	228.616 {447}	axial	In2306
Copper	Cu3247	324.754 {104}	axial	Y_3600
Iron **	Fe2599	259.94 {130}	radial	Y_3710
Iron **	Fe2714	271.441 {124}	radial	Y_3710
Lead	Pb2203	220.353 {453}	axial	In2306
Magnesium	Mg2790	279.079 {121}	radial	Y_3710
Manganese **	Mn2576	257.610 {131}	axial	Y_2243
Manganese **	Mn2576-2	257.610 {131}	radial	Y_3710
Molybdenum	Mo2020	202.030 {467}	axial	Y_2243
Nickel	Ni2316	231.604 {445}	axial	In2306
Potassium	K_7664	766.491 {44}	radial	Y_3710
Selenium	Se1960	196.090 {472}	axial	Y_2243
Silver	Ag3280	328.289 {103}	axial	Y_3600
Sodium	Na5895	589.592 {57}	radial	Y_3710
Strontium	Sr4215	421.552 {80}	radial	Y_3710
Thallium	Tl1908	190.864 {477}	axial	In2306
Tin	Sn1899	189.989 {477}	axial	In2306
Vanadium	V_2908	290.882 {116}	axial	Y_3600
Vanadium	V_2908-2	290.882 {116}	radial	Y_3710
Vanadium *	V_2924	292.402 {115}	axial	Y_3600
Vanadium	V_2924-2	292.402 {115}	radial	Y_3710
Zinc	Zn2062	206.200 {463}	axial	In2306
Zinc *	Zn2062R	206.200 {163}	radial	Y_2243R

* Indicates the line normally used for reporting results.

** The choice of iron manganese lines to use depends on the concentration in the samples.



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APPENDIX D

Procedure for Determination of Total Percent (%) Solids

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PROCEDURE FOR DETERMINATION OF TOTAL PERCENT (%) SOLIDS

1.0 APPLICABILITY

This procedure applies to soil, sediment, and other solid samples received for laboratory analysis of total percent solid at the Scientific, Engineering, Response and Analytical Services (SERAS) Laboratory.

2.0 PURPOSE

A step by step procedure is outlined for the determination of total percent solids in soil, sediment and other solid samples.

3.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, Ed. by L. S. Clesceri, A. E. Greenberg, and A. D. Eaton, APHA, AWWA, WEF, Washington, D.C., latest online edition, Methods 2540 B and 2540 G. (With modification-instead of repeating drying, cooling and weighing cycle; overnight drying followed by cooling is allowed to achieve constant weight).

4.0 EQUIPMENT:

-Laboratory oven with temperature control capable of maintaining 103 - 105°C

-Desiccator with a desiccant containing a color indicator for moisture content.

-Aluminum weighing dishes for weighing/drying samples.

-Spatulas or equivalent for transferring samples to the aluminum weighing dishes.

-Analytical balance, capable of weighing samples to 0.01 grams (g), calibrated annually by an outside vendor

Note: The drying oven should be monitored so that proper temperature is maintained during the drying process and care should be taken to avoid excessively high temperatures. The oven must be well ventilated to prevent potentially harmful vapors from escaping to the surrounding air.

5.0 PROCEDURE

The analyst should determine if there is sufficient mass for % solids determination on a sample to sample basis.

1. Sample Preparation

Samples should be homogenized (as much as possible) and at room temperature prior to % Solids determination.

2. Balance Preparation



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Check the balance using class S weights at 0.1, 1.0, and 10.0 g prior to weighing samples.

3 Total % Solids Determination

- Weigh a labeled, empty aluminum weighing dish on the balance; record weight (to the nearest 0.01 g).

- Transfer 10-25 g sample to the dish and record weight (to the nearest 0.01 g) for wet sample plus dish.

- Place the sample dish in the drying oven for a minimum of eight hours at 103 - 105°C.

- Record the initial and final oven temperatures, dates and time in the percent solids log book.

- Cool the samples in a desiccator (to room temperature)

- Record the weight of the dry sample plus dish (to the nearest 0.01g).

- Calculate % solids as described below.

Note: The analyst should adjust the amount of sample used and/or drying time based on the condition of the sample; very wet materials will require larger sample amounts and drying times to get accurate % solids results.

6.0 CALCULATIONS FOR PERCENT SOLIDS

The result for total percent solids is calculated as follows:

$$\%S = [(C - A) / (B - A)] * 100$$

Where:

%S = Percent Solids

A = weight of empty dish (g)

B = weight of wet sample plus dish (g)

C = weight of dry sample plus dish (g)

Record the value in the percent solids log (to the appropriate number of significant figures).

NOTE: Alternatively, the calculation may be performed using a verified spreadsheet on a computer and/or using a Laboratory Information Management System (LIMS).



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APPENDIX E

iCAP6500 Water LLQC Standard Preparation

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iCAP6500 LLQC Standard Preparation Log - Water Matrix

Name: iCAP6500 LLQC1 Intermediate solution (water)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		0.4	0.4	0.4	400
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		6.6	0.66	7	7000
3	100	Sr		0.4	0.4	0.4	400

LLQC1 - 1 mL intermediate to 100 mL final volume

TOTAL 1.46



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iCAP6500 LLQC Standard Preparation Log - Water Matrix

Name: iCAP6500 LLQC2 Intermediate solution (water)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		1	1	1	1000
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		14	1.4	15	15000
3	1000	B		1	0.1	1	1000
4	1000	Sn		1	0.1	1	1000

LLQC2 - 1 mL intermediate to 100 mL final volume

TOTAL 2.6



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iCAP6500 LLQC Standard Preparation Log - Water Matrix

Name: iCAP6500 LLQC3 Intermediate solution (water)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		2	2	2	2000
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		28	2.8	30	30000
3	1000	B		2	0.2	2	2000
4	1000	Sn		2	0.2	2	2000

LLQC3 - 1 mL intermediate to 100 mL final volume

TOTAL 5.2



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iCAP6500 LLQC Standard Preparation Log - Water Matrix

Name: iCAP6500 LLQC4 Intermediate solution (water)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		3	3	3	3000
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		47	4.7	50	50000

LLQC4 - 1 mL intermediate to 100 mL final volume

TOTAL 7.7



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iCAP6500 LLQC Standard Preparation Log

Name: 1000 ppm intermediate

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add
1	5000	VHG - Al, Ca, Fe, Mg, K, Na		1000	20 0 0

TOTAL 20



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iCAP6500 LLQC Standard Preparation Log

Name: 100 ppm Sr intermediate

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add
1	1000	Sr		100	10

TOTAL 10



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PARAMETER	LOQ (µg/L)	2 x LOQ (µg/L)	LOQ Validation Range (µg/L)	iCAP6500 LLQC Standards (µg/L) - Water Matrix			
				LLQC1	LLQC2	LLQC3	LLQC4
Aluminum3082	150	300	150 - 300			300	
Aluminum3961	85	170	85 - 170		150		
Antimony	10	20	10 - 20			20	
Arsenic	19	38	19 - 38				30
Barium	3	6	3 - 6	4			
Beryllium	2	4	2 - 4	4			
Boron	10	20	10 - 20			20	
Cadmium	2	4	2 - 4	4			
Calcium	60	120	60 - 120	70			
Chromium	3	6	3 - 6	4			
Cobalt	2	4	2 - 4	4			
Copper	8	16	8 - 16		10		
Iron2599	35	70	35 - 70	70			
Iron2714	300	600	300 - 600				500
Lead	10	20	10 - 20			20	
Magnesium	200	400	200 - 400			300	
Manganese2576	2	4	2 - 4	4			
Manganese2576-2	2	4	2 - 4	4			
Molybdenum	5	10	5 - 10		10		
Nickel	2	4	2 - 4	4			
Potassium	250	500	250 - 500			300	
Selenium	14	28	14 - 28			20	
Silver	8	16	8 - 16		10		
Sodium	200	400	200 - 400			300	
Strontium	2	4	2 - 4	4			
Thallium	17	34	17 - 34				30
Tin	10	20	10 - 20			20	
Vanadium2908	6	12	6 - 12		10		
Vanadium2908-2	20	40	20 - 40				30
Vanadium2924	2	4	2 - 4	4			
Vanadium2924-2	20	40	20 - 40				30
Zinc2062	3	6	3 - 6	4			
Zinc2062R	20	40	20 - 40				30

LOQ - Limit of Quantitation
LLQC - LOQ Validation Standard



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APPENDIX F

iCAP6500 Soil (Microwave 3051A Digestion) LLQC Standard Preparation

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iCAP6500 LLQC Standard Preparation Log - Soil (3051A dig) Matrix

Name: iCAP6500 LLQC1 Intermediate spike solution (soil 3051)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		0.4	0.4	0.4	400
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		7.6	0.76	8	8000
3	100	Sr		0.4	0.4	0.4	400

LLQC1 - 0.50 mL intermediate to 50 mL final volume

TOTAL 1.56



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iCAP6500 LLQC Standard Preparation Log - Soil (3051A dig) Matrix

Name: iCAP6500 LLQC2 Intermediate spike solution (soil 3051)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		1	1	1	1000
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		11	1.1	12	12000
3	1000	B		1	0.1	1	1000
4	1000	Sn		1	0.1	1	1000

LLQC2 - 0.50 mL intermediate to 50 mL final volume

TOTAL 2.3



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iCAP6500 LLQC Standard Preparation Log - Soil (3051A dig) Matrix

Name: iCAP6500 LLQC3 Intermediate spike solution (soil 3051)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		2	2	2	2000
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		28	2.8	30	30000
3	1000	B		2	0.2	2	2000
4	1000	Sn		4	0.4	4	4000

LLQC3 - 0.50 mL intermediate to 50 mL final volume

TOTAL 5.4



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iCAP6500 LLQC Standard Preparation Log - Soil (3051A dig) Matrix

Name: iCAP6500 LLQC4 Intermediate spike solution (soil 3051)

Final Vol(mL) 100

Starting Materials				Final Standard		TOTAL	
Number	Conc. (mg/L)	Elements	SERAS Std. No.	Desired Conc. (mg/L)	mL to add	mg/L	ug/L
1	100	VHG SM23		3.4	3.4	3.4	3400
2	1000	VHG - Al, Ca, Fe, Mg, K, Na		56.6	5.66	60	60000

LLQC4 - 0.50 mL intermediate to 50 mL final volume

TOTAL 9.06



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iCAP6500 LLQC Standard Preparation Log

Name: 1000 ppm intermediate

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	seras Std. No.	Desired Conc. (mg/L)	mL to add
1	5000	VHG - Al, Ca, Fe, Mg, K, Na		1000	20

TOTAL 20



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iCAP6500 LLQC Standard Preparation Log

Name: 100 ppm Sr intermediate

Final Vol(mL) 100

Starting Materials				Final Standard	
Number	Conc. (mg/L)	Elements	REAC Std. No.	Desired Conc. (mg/L)	mL to add
1	1000	Sr		100	10

TOTAL 10



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PARAMETER	LOQ (µg/L)	2 x LOQ (µg/L)	LOQ Validation Range (µg/L)	iCAP6500 LLQC Standards (µg/L) - Soil (microwave 3051A dig) Matrix			
				LLQC1	LLQC2	LLQC3	LLQC4
Aluminum3082	170	340	170 - 340			300	
Aluminum3961	85	170	85 - 170		120		
Antimony	10	20	10 - 20			20	
Arsenic	10	20	10 - 20			20	
Barium	3	6	3 - 6	4			
Beryllium	2	4	2 - 4	4			
Boron	10	20	10 - 20			20	
Cadmium	2	4	2 - 4	4			
Calcium	60	120	60 - 120		120		
Chromium	4	8	4 - 8	4			
Cobalt	2	4	2 - 4	4			
Copper	5	10	5 - 10		10		
Iron2599	77	154	77 - 154		120		
Iron2714	300	600	300 - 600				600
Lead	10	20	10 - 20			20	
Magnesium	200	400	200 - 400			300	
Manganese2576	2	4	2 - 4	4			
Manganese2576-2	3	6	3 - 6	4			
Molybdenum	5	10	5 - 10		10		
Nickel	3	6	3 - 6	4			
Potassium	500	1000	500 - 1000				600
Selenium	15	30	15 - 30			20	
Silver	5	10	5 - 10		10		
Sodium	300	600	300 - 600				600
Strontium	2	4	2 - 4	4			
Thallium	17	34	17 - 34				34
Tin	20	40	20 - 40			40	
Vanadium2908	6	12	6 - 12		10		
Vanadium2908-2	34	68	34 - 68				34
Vanadium2924	2	4	2 - 4	4			
Vanadium2924-2	20	40	20 - 40				34
Zinc2062	9	18	9 - 18		10		
Zinc2062R	21	42	21 - 42				34

LOQ - Limit of Quantitation
 LLQC - LOQ Validation Standard