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STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

SOP No.: MET-ICP

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Approved by: _____ Supervisor — 9/28/11 Date

_____ QA Manager — 9/29/11 Date

_____ Laboratory Manager — 9/29/11 Date

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DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

1 SCOPE AND APPLICATION

1.1 This procedure describes the steps taken for the analysis of soil, sludge surface water and drinking water digestates using EPA methods 6010C, 200.7, and CLP ILM04.0 for a variety of elements. The digested samples and QC standards are all diluted in a similar acid matrix. A procedure is also given for calculation of hardness by Standard Methods 2340B.

1.2 The Method Reporting Limits (MRLs) for common elements are listed in Table 1. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL). Therefore, MRL=EQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. The Method Detection Limits (MDLs) that have been achieved are listed in Table 1. The MDL and MRL may change as annual studies are performed.

1.3 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP or project which require older versions of EPA methods (i.e. 6010B). QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)* may supersede the requirements defined in this SOP.

2 METHOD SUMMARY

2.1 A representative aliquot of sample is prepared as described in the applicable digestion SOP. The digestate is analyzed for the elements of interest using ICP spectrometry. The instrument measures characteristic emission spectra by optical spectrometry. The intensity of emission lines are monitored.

2.2 Final results are calculated using the digestion information and the results from the ICP analysis. Data is reported using standard CAS procedures and formats, or following project specific reporting specifications.

2.3 Deviations from the reference method(s): This SOP contains no deviations from the reference methods.

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3 DEFINITIONS

3.1 **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample digestates interspersed with calibration standards.

3.2 **Independent Calibration Verification** (ICV) - ICV solutions are made from stock solutions different from the stock used to prepare calibration standards and are used to verify the validity of the standardization.

3.3 **Laboratory Control Sample** (LCS): A laboratory blank that has been fortified with target analyte and used to determine that the analysis is in control. For solids, a reference material may be used unless prohibited by project protocols.

3.4 **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

3.5 **Matrix Spike Duplicate** (MSD) - In the matrix spike duplicate analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike duplicate is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the matrix spikes is calculated and used to assess analytical precision.

3.6 **Duplicate Sample** (DUP) - A laboratory duplicate is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.7 **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.8 **Continuing Calibration Verification Standard (CCV)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.9 **Instrument Blank (CCB)** - The instrument blank (also called continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

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3.10 **Laboratory fortified Blank (LFB)**- A laboratory blank that has been fortified with target analyte at the method reporting limit and used to determine if the laboratory can detect contaminants at the method reporting limit.

4 INTERFERENCES

4.1 Interferences from contaminated reagents must be eliminated. The purity of acids must be established by the laboratory as being high enough to eliminate the introduction of contamination above the MRL (or above ½ the RL for DoD work).

4.2 Background emission and stray light can be compensated by background correction.

4.3 Spectral overlaps resulting in interelement contributions can be corrected for by using interelement correction factors. Interelement correction factors are established for each instrument and are maintained by the analyst at the workstation.

5 SAFETY

5.1 Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.2 Hydrochloric, Nitric and Hydrofluoric Acids are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. Safety glasses, lab coat and gloves should be worn while working with the solutions.

5.3 High Voltage - The power unit supplies high voltage to the RF generator which is used to form the plasma. The unit should never be opened. Exposure to high voltage can cause injury or death.

5.4 UV Light -The plasma when lit is a very intense light, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available.

6 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Samples are prepared using methods 3005A, 3010A, 3050, or CLPILM04.0 (CAS SOPs MET-3005A, MET-3010A, MET-3050, and MET-DIG). Samples are received in the ICP lab as completed digestates. Samples are stored in 50 mL plastic centrifuge tubes, 100 mL digestion vessels or in 100 mL volumetric flasks.

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6.2 Water samples analyzed by EPA method 200.7 are preserved after arrival at the laboratory. These samples are held for a minimum of 16 hours and the pH verified to be <2 prior to digestion.

6.3 Soil samples are diluted prior to instrumental analysis by a factor of 2. This allows the method to meet the required 1 g of sample to 200 mL dilution during digestion.

6.4 Following analysis, digestates are stored until two weeks after all results have been reviewed and then brought to 3< pH<10 and disposed of through the sewer system.

7 APPARATUS & EQUIPMENT

7.1 Inductively Coupled Plasma Atomic Emission Spectrometer

7.1.1 Thermo Jarrell Ash IRIS.
7.1.2 Thermo Scientific ICAP 6500

7.2 Concentric nebulizers.

7.3 Microflow nebulizer for ICAP 6500.

7.4 Torches and injector tips for each ICP.

7.5 Cyclonic spray chambers for each instrument.

7.6 Water coolers for each ICP (internal on the IRIS.)

7.7 Argon Humidifiers for the IRIS and ICAP 6500.

7.8 ESI SC4 DX Autosampler with Fast System for ICAP 6500.

7.9 Peristaltic Pumps for each Spectrometer.

7.10 ASX-520 autosamplers for the IRIS.

7.11 RF Generators for each ICP (internal on the IRIS and ICAP 6500).

7.12 Computer system interfaced to each ICP. A compatible Windows-based data system is used to acquire, store, and perform calculations on raw data.

8 STANDARDS, REAGENTS, & CONSUMABLE MATERIALS

8.1 Standards Preparation

8.1.1 Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP

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Reagent/Standards Login and Tracking (ADM-RTL) for the complete procedure and documentation requirements. Manufacturer's expiration dates are used to determine the viability of standards.

8.1.2 Calibration Standards

Calibration standards are prepared from commercially purchased single element 1000 ppm or 10,000 ppm stock standards as well as pre-mixed multi element stock standards. All standards are aliquoted using Class A volumetric pipettes, or calibrated fixed and adjustable volume autopipetters. All dilutions are made in Class A volumetric glassware.

The standard mixes for each ICP system vary based on the requirements of each instrument. The composition of the IRIS calibration standards are outlined in Table 3. The composition of the ICAP 6500 standards are outlined in Table 4.

8.1.3 Continuing Calibration Verification (CCV) Standards

CCV standards are analyzed at the midpoint of the calibration. These standards are produced by making a two-fold dilution of each calibration standard. The CCV standards are then run in sequence during the analytical run.

8.1.4 Initial Calibration Verification (ICV) Standards

The ICV working standards are produced by direct dilution of three certified mixed stock solutions (QCP-CICV1, QCP-CICV2, and QCP-CICV3) purchased from Inorganic Ventures or another qualified vendor and various single element stock solutions from sources different than the calibration standards. The composition of these standards is outlined in Table 5.

8.1.5 Interference Check Solutions (ICSA & ICSAB)

The ICSA and ICSAB working standards are produced by direct dilution of certified mixed stock solutions (CLPP-ICS-A and CLPP-ICS-B or equivalent.) Antimony is also added to the ICSAB solution from a 1000 ppm single element stock standard. The composition of these standards is outlined in Table 6.

8.1.6 CRI/Low Level Calibration Verification

The CRI, Low Level Initial Calibration Verification (LLICV), and Low Level Continuing Calibration Verification (LLCCV) are produced by diluting 1000 or 10000ppm single stock standards into a 100X intermediate standard and then diluted 1/100 to obtain the MRL level. Note: The level used is that of the normal MRL used for both instruments.

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8.1.7 The solutions and materials used for the LCS and matrix spikes are described in the applicable digestion SOP.

8.1.8 Standard Log

The analyte, source, initial volume, final volume, final concentration and expiration date are recorded in a standard logbook kept in the ICP lab. The operator who prepares the standard must date and initial the entry in the standards logbook. The operator also places his initials and the date prepared on the standard container. In addition to working standards used in calibration, all other standards used in the analytical run such as ICVs, MRL standards, and other project or client specific standards shall be documented in the standard logbook.

8.2 High Purity Argon.

8.3 Capillary, rinse and peristaltic pump tubing.

8.4 17 x 100mm polypropylene test tubes.

9 PREVENTIVE MAINTENANCE

9.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

9.2 Torch, nebulizer, and spray chambers are cleaned as required. All instrument filters are vacuumed monthly. Dirty ICP torches and mixing chambers are soaked in aqua regia overnight, rinsed and placed in a clean dry area. The conical nebulizer is back flushed with acid or DI water as needed. The microflow nebulizer is not back flushed. Use the obstruction removal kit with fused silica.

9.3 IRIS-specific procedures

9.3.1 Each time the nebulizer is changed, the instrument optimization procedure is performed. A 10 ppm As standard is aspirated and the intensity counts are maximized by adjusting the depth to which the nebulizer is inserted into the spray chamber.

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with

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the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2 It is the responsibility of the department supervisor/manager to document analyst training. Training and proficiency is documented in accordance with the SOP *ADM-TRANDOC*.

11 PROCEDURE

11.1 Operating Parameters

11.1.1 For the Thermo Jarrell Ash IRIS, the operating parameters are defined in the operating system *Method* file. Default operating parameters are given in file GALILEO. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct *Method* file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the IRIS and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

11.1.2 For the Thermo Scientific ICAP 6500, the operating parameters are defined in the *Method* file. Default operating parameters are given in *Tools/Options/New Method Parameters*. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct *Method* file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the ICAP 6500 and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

11.2 Calibration/Standardization

11.2.1 IRIS

11.2.1.1 Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.2.1.2 An internal standard is used for routine analyses on this instrument. Scandium is used as the internal standard. The internal standard solution is introduced into the analyzed solutions (standards, blanks, QC, samples, etc.) at 1ug/mL.

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11.2.1.3 Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

11.2.1.4 The Cu/Mn ratio is calculated from Standard A. Ratio is to remain within 20% of the value when IECs were determined. This data is recorded in the instrument logbook.

11.2.2 ICAP 6500

11.2.2.1 Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.2.2.2 An internal standard is used for routine analyses on this instrument. Yttrium and Indium are used as internal standards. The internal standard solution is introduced into the analyzed solutions (standards, blanks, QC, samples, etc.) at 0.8 ug/mL for Y, and 1.6 ug/mL for In.

11.2.2.3 Run a peak check standard and adjust peaks as needed.

11.2.2.4 Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

11.2.2.5 The Cu/Mn ratio is calculated from Standard A using Mn2576. Ratio is to remain within 20% of the value when IECs were determined. This data is recorded in the instrument logbook.

11.2.3 Standardization is completed by analyzing an ICV for each analyte to be determined. For method 200.7 the result must be within ± 5% of the true value. For method 6010B/C the result must be within ± 10% of the true value. If the ICV fails when running method 6010C, either the calibration standards or the ICV must be prepared fresh and the instrument re-standardized. If the ICV fails when running methods 200.7 and 6010B only re-standardization is necessary.

11.2.4 Method 6010C also requires a LLICV be analyzed at the MRL level. The result must be within ± 30% of the true value. The LLICV need not be made up with stock standards different than those of the calibration standards. A LLICV is not necessary when running methods 200.7 and 6010B.

11.3 Analytical Run

11.3.1 Following standardization and ICV analysis, the remainder of the run is determined by what analytical method is being performed. These are listed below.

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11.3.1.1 CLP ILM04.0: ICB, CCV, CCB, CRI, ICSA, ICSAB, CCV, CCB, routine samples. The CRI, ICSA, and ICSAB will be analyzed every 20 samples. They will be labeled with an F indicating Final. Each set will be numbered in increasing order, i.e. ICSAF1, ICSAF2.

11.3.1.2 Methods 200.7 and 6010B/C: ICB, CCV, CCB, CRI, ICSA, ICSAB, routine samples.

11.3.2 Evaluate the initial QC using the following criteria:

11.3.2.1 For methods 200.7 and 6010B/C, the following criteria apply:

- The ICB and CCB results are evaluated using method specified requirements. The following guidelines should also be used to determine acceptability:

- For 200.7, the result should be less than 3 times the standard deviation of the mean background signal.

- For method 6010B, the result should be less than the Method Detection Limit (MDL). In cases where the associated sample results are being reported to the Method Reporting Limit (MRL) the result may be greater than the MDL if the result does not adversely impact data quality.

- For method 6010C, the result should be less than the Lower Limit of Quantitation (LOQ).

- Where project specifications allow, the result may be over the MDL if the result does not adversely impact data quality.

- The CCV immediately following standardization must verify within ± 10% of the true values with a relative standard deviation of <5% from 2 replicate integrations for methods 6010B/C. For 200.7, the first CCV must verify within ± 5% with a RSD of <3% from 4 replicates. Calculate %RSD as follows:

$$\%RSD = \frac{StdDev_{CCV}}{Average_{CCV}} x\,100$$

where: $StdDev_{ccv}$ = Standard deviation of the replicate integrations
$Average_{ccv}$ = Average of the replicate CCV integrations

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- The CRI is a low level standard with concentrations at the RL. For DoD projects, the CRI standard concentrations will be equal to the project RLs and results must verify within 20% of the true value. For method 6010C the CRI results should be within 30% of the true value. For 200.7 and 6010B the CRI results should be greater than the MDL and less than 2X the MRL. For method 6010C, the CRI may be run as the LICV/CRI and the results should be within 30% of the true value.

- The ICSA is run to check the validity of the Interelement Correction Factors (IECs).

Note: DoD QSM requires this to be run at the beginning of each analytical run.

- The ICSAB must be within 20% of the expected value for the CLPP-ICS-B elements and Sb.

11.3.2.2 The ICV, LLICV, ICB, CCV, CCB, CRI, and ICSAB must meet the criteria listed. Reanalyze any elements that fail.

11.3.2.3 For CLP, refer to SOW ILM04.0 for acceptance criteria.

11.3.3 Continuing Calibration Verification

11.3.3.1 CCVs are analyzed after every 10 samples and at the end of the analytical run. They must verify within ±10% of the expected value with a RSD of <10%.

11.3.3.2 CCBs are analyzed after every 10 samples and at the end of the analytical run. CCBs are evaluated as in section 11.3.2.1.

11.3.3.3 Method 6010C requires a LLCCV be analyzed at the end of each analysis batch. The LLCCV is at the MRL level and must verify within ± 30% of the true value. Reanalyze any elements to be reported at low levels that are bracketed by the LLCCV if the standard fails.

11.3.4 If the CCV, CCB or ICS solutions fail, reanalyze any elements to be reported.

12 QA/QC REQUIREMENTS

12.1 Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery for each analyte must meet LCS criteria and the RSD< 30%.

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12.2 Method Detection Limits

12.2.1 A Method Detection Limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates at a level near or below the MRL. Follow the procedures in Section 11 to analyze the samples. Refer to the CAS SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL.)*

12.2.2 Calculate the average concentration found (x) and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. MDLs must be performed whenever there is a significant change in the background or instrument response.

12.2.3 A Limit of Detection (LOD) check must be performed after establishing the MDL and at least annually (quarterly if DoD) afterward. A blank is spiked with analytes at 1-4X the MDL and carried through the preparation and analytical procedure. The LOD is verified when the signal/noise ratio is > 3 for all analytes.

12.3 Limit of Quantitation Check(LOQ)/Lower Limit of Quantitation Check(LLQC)

For Method 6010C and drinking waters by method 200.7 a Lower Limit of Quantitation Check (LOQ/LLOQ) sample must be analyzed after establishing the MRL and at least annually (quarterly if DoD) afterward to demonstrate the desired detection capability. The LOQ/LLOQ sample is spiked at 1-2X the MRL and must be carried through the entire preparation and analytical procedure. Limits of quantitation are verified when all analytes are detected within 30% of their true value.

12.4 Linear Dynamic Range

The upper limit of the LDR must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing at least three succeeding higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% above or below the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified quarterly or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

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12.5 Instrument Detection Limit

On a quarterly basis, the instrument detection limits for all analytes are determined as per procedures outlined in ILM04.0 (Section E, paragraph 10, 12 resp.). IDLs are determined using blanks and this data is kept on file.

12.6 Interelement Correction Factors

Semi-annually, instrument interferences are calculated as per ILM04.0 (Section E, paragraph 11) and Method 6010B/C. During the course of routine work, other interferences may be found. They are verified by the operator during the analytical run and data is manually corrected. Copies of this data are kept on file.

12.7 Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for *Sample Batches*. Additional QC Samples may be required in project specific quality assurance plans (QAPP). For example projects managed under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories.* General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD).* General QC Samples are:

12.7.1 Each sample preparation batch must have a method blank associated with it. The method blank result should be < MRL. If the method blank is found to be contaminated, it may be reported if the concentration in the associated samples is at least 20 times the amount found in the method blank for methods 200.7 and 6010B, otherwise redigest the batch. For Method 6010C, the method blank may be reported if the concentration in the associated samples is at least 10 times the amount found in the method blank.A contaminated method blank (MB) may also be reported if all of the associated samples are non-detect (ND).

Note: DoD QSM requires contamination in the MB be <1/2 the RL or < 1/10 any sample amount.

12.7.2 A Laboratory Control Sample (LCS) is digested one per batch, or per 20 samples. For method 200.7, the LCS recovery criteria is 85-115% for water samples. For method 6010B/C, the control limits are derived from lab data and are listed in Table 2. These limits should be no wider than 80-120%. For soil samples, the recovery must fall within the ranges specified for the reference material. For CLP, use the prescribed limits for the SOW in use. In all cases, project-specific QC limits may be required. If the LCS fails the acceptance criteria, redigest the batch of samples. For specifics on the preparation and composition of LCS samples refer to the appropriate digestion SOP.

12.7.3 A Duplicate sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analyses. The default

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criteria may be used if statistically generated criteria are broader or insufficient points are available for accurate statistical limits. Currently, statistically generated criteria are broader and the default is used for all elements but Manganese, for which the limit is 17% RPD. The RPD criteria are <30% for soil samples and <20% for water samples for methods 200.7 and 6010B. The RPD criteria is <20% for both soils and waters for method 6010C. Criteria are subject to change as statistical data are generated. If the RPD is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous).

12.7.4 A Laboratory fortified Blank (LFB) at the MRL is digested and analyzed with every batch of drinking water samples (method 200.7). The default acceptance criteria of 50-150% are to be used until sufficient data points are acquired to calculate in-house control limits.

12.7.5 A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analyses. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria for method 200.7 is 70-130% for both water and soil samples. For 6010B, the control limits are derived from lab data and are listed in Table 2. For 6010C, Table 2 is used unless the control limits are broader than the default criteria of 75-125% in which case the default criteria are used. For CLP, use the prescribed limits for the SOW in use. In all cases, project-specific QC limits may be required. If the recovery is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous). If the sample concentration is >4x the spike level, no action is required and data is flagged accordingly. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

12.7.6 Matrix Interference

12.7.6.1 When an analyst suspects that there may be any matrix interferences present, a 1:5 serial dilution test shall be performed. The dilution should be within ± 10% of the original result.

12.7.6.2 A 1:5 serial dilution shall be performed for all Tier III or IV deliverables.

12.7.6.3 If the analyte concentration is too low to permit a dilution test, then a post digestion spike may be performed. The recovery should be ± 20%.

12.7.6.4 For Method 6010C, lower tier batches that exhibit MS failure require a serial dilution test as well as a post digestion spike.

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Note: DoD QSM recovery acceptance limits are 75-125%.

12.8 Additional QC measures include control charting and compiling of QC data for generation of control limits.

12.9 CLP analyses are performed as per the QA/QC guidelines in the most current CLP SOW.

13 DATA REDUCTION, REVIEW, AND REPORTING

13.1 Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result. The wavelengths used to quantify each metal are summarized in Table 7 for the IRIS and Table 8 for the ICAP6500.

Aqueous samples are reported in µg/L:

$$\mu g/L\,(Sample) = C^{*}\ x\ Digestion\ Dilution\ Factor\ x\ Post\ Digestion\ Dilution\ Factor \times 1000\,\mu g/mg$$

Solid samples are reported in mg/Kg:

$$mg/Kg\,(Sample) = C^{*}\ x\ Post\ Digestion\ Dilution\ Factor\ x\ \frac{Digestion\ Vol\,(ml)}{Sample\ wt\,(g)}\ x\ \frac{1L}{1000ml}\ x\ \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in mg/L.

13.2 If total hardness is to be reported, use Calcium and Magnesium results to calculate as follows. For reporting calcium hardness, use only the calcium portion of the equation.

$$Hardness,\ mg\ equivalent\ CaCO_3/L = 2.497\left[Ca, mg/L\right] + 4.118\left[Mg, mg/L\right]$$

13.3 A daily run log of all samples analyzed is maintained. All CLP data should be printed and stored after operator has checked for evenness of burns. A copy of this document will go with each package of Tier III or higher data run that day.

13.4 Data Review and Reporting

13.4.1 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12. The data is then placed in a work order file until complete. When the work order is complete, a report is generated. A final review is performed and the data is delivered to the project management department.

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14 CORRECTIVE ACTION

14.1 Refer to the SOP for *Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2 Handling out-of-control or unacceptable data

14.2.1 On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2 Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when:

- Corrective action is not taken or not possible
- Corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis.
- Reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15 METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

15.1 The method detection limit (MDL) is established using the procedure described in the SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification (ADM-MDL).* Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16 POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible and within method requirements. Standards are prepared in volumes consistent with the laboratory use in order to minimize the volume of expired standards to be disposed. The threat to the environment from reagents used in this method may be minimized when recycled or disposed of properly.

17 WASTE MANAGEMENT

17.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2 This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 3-10 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18 TRAINING

18.1 Refer to the *SOP for Documentation of Training* for standard procedures.

18.2 Training outline

18.2.1 Review literature (see references section). Review the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.2.2 The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of approximately two weeks. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.2.3 Perform initial precision and recovery (IPR) study as described in Section 12.1 for water samples. Summaries of the IPR are reviewed and signed by the supervisor.

18.3 Training and proficiency is documented in accordance with the SOP ADM-TRANDOC.

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19 CHANGES SINCE THE LAST REVISION

19.1 Sec 1.2 removed PQL – outdated definition
19.2 Sec 8.1.1 updated to reflect current lab QA system requirements
19.3 7.2: Added Microflow nebulizer for ICAP 6500.
19.4 7.8: Added ESI SC4 DX autosampler with fast system for ICAP 6500.
19.5 Sec 9.1 updated to reflect current lab QA system requirements
19.6 Sec 9.2 Added maintenance procedure for the Microflow nebulizer.
19.7 11.2.2.2: Updated internal standards: y = 0.8 µg/mL; 1n =1.6 µg/mL
19.8 11.3.2.1: 6010- added use of the CRI for the LLCV/CRI and acceptance criteria.
19.9 12.7.1: Added reporting of a contaminated MB if associated samples are ND.
19.10 12.7.6: Revised section on matrix interference.
19.11 11.3.3.3: Removed requirement to run ICS solutions every 8 hours.
19.12 Table 3 Standard B Iris ICP-OES, Mn aliquot 10 mL; final concentration 10 ppm.
19.13 Table 5 ICVB Solution updates for Li -2ppm; Fe and Mn -10ppm;
19.14 Table 8 update to include alternate frequency wavelengths for Cadmium, Cobalt, Copper Phosphorous, Nickel and Zinc.
19.15 Corporate SOP name change; ADM-DATANTRY *Making Entries into Logbooks and onto Analytical Records.*
19.16 Tables 1 & 2 updated

20 REFERENCES

20.1 USEPA, Contract Laboratory Program, SOW #ILM04.0

20.2 Thermo Jarrell Ash ICAP61 Manual

20.3 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010B, Revision 2, December 1996.

20.4 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010C, Revision 3, February 2007.

20.5 USEPA, Methods for Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111, Method 200.7, Revision 4.4, May 1994.

20.6 *Hardness by Calculation, Method 2340B,* Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

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CAS/KELSO DATA QUALITY OBJECTIVES **Table 1 & 2**

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
200.7	Aluminum	7429-90-5	Soil	6	10	mg/kg	41-158	70-130	30
200.7	Antimony	7440-36-0	Soil	3	10	mg/kg	50-150	70-130	30
200.7	Arsenic	7440-38-2	Soil	4	20	mg/kg	75-125	70-130	30
200.7	Barium	7440-39-3	Soil	0.3	2	mg/kg	81-119	70-130	30
200.7	Beryllium	7440-41-7	Soil	0.03	1	mg/kg	83-117	70-130	30
200.7	Boron	7440-42-8	Soil	0.4	10	mg/kg	67-133	70-130	30
200.7	Cadmium	7440-43-9	Soil	0.2	1	mg/kg	81-119	70-130	30
200.7	Calcium	7440-70-2	Soil	2	10	mg/kg	79-121	70-130	30
200.7	Chromium	7440-47-3	Soil	0.4	2	mg/kg	80-119	70-130	30
200.7	Cobalt	7440-48-4	Soil	0.3	2	mg/kg	82-118	70-130	30
200.7	Copper	7440-50-8	Soil	0.6	2	mg/kg	83-116	70-130	30
200.7	Iron	7439-89-6	Soil	0.7	4	mg/kg	50-149	70-130	30
200.7	Lead	7439-92-1	Soil	3	20	mg/kg	79-121	70-130	30
200.7	Lithium	7439-93-2	Soil	0.5	2	mg/kg	75-125	70-130	30
200.7	Magnesium	7439-95-4	Soil	0.3	4	mg/kg	73-127	70-130	30
200.7	Manganese	7439-96-5	Soil	0.04	2	mg/kg	81-119	70-130	30
200.7	Molybdenum	7439-98-7	Soil	0.5	2	mg/kg	75-125	70-130	30
200.7	Nickel	7440-02-0	Soil	0.5	4	mg/kg	81-118	70-130	30
200.7	Phosphorus	7723-14-0	Soil	3	40	mg/kg	75-125	70-130	30
200.7	Potassium	7440-09-7	Soil	20	80	mg/kg	73-126	70-130	30
200.7	Selenium	7782-49-2	Soil	4	20	mg/kg	75-125	70-130	30
200.7	Silver	7440-22-4	Soil	0.4	2	mg/kg	66-134	70-130	30
200.7	Sodium	7440-23-5	Soil	4	40	mg/kg	74-126	70-130	30
200.7	Strontium	7440-24-6	Soil	0.02	2	mg/kg	79-121	70-130	30
200.7	Thallium	7440-28-0	Soil	3	20	mg/kg	75-125	70-130	30
200.7	Tin	7440-31-5	Soil	2	10	mg/kg	75-124	70-130	30
200.7	Titanium	7440-32-6	Soil	0.5	2	mg/kg	75-125	70-130	30
200.7	Vanadium	7440-62-2	Soil	0.4	2	mg/kg	79-121	70-130	30
200.7	Zinc	7440-66-6	Soil	0.3	2	mg/kg	73-121	70-130	30

a Method Detection Limits are subject to change as new MDL studies are completed.

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CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
200.7	Aluminum	7429-90-5	Water	30	50	28	50	ug/L	85-115	70-130	20
200.7	Antimony	7440-36-0	Water	20	50	30	50	ug/L	85-115	70-130	20
200.7	Arsenic	7440-38-2	Water	20	100	30	100	ug/L	85-115	70-130	20
200.7	Barium	7440-39-3	Water	1	5	1.5	5	ug/L	85-115	70-130	20
200.7	Beryllium	7440-41-7	Water	0.2	5	0.3	5	ug/L	85-115	70-130	20
200.7	Boron	7440-42-8	Water	3	50		50	ug/L	85-115	70-130	20
200.7	Cadmium	7440-43-9	Water	0.8	5	2	5	ug/L	85-115	70-130	20
200.7	Calcium	7440-70-2	Water	10	50	25	50	ug/L	85-115	70-130	20
200.7	Chromium	7440-47-3	Water	3	5	2.5	5	ug/L	85-115	70-130	20
200.7	Cobalt	7440-48-4	Water	2	10	2.5	10	ug/L	85-115	70-130	20
200.7	Copper	7440-50-8	Water	5	10	4.5	10	ug/L	85-115	70-130	20
200.7	Iron	7439-89-6	Water	2	20	4	20	ug/L	85-115	70-130	20
200.7	Lead	7439-92-1	Water	8	50	30	50	ug/L	85-115	70-130	20
200.7	Lithium	7439-93-2	Water	2	10	3.5	10	ug/L	85-115	70-130	20
200.7	Magnesium	7439-95-4	Water	0.7	20	0.5	20	ug/L	85-115	70-130	20
200.7	Manganese	7439-96-5	Water	0.4	5	0.5	5	ug/L	85-115	70-130	20
200.7	Molybdenum	7439-98-7	Water	3	10	5	10	ug/L	85-115	70-130	20
200.7	Nickel	7440-02-0	Water	2	20	4	20	ug/L	85-115	70-130	20
200.7	Phosphorus	7723-14-0	Water	30	200	40	200	ug/L	85-115	70-130	20
200.7	Potassium	7440-09-7	Water	50	400	80	400	ug/L	85-115	70-130	20
200.7	Selenium	7782-49-2	Water	30	100	40	100	ug/L	85-115	70-130	20
200.7	Silicon	7440-21-3	Water	10	400		400	ug/L	85-115	70-130	20
200.7	Silver	7440-22-4	Water	5	10	5	10	ug/L	85-115	70-130	20
200.7	Sodium	7440-23-5	Water	30	200	20	200	ug/L	85-115	70-130	20
200.7	Strontium	7440-24-6	Water	0.06	10			ug/L	85-115	70-130	20
200.7	Thallium	7440-28-0	Water	30	100	60	100	ug/L	85-115	70-130	20
200.7	Tin	7440-31-5	Water	20	50	30	50	ug/L	85-115	70-130	20
200.7	Titanium	7440-32-6	Water	5	10	5	10	ug/L	85-115	70-130	20
200.7	Vanadium	7440-62-2	Water	5	10	5	10	ug/L	85-115	70-130	20
200.7	Zinc	7440-66-6	Water	2	10	2.5	10	ug/L	85-115	70-130	20

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CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
6010C	Aluminum	7429-90-5	Water	40	50	40	50	ug/L	92-112	75-125	20
6010C	Antimony	7440-36-0	Water	10	50	30	50	ug/L	90-113	75-125	20
6010C	Arsenic	7440-38-2	Water	20	100	30	100	ug/L	90-112	75-125	20
6010C	Barium	7440-39-3	Water	0.5	5	1.5	5	ug/L	91-113	75-125	20
6010C	Beryllium	7440-41-7	Water	0.2	5	0.3	5	ug/L	91-113	75-125	20
6010C	Boron	7440-42-8	Water	2	50	4	50	ug/L	91-112	75-125	20
6010C	Cadmium	7440-43-9	Water	0.9	5	2	5	ug/L	93-113	75-125	20
6010C	Calcium	7440-70-2	Water	9	50	25	50	ug/L	85-116	75-125	20
6010C	Chromium	7440-47-3	Water	2	5	5	5	ug/L	93-114	75-125	20
6010C	Cobalt	7440-48-4	Water	2	10	2.5	10	ug/L	93-114	75-125	20
6010C	Copper	7440-50-8	Water	5	10	9	10	ug/L	91-111	75-125	20
6010C	Iron	7439-89-6	Water	3	20	4	20	ug/L	92-111	75-125	20
6010C	Lead	7439-92-1	Water	8	50	30	50	ug/L	92-113	75-125	20
6010C	Lithium	7439-93-2	Water	2	10	3.5	10	ug/L	80-120	75-125	20
6010C	Magnesium	7439-95-4	Water	0.4	20	0.5	20	ug/L	86-115	75-125	20
6010C	Manganese	7439-96-5	Water	0.7	5	0.5	5	ug/L	92-112	75-125	20
6010C	Molybdenum	7439-98-7	Water	2	10	5	10	ug/L	92-113	75-125	20
6010C	Nickel	7440-02-0	Water	3	20	4	20	ug/L	91-118	75-125	20
6010C	Phosphorus	7723-14-0	Water	60	200	40	200	ug/L	80-120	75-125	20
6010C	Potassium	7440-09-7	Water	40	400	80	400	ug/L	89-114	75-125	20
6010C	Selenium	7782-49-2	Water	20	100	40	100	ug/L	88-113	75-125	20
6010C	Silicon	7440-21-3	Water	6	400	8	400	ug/L	80-120	75-125	20
6010C	Silver	7440-22-4	Water	5	10	10	10	ug/L	93-110	75-125	20
6010C	Sodium	7440-23-5	Water	20	200	20	200	ug/L	80-120	75-125	20
6010C	Strontium	7440-24-6	Water	0.9	10	0.25	10	ug/L	80-120	75-125	20
6010C	Thallium	7440-28-0	Water	30	100	60	100	ug/L	80-120	75-125	20
6010C	Tin	7440-31-5	Water	9	50	30	50	ug/L	80-120	75-125	20
6010C	Titanium	7440-32-6	Water	4	10	10	10	ug/L	80-120	75-125	20
6010C	Vanadium	7440-62-2	Water	6	10	6.25	10	ug/L	92-111	75-125	20
6010C	Zinc	7440-66-6	Water	2	10	2.5	10	ug/L	92-112	75-125	20

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METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
6010C	Aluminum	7429-90-5	Soil	6	10	10	10	mg/kg	41-158	75-125	20
6010C	Antimony	7440-36-0	Soil	3	10	6	10	mg/kg	50-150	75-125	20
6010C	Arsenic	7440-38-2	Soil	4	20	8	20	mg/kg	78-122	75-125	20
6010C	Barium	7440-39-3	Soil	0.08	2	0.16	2	mg/kg	81-119	75-125	20
6010C	Beryllium	7440-41-7	Soil	0.02	1	0.04	1	mg/kg	83-117	75-125	20
6010C	Boron	7440-42-8	Soil	0.4	10	0.8	10	mg/kg	67-133	75-125	20
6010C	Cadmium	7440-43-9	Soil	0.3	1	0.6	1	mg/kg	81-119	75-125	20
6010C	Calcium	7440-70-2	Soil	2	10	4	10	mg/kg	79-121	75-125	20
6010C	Chromium	7440-47-3	Soil	0.5	2	1	2	mg/kg	80-119	75-125	20
6010C	Cobalt	7440-48-4	Soil	0.4	2	0.8	2	mg/kg	82-118	75-125	20
6010C	Copper	7440-50-8	Soil	0.7	2	1.4	2	mg/kg	83-116	75-125	20
6010C	Iron	7439-89-6	Soil	0.7	4	1.4	4	mg/kg	50-149	75-125	20
6010C	Lead	7439-92-1	Soil	3	20	6	20	mg/kg	79-121	75-125	20
6010C	Lithium	7439-93-2	Soil	0.5	2	0.8	2	mg/kg	75-125	75-125	20
6010C	Magnesium	7439-95-4	Soil	0.08	4	0.16	4	mg/kg	73-127	75-125	20
6010C	Manganese	7439-96-5	Soil	0.04	2	0.08	2	mg/kg	81-119	75-125	20
6010C	Molybdenum	7439-98-7	Soil	0.7	2	1.4	4.5	mg/kg	75-125	75-125	20
6010C	Nickel	7440-02-0	Soil	0.6	4	1.2	4	mg/kg	81-118	75-125	20
6010C	Phosphorus	7723-14-0	Soil	4	40	8	40	mg/kg	75-125	75-125	20
6010C	Potassium	7440-09-7	Soil	20	80	40	80	mg/kg	73-126	75-125	20
6010C	Selenium	7782-49-2	Soil	5	20	10	20	mg/kg	80-120	75-125	20
6010C	Silver	7440-22-4	Soil	2	2	4	4	mg/kg	66-134	75-125	20
6010C	Sodium	7440-23-5	Soil	4	40	8	40	mg/kg	74-126	75-125	20
6010C	Strontium	7440-24-6	Soil	0.02	2	0.04	2	mg/kg	79-121	75-125	20
6010C	Thallium	7440-28-0	Soil	7	20	14	20	mg/kg	79-120	75-125	20
6010C	Tin	7440-31-5	Soil	4	10	8	10	mg/kg	75-124	75-125	20
6010C	Titanium	7440-32-6	Soil	0.8	2	1.6	2	mg/kg	75-125	75-125	20
6010C	Vanadium	7440-62-2	Soil	2	2	2	2	mg/kg	79-121	75-125	20
6010C	Zinc	7440-66-6	Soil	0.3	2	0.6	2	mg/kg	73-121	75-125	20

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6010C LL	Aluminum	7429-90-5	Soil	0.4	1	0.8	2	mg/kg	41-158	75-125	20
6010C LL	Antimony	7440-36-0	Soil	0.5	2	1	3	mg/kg	50-150	75-125	20
6010C LL	Arsenic	7440-38-2	Soil	0.9	2	1.8	5	mg/kg	78-122	75-125	20
6010C LL	Barium	7440-39-3	Soil	0.06	0.5	0.12	0.5	mg/kg	81-119	75-125	20
6010C LL	Beryllium	7440-41-7	Soil	0.03	0.1	0.06	0.2	mg/kg	83-117	75-125	20
6010C LL	Boron	7440-42-8	Soil	0.4	2	1.6	10	mg/kg	67-133	75-125	20
6010C LL	Cadmium	7440-43-9	Soil	0.03	0.1	0.06	0.18	mg/kg	81-119	75-125	20
6010C LL	Calcium	7440-70-2	Soil	0.6	2	1.2	3.6	mg/kg	79-121	75-125	20
6010C LL	Chromium	7440-47-3	Soil	0.2	0.5	0.4	1.2	mg/kg	80-119	75-125	20
6010C LL	Cobalt	7440-48-4	Soil	0.2	0.5	0.4	1.2	mg/kg	82-118	75-125	20
6010C LL	Copper	7440-50-8	Soil	0.3	0.6	0.6	1.8	mg/kg	83-116	75-125	20
6010C LL	Iron	7439-89-6	Soil	0.7	2	1.4	4.2	mg/kg	50-149	75-125	20
6010C LL	Lead	7439-92-1	Soil	0.4	2	0.8	2	mg/kg	79-121	75-125	20
6010C LL	Lithium	7439-93-2	Soil	0.5	2	1	3	mg/kg	80-120	75-125	20
6010C LL	Magnesium	7439-95-4	Soil	0.06	0.5	0.12	0.5	mg/kg	73-127	75-125	20
6010C LL	Manganese	7439-96-5	Soil	0.02	0.2	0.04	0.2	mg/kg	81-119	75-125	20
6010C LL	Molybdenum	7439-98-7	Soil	0.08	0.4	0.06	0.4	mg/kg	75-125	75-125	20
6010C LL	Nickel	7440-02-0	Soil	0.07	0.4	0.14	0.4	mg/kg	81-118	75-125	20
6010C LL	Phosphorus	7723-14-0	Soil	3	6	6	40	mg/kg	80-120	75-125	20
6010C LL	Potassium	7440-09-7	Soil	20	60	40	120	mg/kg	73-126	75-125	20
6010C LL	Selenium	7782-49-2	Soil	0.7	4	1.4	4.2	mg/kg	80-120	75-125	20
6010C LL	Silver	7440-22-4	Soil	0.2	0.5	0.4	1.2	mg/kg	66-134	75-125	20
6010C LL	Sodium	7440-23-5	Soil	4	40	8	40	mg/kg	74-126	75-125	20
6010C LL	Strontium	7440-24-6	Soil	0.02	2	0.06	2	mg/kg	80-120	75-125	20
6010C LL	Thallium	7440-28-0	Soil	0.4	2	0.4	20	mg/kg	79-120	75-125	20
6010C LL	Tin	7440-31-5	Soil	0.7	10	1.6	10	mg/kg	75-124	75-125	20
6010C LL	Titanium	7440-32-6	Soil	0.05	0.2	0.16	2	mg/kg	80-120	75-125	20
6010C LL	Vanadium	7440-62-2	Soil	0.3	1	0.6	2	mg/kg	79-121	75-125	20
6010C LL	Zinc	7440-66-6	Soil	0.3	1	0.6	2	mg/kg	73-121	75-125	20

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CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
6010C LL	Aluminum	7429-90-5	Water	0.5	2	6	18	ug/L	92-112	75-125	20
6010C LL	Antimony	7440-36-0	Water	3	10	6	18	ug/L	90-113	75-125	20
6010C LL	Arsenic	7440-38-2	Water	4	10	8	24	ug/L	90-112	75-125	20
6010C LL	Barium	7440-39-3	Water	0.4	2	0.8	2.4	ug/L	91-113	75-125	20
6010C LL	Beryllium	7440-41-7	Water	0.09	0.2	0.18	0.6	ug/L	91-113	75-125	20
6010C LL	Boron	7440-42-8	Water	2	10	8	50	ug/L	91-112	75-125	20
6010C LL	Cadmium	7440-43-9	Water	0.3	0.5	0.6	1.8	ug/L	93-113	75-125	20
6010C LL	Calcium	7440-70-2	Water	2	4	20	50	ug/L	85-116	75-125	20
6010C LL	Chromium	7440-47-3	Water	0.4	2	0.8	2.4	ug/L	93-114	75-125	20
6010C LL	Cobalt	7440-48-4	Water	0.4	1	0.8	2.4	ug/L	93-114	75-125	20
6010C LL	Copper	7440-50-8	Water	2	2	4	12	ug/L	91-111	75-125	20
6010C LL	Iron	7439-89-6	Water	3	10	6	18	ug/L	92-111	75-125	20
6010C LL	Lead	7439-92-1	Water	4	10	8	24	ug/L	92-113	75-125	20
6010C LL	Lithium	7439-93-2	Water	2	10	4	12	ug/L	80-120	75-125	20
6010C LL	Magnesium	7439-95-4	Water	0.4	2	6	20	ug/L	86-115	75-125	20
6010C LL	Manganese	7439-96-5	Water	0.2	0.6	0.4	2.4	ug/L	92-112	75-125	20
6010C LL	Molybdenum	7439-98-7	Water	0.6	2	1.2	3.6	ug/L	92-113	75-125	20
6010C LL	Nickel	7440-02-0	Water	0.7	2	1.4	4.2	ug/L	91-118	75-125	20
6010C LL	Phosphorus	7723-14-0	Water	7	20	7	400	ug/L	80-120	75-125	20
6010C LL	Potassium	7440-09-7	Water	50	100	100	300	ug/L	89-114	75-125	20
6010C LL	Selenium	7782-49-2	Water	5	20	10	30	ug/L	88-113	75-125	20
6010C LL	Silicon	7440-21-3	Water	10	50	10	400	ug/L	80-120	75-125	20
6010C LL	Silver	7440-22-4	Water	0.7	2	1.4	4.2	ug/L	93-110	75-125	20
6010C LL	Sodium	7440-23-5	Water	70	200	140	420	ug/L	80-120	75-125	20
6010C LL	Strontium	7440-24-6	Water	0.07	0.2	0.07	10	ug/L	80-120	75-125	20
6010C LL	Thallium	7440-28-0	Water	2	10	6	18	ug/L	80-120	75-125	20
6010C LL	Tin	7440-31-5	Water	2	10	2	10	ug/L	80-120	75-125	20
6010C LL	Titanium	7440-32-6	Water	0.2	1	0.8	10	ug/L	80-120	75-125	20
6010C LL	Vanadium	7440-62-2	Water	1	2	2	6	ug/L	92-111	75-125	20
6010C LL	Zinc	7440-66-6	Water	0.7	2	1.4	4.2	ug/L	92-112	75-125	20

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CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
6010C/PSEP	Aluminum	7429-90-5	Tissue	0.07	1	mg/kg	75-125	70-130	30
6010C/PSEP	Antimony	7440-36-0	Tissue	0.4	5	mg/kg	75-125	70-130	30
6010C/PSEP	Arsenic		Tissue	0.6	10	mg/kg	75-125	70-130	30
6010C/PSEP	Barium	7440-39-3	Tissue	0.04	0.5	mg/kg	75-125	70-130	30
6010C/PSEP	Beryllium		Tissue	0.02	0.5	mg/kg	75-125	70-130	30
6010C/PSEP	Boron	7440-42-8	Tissue	0.2	5	mg/kg	75-125	70-130	30
6010C/PSEP	Cadmium	7440-43-9	Tissue	3	5	mg/kg	75-125	70-130	30
6010C/PSEP	Calcium	7440-70-2	Tissue	2	5	mg/kg	75-125	70-130	30
6010C/PSEP	Chromium	7440-47-3	Tissue	0.08	0.2	mg/kg	75-125	70-130	30
6010C/PSEP	Cobalt		Tissue	0.05	1	mg/kg	75-125	70-130	30
6010C/PSEP	Copper	7440-50-8	Tissue	0.2	1	mg/kg	75-125	70-130	30
6010C/PSEP	Iron	7439-89-6	Tissue	0.4	2	mg/kg	75-125	70-130	30
6010C/PSEP	Lead		Tissue	0.2	5	mg/kg	75-125	70-130	30
6010C/PSEP	Lithium	7439-93-2	Tissue	0.3	0.5	mg/kg	75-125	70-130	30
6010C/PSEP	Magnesium	7439-95-4	Tissue	0.4	2	mg/kg	75-125	70-130	30
6010C/PSEP	Manganese	7439-96-5	Tissue	0.03	0.5	mg/kg	75-125	70-130	30
6010C/PSEP	Molybdenum	7439-98-7	Tissue	0.05	1	mg/kg	75-125	70-130	30
6010C/PSEP	Nickel	7440-02-0	Tissue	0.06	2	mg/kg	75-125	70-130	30
6010C/PSEP	Phosphorus	7723-14-0	Tissue	2	20	mg/kg	75-125	70-130	30
6010C/PSEP	Potassium	7440-09-7	Tissue	6	40	mg/kg	75-125	70-130	30
6010C/PSEP	Selenium	7782-49-2	Tissue	0.7	10	mg/kg	75-125	70-130	30
6010C/PSEP	Silver	7440-22-4	Tissue	0.1	1	mg/kg	75-125	70-130	30
6010C/PSEP	Sodium	7440-23-5	Tissue	4	20	mg/kg	75-125	70-130	30
6010C/PSEP	Strontium	7440-24-6	Tissue	0.02	1	mg/kg	75-125	70-130	30
6010C/PSEP	Thallium	7440-28-0	Tissue	0.3	10	mg/kg	75-125	70-130	30
6010C/PSEP	Tin	7440-31-5	Tissue	0.3	5	mg/kg	75-125	70-130	30
6010C/PSEP	Titanium	7440-32-6	Tissue	0.09	1	mg/kg	75-125	70-130	30
6010C/PSEP	Vanadium	7440-62-2	Tissue	0.07	1	mg/kg	75-125	70-130	30
6010C/PSEP	Zinc	7440-66-6	Tissue	0.06	1	mg/kg	75-125	70-130	30

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TABLE 3
Standard A for IRIS ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	(1)	100	10	1000	1.0
Antimony	Elemental Stock	1000	4	1000	4.0
Arsenic	(1)	100	10	1000	1.0
Arsenic	Elemental Stock	1000	4	1000	4.0
Boron	(1)	100	10	1000	1.0
Cadmium	(1)	100	10	1000	1.0
Calcium	(1)	100	10	1000	1.0
Calcium	Elemental Stock	10000	0.4	1000	4.0
Chromium	(1)	100	10	1000	1.0
Cobalt	(1)	100	10	1000	1.0
Copper	(1)	100	10	1000	1.0
Iron	(1)	100	10	1000	1.0
Lead	(1)	100	10	1000	1.0
Lead	Elemental Stock	1000	4	1000	4.0
Magnesium	(1)	100	10	1000	1.0
Magnesium	Elemental Stock	1000	3	1000	3.0
Manganese	(1)	100	10	1000	1.0
Manganese	Elemental Stock	1000	1	1000	1.0
Molybdenum	(1)	100	10	1000	1.0
Molybdenum	Elemental Stock	1000	1	1000	1.0
Nickel	(1)	100	10	1000	1.0
Selenium	(1)	100	10	1000	1.0
Selenium	Elemental Stock	1000	4	1000	4.0
Silver	(1)	100	10	1000	1.0
Tin	Elemental Stock	1000	5	1000	5.0
Thallium	(1)	100	10	1000	1.0
Thallium	Elemental Stock	1000	9	1000	9.0
Titanium	(1)	100	10	1000	1.0
Vanadium	(1)	100	10	1000	1.0
Zinc	(1)	100	10	1000	1.0
Hydrochloric Acid	-	-	50	1000	5%
Nitric Acid	-	-	10	1000	1%

(1) Mixed Standard, QCS-26

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TABLE 3 continued
Standard B for IRIS ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	1	1000	10
Barium	Elemental Stock	10000	0.5	1000	5.0
Beryllium	Elemental Stock	1000	0.1	1000	0.1
Calcium	Elemental Stock	10000	5	1000	50
Iron	Elemental Stock	10000	5	1000	50
Lithium	Elemental Stock	1000	1	1000	1.0
Magnesium	Elemental Stock	10000	5	1000	50
Manganese	Elemental Stock	1000	10	1000	10
Phosphorus	Elemental Stock	10000	2	1000	20
Potassium	Elemental Stock	10000	2	1000	20
Silicon	Elemental Stock	10000	0.5	1000	5
Sodium	Elemental Stock	10000	2	1000	20
Strontium	Elemental Stock	1000	1	1000	1.0
HCl	-	-	50	1000	5%
HNO3	-	-	10	1000	1%

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TABLE 4
Standard A for ICAP 6500 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	(1)	100	5	1000	0.5
Beryllium	(1)	100	5	1000	0.5
Boron	(1)	100	5	1000	0.5
Cadmium	(1)	100	5	1000	0.5
Calcium	(1)	100	5	1000	0.5
Chromium	(1)	100	5	1000	0.5
Cobalt	(1)	100	5	1000	0.5
Copper	(1)	100	5	1000	0.5
Iron	(1)	100	5	1000	0.5
Lead	(1)	100	5	1000	0.5
Magnesium	(1)	100	5	1000	0.5
Manganese	(1)	100	5	1000	0.5
Molybdenum	(1)	100	5	1000	0.5
Nickel	(1)	100	5	1000	0.5
Selenium	(1)	100	5	1000	0.5
Silver	(1)	100	5	1000	0.5
Tin	Elemental Stock	1000	0.5	1000	0.5
Thallium	(1)	100	5	1000	0.5
Titanium	(1)	100	5	1000	0.5
Vanadium	(1)	100	5	1000	0.5
Zinc	(1)	100	5	1000	0.5
Hydrochloric Acid	-	-	50	1000	5%
Nitric Acid	-	-	10	1000	1%

(1) Mixed Standard, QCS-26

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TABLE 4 continued
Standard B for ICAP 6500 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	2	1000	20
Arsenic	Elemental Stock	1000	2	1000	2
Barium	Elemental Stock	10000	2	1000	20
Calcium	Elemental Stock	10000	2	1000	20
Iron	Elemental Stock	10000	2	1000	20
Lithium	Elemental Stock	1000	2	1000	2
Magnesium	Elemental Stock	10000	2	1000	20
Phosphorus	Elemental Stock	10000	2	1000	20
Potassium	Elemental Stock	10000	2	1000	20
Silicon	Elemental Stock	10000	2	1000	20
Sodium	Elemental Stock	10000	2	1000	20
Strontium	Elemental Stock	1000	2	1000	2
HCl	-	-	50	1000	5%
HNO3	-	-	10	1000	1%

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TABLE 5
ICP ICV Standards

ICV1 Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	QCP-CICV-1	1000	2.5	500	5.0
Antimony	QCP-CICV-2	500	2.5	500	2.5
Arsenic	QCP-CICV-3	500	2.5	500	2.5
Barium	QCP-CICV-1	1000	2.5	500	5.0
Beryllium	QCP-CICV-1	25	2.5	500	0.125
Cadmium	QCP-CICV-3	250	2.5	500	1.25
Calcium	QCP-CICV-1	2500	2.5	500	12.5
Chromium	QCP-CICV-1	100	2.5	500	0.5
Cobalt	QCP-CICV-1	250	2.5	500	1.25
Copper	QCP-CICV-1	125	2.5	500	0.625
Iron	QCP-CICV-1	500	2.5	500	2.5
Lead	QCP-CICV-3	500	2.5	500	2.5
Magnesium	QCP-CICV-1	2500	2.5	500	12.5
Manganese	QCP-CICV-1	250	2.5	500	1.25
Molybdenum	Elemental Stock	1000	1.0	500	2.0
Nickel	QCP-CICV-1	250	2.5	500	1.25
Potassium	QCP-CICV-1	2500	2.5	500	12.5
Selenium	QCP-CICV-3	500	2.5	500	2.5
Silver	QCP-CICV-1	125	2.5	500	0.625
Sodium	QCP-CICV-1	2500	2.5	500	12.5
Thallium	QCP-CICV-3	500	2.5	500	2.5
Titanium	Elemental Stock	1000	1.0	500	2.0
Vanadium	QCP-CICV-1	250	2.5	500	1.25
Zinc	QCP-CICV-1	250	2.5	500	1.25
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

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TABLE 5 continued

ICVB Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	1000	0.5	500	1
Boron	Elemental Stock	1000	2.5	500	5
Calcium	Elemental Stock	1000	2.5	500	5
Iron	Elemental Stock	1000	5	500	10
Lithium	Elemental Stock	1000	1	500	2
Magnesium	Elemental Stock	1000	2.5	500	5
Manganese	Elemental Stock	1000	5	500	10
Phosphorus	Elemental Stock	1000	2.5	500	5
Silicon	Elemental Stock	1000	2.5	500	5
Strontium	Elemental Stock	1000	1	500	2
Tin	Elemental Stock	1000	2.5	500	5
Hydrochloric Acid	-		25	500	5%
Nitric Acid	-		5	500	1%

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TABLE 6
ICP Interference Check Solutions

ICSA Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Calcium	CLPP-ICS-A	5000	50	500	500
Iron	CLPP-ICS-A	2000	50	500	200
Magnesium	CLPP-ICS-A	5000	50	500	500
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

ICSAB Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Antimony	Elemental Stock	1000	0.5	500	1
Barium	CLPP-ICS-B	50	5	500	0.5
Beryllium	CLPP-ICS-B	50	5	500	0.5
Cadmium	CLPP-ICS-B	100	5	500	1
Calcium	CLPP-ICS-A	5000	50	500	500
Chromium	CLPP-ICS-B	50	5	500	0.5
Cobalt	CLPP-ICS-B	50	5	500	0.5
Copper	CLPP-ICS-B	50	5	500	0.5
Iron	CLPP-ICS-A	2000	50	500	200
Lead	CLPP-ICS-B	100	5	500	1
Magnesium	CLPP-ICS-A	5000	50	500	500
Manganese	CLPP-ICS-B	50	5	500	0.5
Nickel	CLPP-ICS-B	100	5	500	1
Silver	CLPP-ICS-B	100	5	500	1
Vanadium	CLPP-ICS-B	50	5	500	0.5
Zinc	CLPP-ICS-B	100	5	500	1
HCl	-	-	25	500	0.05
HNO3	-	-	5	500	0.01

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TABLE 7
IRIS Analytical Wavelengths

Analyte	Wavelength	
Aluminum	237.3	
Antimony	206.8	
Arsenic	189.0	
Barium	233.5	
Beryllium	313.0	
Boron	249.7	
Cadmium	226.5	
Calcium	317.9	
Calcium	211.2	High Line
Chromium	267.7	
Cobalt	228.6	
Copper	324.7	
Iron	259.9	
Iron	271.4	High Line
Lead	220.3	
Lithium	670.7	
Magnesium	279.5	
Magnesium	202.5	High Line
Manganese	257.6	
Manganese	293.9	High Line
Molybdenum	202.0	
Nickel	231.6	
Phosphorus	214.9	
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	589.5	
Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	323.4	
Vanadium	310.2	
Zinc	206.2	

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TABLE 8
ICAP 6500 Analytical Wavelengths

Analyte	Wavelength	
Aluminum	167.0	Low Line
Aluminum	394.4	
Antimony	206.8	
Antimony	217.5	Alternate
Arsenic	189.0	
Barium	455.4	
Beryllium	234.8	
Boron	249.6	
Cadmium	226.5	
Cadmium	214.4	Alternate
Calcium	315.8	
Calcium	393.3	Low Line
Chromium	267.7	
Cobalt	230.7	
Cobalt	228.6	Alternate
Copper	327.3	
Copper	224.7	Alternate
Iron	259.9	
Lead	220.3	
Lithium	670.7	
Magnesium	279.0	High Line
Magnesium	279.5	Low Line
Magnesium	285.2	
Manganese	257.6	
Manganese	260.5	High Line
Molybdenum	202.0	
Nickel	221.6	
Nickel	231.6	Alternate
Phosphorus	214.9	
Phosphorus	178.2	Alternate
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	588.9	Alternate
Sodium	589.5	

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TABLE 8
ICAP 6500 Analytical Wavelengths
continued

Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	336.1	
Vanadium	292.4	
Zinc	206.2	
Zinc	213.8	Alternate