

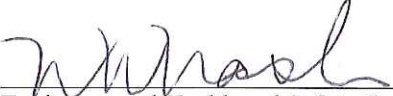



## FACILITY SOP ATTACHMENT

<b>SOP NUMBER:</b> IR-MET-ICP, Rev. 3	<b>CHANGE FORM ID:</b> CF1
<b>SOP TITLE:</b> Metals by ICP – EPA Method 200.7/6010	
<b>REASON FOR ADDITION OR CHANGE (Use additional sheets if necessary):</b> Addition of a digested Reporting Limit Verification (MRL) for drinking water samples that required digestion.	
<b>CHANGE OR ADDITION (Use additional sheets if necessary):</b> <u>Add section 9.1.4 Method Reporting Limit (MRL) for drinking water samples</u>  Prepare and analyze an MRL for every batch of 20 samples or less. The analyte recoveries for the MRL samples must fall within $\pm 50\%$ of the theoretical value for Arizona samples and must be detected for CA and Federal samples. Note: this requirement is a drinking water compliance requirement, not a method requirement. If the MRL is outside of the limit for a requested metal, re-prepare and re-analyze once: <ul style="list-style-type: none"> <li>• If the MRL is still below the acceptance limit, the affected samples must be re-digested and re-analyzed.</li> <li>• If the MRL is above the acceptance limits and samples are ND, the results may be reported. The results must be flagged and a non-conformance memo (NCM) written.</li> <li>• All positive samples must be re-analyzed with the MRL if the MRL is re-analyzed.</li> </ul>	
<b>Prepared By:</b> L. Hoang	
<b>APPROVED BY:</b>	
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**Title: Metals by ICP  
EPA Methods 200.7 and 6010B**

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**1.0 SCOPE AND APPLICATION**

**1.1** This method describes the simultaneous multi-elemental determination of trace metals by Inductively Coupled Plasma (ICP), atomic emission spectroscopy (AES). Methods 200.7 and 6010B are used to determine trace metals in the following matrices: water, drinking water, waste water, and for NPDES Compliance Monitoring. More information summarizing the principles of ICP can be found in EPA Methods 200.7 and 6010B.

**1.2** Elements are generally measured at the following wavelengths:

**1.2.1** Routinely analyzed elements:

Element	Wavelength (nm)	Element	Wavelength (nm)
Aluminum (Al)	308.215	Manganese (Mn)	257.610
Antimony (Sb)	206.833	Molybdenum (Mo)	202.030
Arsenic (As)	188.979	Nickel (Ni)	231.604
Boron (B)	249.773	Phosphorus (P)	214.914
Barium (Ba)	233.527	Potassium (K)	766.490
Beryllium (Be)	313.107	Selenium (Se)	196.026
Cadmium (Cd)	214.438	Silicon (Si)	215.611
Calcium (Ca)	315.887	Silver (Ag)	328.068
Cobalt (Co)	228.616	Sodium (Na)	589.592
Chromium (Cr)	205.552	Strontium (Sr)	421.552
Copper (Cu)	324.754	Thallium (Tl)	190.800
Iron (Fe)	238.204	Tin (Sn)	189.933
Lead (Pb)	220.353	Titanium (Ti)	334.941
Lithium (Li)	610.362	Vanadium (V)	292.402
Magnesium (Mg)	279.079	Zinc (Zn)	213.856

See Attachments 1 and 2 for nominal reporting limits.

**1.2.2** Special Request elements\*:

Element	Wavelength (nm)	Element	Wavelength (nm)
Bismuth (Bi)	223.061	Scandium (Sc)	357.253, 361.383, 424.683
Gallium (Ga)	294.364 & 417.206	Zirconium (Zr)	343.823
Tungsten (W)	207.912		

\*Nominal reporting limits for waters are 0.1 mg/L for Bi, Ga, and Sc. 0.2 mg/L for Zr

- 1.3 If silver (Ag) analysis is requested, the sample must be digested in all cases. When silver concentrations exceed the concentration of the LCS (0.25 ppm) for 200.7, a dilution must be performed by re-digestion, rather than diluting the digestate at the instrument. Boron and silica digestions must be performed in quartz or Teflon beakers.
- 1.4 **Method 200.7 is not recommended for low-level analysis of elements such as Antimony, Arsenic, Beryllium, Cadmium, Selenium and Thallium in Drinking Waters.** For drinking waters, they must be analyzed using EPA 200.8 (ICPMS).
- 1.5 Drinking Water Maximum Contaminant Levels (MCL) and California Detection Limits for reporting purposes (DLR) are found below. If a sample is analyzed for drinking waters, depending on the requested analytes, the sample may have to be concentrated, analyzed by an alternative method (e.g. 200.8), or reported with J flags (down to MDL) in order to meet the DLR requirements. See attached Analysis codes for the laboratory's standard analyte list and reporting limits.

Analyte	DLR (mg/l)	MCL (mg/l)	Analyte	DLR (mg/L)	MCL (mg/l)
Aluminum	0.050	1.0	Iron	0.10	0.30
Antimony	0.0060	0.0060	Lead	0.0050	0.0
Arsenic	0.002	0.010	Manganese	0.020	0.050
Barium	0.10	1.0	Nickel	0.010	0.10
Beryllium	0.0010	0.0040	Selenium	0.0050	0.050
Cadmium	0.0010	0.0050	Silver	0.010	0.10
Chromium (total)	0.010	0.050	Thallium	0.0010	0.0020
Copper	0.050	1.0	Zinc	0.050	5.0

From Chemicals and Parameters in California Drinking Water Quality Database (12/19/07)

- 1.6 On occasion, clients may request modifications to this SOP. These modifications are handled following the procedures outlined in "Validation of Methods" in the Quality Assurance Manual.

## 2.0 SUMMARY OF METHOD

- 2.1 Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation method (EPA 200.2, EPA 3005A, EPA 3050B or EPA 3010A). For dissolved metals, acid digestion is not necessary if the samples are filtered through a 0.45 µm membrane filter and acid preserved prior to analysis. For drinking water, if the measured turbidity is <1 NTU, digestion is not required but samples must be matrix matched to calibration standards. Yttrium internal standard is added to all samples and QC.
- 2.2 Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by optical photosensitive devices.
- 2.3 Concentrations are quantitated using a linear regression calibration curve.

## 3.0 DEFINITIONS

- 3.1 IEC--Inter-element correction
- 3.2 MSF--Multi-component spectral fitting
- 3.3 NCM—Non-Conformance Memo

There are no additional specific definitions associated with this test. See the laboratory QA manual and EPA methods 200.7 and 6010B for general definitions.

#### 4.0 INTERFERENCES

##### 4.1 Spectral Interferences

4.1.1 Spectral interferences are caused by background emission from stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.2 Spectral interference effects are minimized by maintaining constant plasma conditions and through the use of background corrections (IECs and/or MSF).

4.1.3 Background emission must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

##### 4.2 Physical interferences

4.2.1 Physical interferences are effects associated with the sample nebulization and transport process.

4.2.2 Samples with high dissolved solids (TDS) or high acid concentrations can cause changes in viscosity and surface tension which, in turn, affects the sample nebulization and transport.

4.2.3 Physical interference effects can be minimized through the use of a peristaltic pump and internal standard.

##### 4.3 Chemical interferences

4.3.1 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects.

4.3.2 These types of interferences are not normally a problem with ICP analysis. However maintaining consistent plasma conditions and the use of special procedures such as buffering the solutions, matrix matching standards or the method of standard addition (MSA) may help reduce chemical interferences.

##### 4.4 Memory interferences

4.4.1 Memory interference result when analytes in a previous sample contribute to the signals measured in a new sample.

**4.4.2** Optimizing the rinse time between samples and using a rinse solution with the proper acid strength can minimize memory effects.

**4.4.3** Even using these precautions, a sample may be too high to rinse completely under normal circumstances. The analyst must be aware of this situation, and samples immediately following a high sample should be re-analyzed.

## **5.0 SAFETY**

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats, and closed-toe, nonabsorbent shoes are a minimum.

### **5.1 Specific Safety Concerns or Requirements**

Personal Protective Equipment Required: safety glasses, face shield, lab coat and nitrile gloves.

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

### **5.2 Primary Materials Used**

The following is a list of the materials used in this method that have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

<b>Material (1)</b>	<b>Hazards</b>	<b>Exposure Limit (2)</b>	<b>Signs and symptoms of exposure</b>
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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm- STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

## 6.0 EQUIPMENT AND SUPPLIES

### 6.1 Instrumentation

- 6.1.1 Inductively coupled plasma (ICP) - OPTIMA 3000XL/4300 or equivalent
- 6.1.2 Autosampler - ESI SC-4DX Fast or equivalent
- 6.1.3 Personal computer for data acquisition, monitor, keyboard, and printer

### 6.2 Supplies

- 6.2.1 Argon gas
- 6.2.2 Pipettes, pipettors and tips
- 6.2.3 Volumetric flasks – various sizes
- 6.2.4 Sample tube racks
- 6.2.5 Graduated centrifuge tubes (15 ml)
- 6.2.6 Plastic containers

## 7.0 REAGENTS AND STANDARDS

### 7.1 Reagents

All purchased and prepared reagents must be made from a traceable (NIST) source material, if available, and documentation of this traceability must be maintained by the laboratory.

- 7.1.1 Yttrium standard solution (10000 ppm)
- 7.1.2 Concentrated Nitric Acid (HNO<sub>3</sub>, instrument grade)
- 7.1.3 Reagent Grade Water (Ultrapure water)

**7.2 Standards**

All purchased standards must be accompanied by a Certificate of Analysis (C of A) which is kept available at the laboratory in order to demonstrate traceability of the standard to certified (NIST-traceable) source material. All prepared standards must be made from a traceable (NIST, if available) source material, if available documentation of this traceability must be maintained by the laboratory.

- 7.2.1 O2SI Metal Stock Standards (or equivalent)
- 7.2.2 Accutrace standard Metal Stock Standards (or equivalent)
- 7.2.3 Accutrace standard or Spex Spectral Interference Check Solution (SICS):  
500ppm Al, Ca and Mg; 200ppm Fe
- 7.2.4 Sodium (1000ppm), Accutrace, Spex or equivalent standard.
- 7.2.5 Potassium (1000ppm), Accutrace, Spex or equivalent standard.
- 7.2.6 10000ppm stock standards: Al, Ca, Mg, Fe, Cr, Cu, Mn, Mo, Ni, Ti, V, Zn, Sn, Co

**8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE**

Sample containers, preservation techniques, and holding times may vary and are dependent on sample matrix, EPA method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters <sup>1,2</sup>	Polyethylene bottles	500 ml	HNO <sub>3</sub> to pH<2	6 months	40 CFR Part 136.3
Soils	Glass jar/ brass sleeve	4 oz	None (Cool >0 to 6°C if Hg needed)	6 months	SW846 Chapter 3

Footnote 1: Water samples received unpreserved must be acidified and allowed to sit for 24 hours prior to analysis.

Footnote 2: For analysis of dissolved metals, samples must be filtered and preserved in the field within 15 minutes of sampling. If samples are not preserved, sample must be acidified and allowed to sit for 24 hours prior to analysis.

Footnote 3: Samples received unpreserved for dissolved metals are filtered as soon as possible (generally within 24 hours) and acidified immediately after filtration. Samples can then be digested/analyzed immediately (i.e. they do not need to sit for 24 hours).

**9.0 QUALITY CONTROL**

**9.1 Sample QC**

The following quality control samples are prepared with each batch of samples. Each of these QC samples may be re-analyzed once if it doesn't pass, prior to sample analysis, in order to verify the failure wasn't due to a physical or mechanical problem.

NOTE: allowances for the reporting of non-detect data associated with high-biased QC (Blank and LCS) may be prohibited by specific programs or client project plans.



### 9.1.1 Method Blank (MB)

Prepare and analyze a method blank (MB) for each matrix and with every batch of 20 samples or less. Check that there are no analytes detected ( $< \pm RL$  if reporting to RL,  $< \pm MDL$  if reporting to MDL per specific client request)

If the method blank (MB) shows contamination for a requested metal, re-prepare and re-analyze MB once unless:

- The sample concentrations are not detected (ND). Flag the result accordingly and write an NCM.
- The sample result is  $> 10x$  the blank level. Flag the result accordingly and write an NCM

If the MB is re-analyzed, all positive samples  $< 10x$  RL must also be reanalyzed.

If the re-analyzed MB still shows contamination, re-prepare and re-digest the affected samples.

### 9.1.2 Laboratory Control Sample (LCS)

Prepare and analyze a primary source laboratory control sample (LCS) for every batch of 20 samples or less at following concentrations:

	All metals	Na, K	Ca, Mg, Si
EPA 200.7	0.5 ppm	5 ppm	2.5 ppm
EPA 6010B	1.0 ppm	10 ppm	5.0 ppm

The LCS recovery must be within **85-115%** for EPA 200.7 and **80-120%** for EPA 6010B and relative percent difference within  $\pm 20\%$

If the LCS is outside of the limit for a requested metal, re-prepare and re-analyze the LCS once:

- If the LCS is still below the acceptance limit, the effected samples must be re-digested and re-analyzed.
- If the LCS is above the acceptance limits and samples are ND, the results may be reported. The results must be flagged and a NCM must be submitted.
- If the LCS is above the acceptance limits and samples are greater than or equal to reporting limit, samples and LCS must be re-digested and/or re-analyzed.
- If the LCS is above the acceptance limits and samples are to be reported down to the MDL (J-flagging), then all samples greater than or equal to the MDL and the LCS must be re-digested and/or re-analyzed.

LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

### 9.1.3 Matrix Spike and Matrix Spike Duplicate (MS/MSD).

The sample for MS/MSD is randomly selected, unless specifically requested by a client. Prepare and analyze a matrix spike (MS) and a matrix spike (MSD) duplicate for each matrix and with every batch of 10 samples, or less for EPA 200.7 and every 20 samples or less for EPA 6010B. The recovery must be within **70-130%** for EPA 200.7 and **75-125%** for EPA 6010B unless the source is 4 times greater than the amount spiked. A relative percent difference within  $\pm 20\%$ , unless there is matrix interference. In the case of matrix interference, flag the results accordingly and write an NCM.

## 9.2 Instrument QC

The following instrument QC samples are run with each analytical sequence. Each of these QC samples may be re-analyzed once if it does not pass in order to verify the failure wasn't due to a physical or mechanical problem. Re-analysis must be performed before the analysis of any associated batch QC or client samples.

### 9.2.1 Calibration Acceptance Summary

Prepare a calibration curve by plotting the response (peak area) of at least two standards and a blank against the corresponding concentrations. The resulting correlation coefficient (r) must be  $\geq 0.995$ . If this criterion is not met re-prepare the calibration standards and repeat the calibration. Refer to the "Calibration Curves" SOP and the "Selection of Calibration Points" SOP for more information on calibrating the instrument.

### 9.2.2 Internal Standard

The percent recovery of Internal Standard in all samples, batch QC and calibration checks must be within 60-140% of the true value.

- If the recovery in any calibration standard is outside acceptance limits, re-calibration is required.
- If the recovery is outside acceptance limits in any other QC sample (not including MS/MSD), re-analysis must be performed.
- If the recovery is outside acceptance limits in any other samples or MS/MSD because of matrix interference, dilute and re-analyze the samples.
- Otherwise, troubleshoot the ICP instrumentation.

### 9.2.3 Initial Calibration Verification (ICV)

Immediately after the initial calibration, analyze a secondary source ICV. Verify that that it meets the following criteria:

	% Recovery	%RSD
EPA 200.7	95-105	5
EPA 6010B	90-110	5

- If the recovery for any requested metal is outside the acceptance criteria, re-prepare and re-analyze the ICV once.
- If the ICV is still below the acceptance limit (<95% for 200.7 or <90% for 6010B) for the requested metal, re-calibrate and re-analyze the ICV.
- If the ICV is above the acceptance limits (105% for 200.7 or 110% for 6010) and samples are ND, the results may be reported with a flag and an appropriate NCM.
  - If the ICV is outside limits (high or low), all samples greater than or equal to RL (or MDL for samples reported to the MDL) must be re-analyzed with a new calibration and passing ICV.

#### 9.2.4 Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

- Analyze the initial calibration blank (ICB) immediately after the ICV and the continuing calibration blank after every CCV. The absolute value of the blanks should not be greater than the reporting limit (RL) for bracketed samples at concentrations above twice the RL (2xRL). For reporting samples with concentrations at or below twice the RL, the ICB/CCB must read less than half the RL or less than the MDL, whichever is greater.
- If any element exceeds the limit, check to see if it is a requested metal. If the element is not required for the analysis run, the analysis can proceed as is.
- If the element is required, re-analyze the ICB and, if it is still out of limits, re-calibrate and re-analyze ICV and ICB.
- Re-analysis is not required if the sample results are  $\geq 10x$  the absolute value of the calibration blank or the calibration blank concentration is above the RL (or MDL for samples reported to the MDL) and samples are not detected (ND).

#### 9.2.5 Continuing Calibration Verification (CCV)

- Analyze a CCV standard after every 10 sample aliquots (injections) or less and at the end of the analysis sequence. The recovery must be within  $\pm 10\%$  of the true value and its RSD must be 5% or less.
- If the CCV is outside recovery limits for the element(s) of interest, re-analyze once.
  - If it is still outside the recovery limits after re-analysis, the instrument must be re-calibrated and samples not bracketed by acceptable CCVs must be re-analyzed.
- If the CCV fails **above** the limits, any ND samples may be reported with a flag and an NCM.
- If a CCV fails during an overnight or unattended run, re-analyze all samples that need analysis of the element(s) not bracketed by acceptable CCVs.
- All samples greater than or equal to RL (or MDL for samples reported to the MDL) must be re-analyzed with a passing CCV.

#### 9.2.6 Reporting Limit (RL) Check

- A Reporting Limit check at 1x or 2x the RL must be run before analyzing samples (and at the end of run if required by specific projects). For drinking water, it must be a 1x RL. A recovery of 50-150% must be achieved.
- If the RL check is outside recovery limits for the element(s) of interest, re-prepared and re-analyze once. If it is still out, the instrument must be recalibrated before analyzing samples.
- If the RL check fails **above** the limits, any ND samples may be reported with a flag and an appropriate NCM.

#### 9.2.7 ICSA/ICSAB

- An ICSA is run before analyzing samples. Verify that the absolute value of any false reading is less than 2x RL of the interfered element.
- An ICSAB is run after the ICSA. Verify that the percent recovery of spiked elements is within 80-120% of the true value of the spike amount.
- If the ICSA or ICSAB are outside recovery limits for the element(s) of interest, re-analyze once.
- If the ICSA or ICSAB are still outside the recovery limits after re-analysis, IEC factor must be adjusted (see Calculation of IEC offset section) and the instrument must be recalibrated.
- If the ICSA or ICSAB fail **above** the requirements, any ND samples may be reported with a flag and an appropriate NCM
- If required by specific projects, close the run sequence with an ICSA and ICSAB.

#### 9.2.8 Precision (RSD%) and Response range

Sample replicates must meet the following precision:

- Results less than 2 x RL must have an RSD < 50%.
- Results equal or greater than 2 x RL must have RSD < 25% between replicates.
- Otherwise, re-analyze samples

#### 9.2.9 Instrument Detection Limit (IDL)

- The IDL is the concentration equivalent to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal of the same element.
- The IDL of each element should be compiled every six months.
- The IDL should not be higher than Method Detection Limit of the same element.
- If the IDL for any element exceeds the MDL, instrument performance should be evaluated and any needed preventative maintenance conducted. If the IDL still exceeds the existing MDL, the higher of the two will be used as the MDL.

#### 9.2.10 Linear dynamic range (LDR)

Every year, verify the response of each reported element by analyzing single element standards at 20 ppm or higher. The percent recovery of each element must be within 90-110% of the true value at the highest reportable range. Document the achieved linear ranges.

Sample readings must not be greater than 90% of the linear dynamic range (LDR), or the samples have to be diluted.

### 9.2.11 Spectral Interference Adjustments

- IEC (Inter-element correction)
  - IEC is used to adjust for spectral interferences between two elements by assigning a factor that corrects the concentration of the target metal based on the concentration of the interferent metals.
  - IECs are based on the fixed spectral characteristics of each metal. For a given wavelength of the target, only specific metals will be interferents. When IECs are established, the corrections used should be verified to ensure they are not erroneously based on other than the spectral characteristics.
  - Verify and update (if necessary) every six months using the IEC factors from the analysis of 20 ppm single element standards (except Fe at 200 ppm, and Al, Ca, Mg at 500 ppm). The percent recovery of each element must be within 90-110% of the spiked value. Ensure that the standards are free of contamination and the background correction points selected for peak integration are clear of spectral interference. NOTE: verification is required only for those metals processed using IECs.
  - Samples that have IEC interferent elements (Al, Ca, Fe, and Mg), at concentrations greater than or equal to 120% of the IEC check levels must be analyzed at dilutions that lower these concentration below 120% of the IEC check level.
- MSF (Multicomponent Spectral Fitting)
  - MSF uses multivariate calibration to determine the concentration of an analyte. Multivariate calibration is analogous to a mathematical filter that can distinguish between the components of a complex spectral profile. The signal contribution of three (3) components can be separated from the analyte: interference, background, and noise.
  - MSF is beneficial when:
    - it is difficult to assess background correction points, or
    - the noise contribution is significant, such as in measurements near the analyte detection limit.
  - MSF is not suitable where there is complete overlap of analyte and interferent. In that case, IEC should be used instead.

## 10.0 PROCEDURE

### 10.1 Standard Preparation

#### 10.1.1 Acidified Reagent Grade Water:

Prepare acidified water to use in the preparation of all subsequent standards. Add the appropriate volume of HNO<sub>3</sub> and HCl to Reagent Grade Water to obtain a 1% (v/v) HNO<sub>3</sub> and 2% (v/v) HCl solution.

**10.1.2 ICP rinse:**

Prepare the ICP rinse solution by adding the appropriate volume of HNO<sub>3</sub> and HCl to Reagent Grade Water to obtain a 5% (v/v) HNO<sub>3</sub> and 5% (v/v) HCl solution.

**10.1.3 Daily Standard:**

Daily, prepare the calibration standards, S1, S2, S3, etc., by adding the appropriate volumes of Accutrace standard stock solutions to acidified Reagent Grade Water. The final concentrations are listed below.

Calibration Std	ppm (mg/L) <sup>1</sup>
Blank	0
S1	0.005 (Ca, Mg, K, Na at 0.05, Si at 0.025, Ag at 0.0025)
S2	0.1 (Ca, Mg, K, Na at 1, Si at 0.5, Ag at 0.05)
S3	1 (Ca, Mg, K, Na at 10, Si at 5.0, Ag at 0.5)

<sup>1</sup>There are metals (Ga, Bi, Sc, Zr, W, etc.) that are not routinely analyzed and the calibration concentrations are not shown above. The calibration concentrations will be shown on the instrument printout.

**10.1.4 RL Check:**

Prepare daily a Reporting Limit (RL) Check Standard at twice the RL (**at the RL for drinking water**) by using appropriate volumes of Accutrace standard stock solutions to acidified Reagent Grade Water.

**10.1.5 ICB and CCB**

The initial calibration blank (ICBV) and the continuing calibration blank (CCB) are prepared by acidifying Reagent Grade Water to the same concentrations of the acids found in the standards and samples.

**10.1.6 ICV (2<sup>nd</sup> source):**

The Initial Calibration Verification (ICV) standard is prepared at 2.0 ppm [20 ppm for Na, K, 10 ppm Si, 1 ppm Ag, 2.0 ppm for Ca (Spex) or 10 ppm Ca and Mg(O<sub>2</sub>Si)] from a secondary source or different lot number than that of the calibration standard.

**10.1.7 CCV:**

The Continuing Calibration Verification (CCV) – is prepared at 1ppm (10ppm for Na, K; 5 ppm for Si; 0.5 for Ag) from the same source as that used for the calibration standards.

**10.1.8 ICSA:**

The Interference Check Solution (ICS) - consists of ICSA and ICSAB portions. The ICSA solution contains 500 ppm for Al, Ca, Mg and 200 ppm for Fe. The ICSA provides a test of the correction factors. Prepare the solution with acidified Reagent Grade Water.

**10.1.9 ICSAB:**

The Interference Check Solution B (ICSAB) is prepared from the initial calibration standard

solution at a concentration of 0.5 ppm for all target elements except: K and Na at 10 ppm; Si at 5ppm; Ag at 0.25ppm; Fe at 200 ppm; Al, Ca, and Mg at 500 ppm.

Document the preparation of all intermediate standards in the data system (LIMS) and ensure it is peer-reviewed before use. Store the solution at room temperature for up to one year.

## **10.2 Sample Preparation**

See the appropriate laboratory SOP for sample preparation procedures.

### **10.2.1 Water samples to be analyzed by EPA 200.7 are to be treated per EPA 200.2.**

- Pour the sample into a 15mL centrifuge tube, dilute if necessary.
- The internal standard is added using the peristaltic pump of the instrument or autosampler.

### **10.2.2 Water samples to be analyzed by EPA 6010 B are to be digested per EPA 3005.**

- Prepare the sample in the same way as EPA 200.7.

### **10.2.3 Soil samples to be analyzed by EPA 6010B are to be digested per EPA 3050.**

- Soil samples are diluted 5X after digestion and before analysis.
- Pipette 2.5 mL of the sample into a centrifuge tube.
- Add 10 mL of acidified Reagent Grade Water.
- The internal standard is added using the peristaltic pump of the instrument or autosampler.

### **10.2.4 Soil samples to be analyzed by EPA 6010B are to be digested per EPA 3050 – Special Low Level**

- Soil samples are diluted 4X after digestion and before analysis.
- Pipette 3 mL of the sample into a centrifuge tube.
- Add 9 mL of acidified Reagent Grade Water.
- The internal standard is added using the peristaltic pump of the instrument or autosampler.

### **10.2.5 Soil samples to be analyzed by EPA 6010B are to be digested per EPA 3051.**

- Soil samples are diluted 2X after digestion and before analysis.
- Pipette 6 mL of the sample into a centrifuge tube.
- Add 6 mL of acidified Reagent Grade Water.
- The internal standard is added using the peristaltic pump of the instrument or autosampler.

### **10.2.6 Leachates (SPLP or TCLP) are to be digested per EPA 3010.**

- Pour the sample into a 12mL centrifuge tube.
- The internal standard is added using the peristaltic pump of the instrument or autosampler.

### **10.2.7 Leachates (STLC)**

- Pipette 0.5mL of a STLC sample into a centrifuge tube.
- Add 9.5 mL of acidified Reagent Grade Water.
- The internal standard is added using the peristaltic pump of the instrument or autosampler..

### **10.2.8 Leachates (DI WET)**

- Pour the sample into a 15mL centrifuge tube.

- The internal standard is added using the peristaltic pump of the instrument or autosampler..

#### **10.2.9 Cation Exchange Capacity (CEC)**

- Pipette 1mL of a CEC sample into a centrifuge tube.
- Add 9 mL of acidified Reagent Grade Water.
- The internal standard is added using the peristaltic pump of the instrument or autosampler.

#### **10.3 Instrument Initialization**

- 10.3.1** Follow the instructions provided by the instrument manufacturer for operating conditions.
- 10.3.2** Prior to daily calibration of the instrument the manganese axial and radial alignment is performed using a 1ppm Mn standard. Note the highest Mn intensity. If the intensity differs by more than 1/3 from one day to the next then maintenance is required for the instrument.
- 10.3.3** Mercury (Hg) alignment is performed whenever the torch is moved, cleaned, or changed.
- 10.3.4** A Cu/Mn intensity ratio is not calculated.
- 10.3.5** Transfer all the standards and samples into their corresponding centrifuge tubes. Place the centrifuge tubes into the autosampler. Yttrium is used as the internal standard, and is added to every sample and standard.

#### **10.4 Calibration**

- 10.4.1** Calibrate the instrument daily using a blank, S1, S2 and S3.
- 10.4.2** Verify the linearity of the initial calibration and its acceptance against a secondary source ICV standard.

#### **10.5 Sample Analysis**

- 10.5.1** A typical daily run sequence is listed below:



	Recovery (%)	RSD (%)
1 Initial calibration		
2 ICV (6010B):	90 – 110%	5%
ICV (200.7):	95 – 105%	5%
3 ICB	<±RL for samples ≥2x RL OR ≤ ±½ RL or MDL (whichever is greater) for samples <2x RL	
4 ICSA	≤ 2x RL	
5 ICSAB	80 – 120%	
6 RL check	50 – 150%	
7 Method Blank	< RL	
8 LCS	200.7: 85-115% RPD: 20% 6010B: 80-120% RPD: 20%	5%
9 Sample		25%*
10 MS	200.7: 70-130% RPD: 20% 6010B: 75-125% RPD: 20%	
11 MSD	Same as above	
12 5 sample aliquots		25%**
13 CCV	90 – 110%	5%
14 CCB	<±RL for samples ≥2x RL OR ≤ ±½ RL or MDL (whichever is greater) for samples <2x RL	
15 10 sample aliquots		25%*
16 CCV	90 – 110%	5%
17 CCB	<±RL for samples ≥2x RL OR ≤ ±½ RL or MDL (whichever is greater) for samples <2x RL	

*ICSA/ICSAB is at the close if required by special project.*

\*For sample results >2X the RL, otherwise RSD must be ≤50%.

No RSD requirements for results < RL

## 10.6 Preventative Maintenance

**10.6.1** Daily maintenance includes, but is not limited to, the inspection, cleaning, and/or replacement of the following items: peristaltic pump tubing; nebulizer assembly and torch; inert gas; and rinse solution and waste containers.

**10.6.2** Document all non-routine maintenance. Document any necessary maintenance performed to bring the instrument back to control status after failing QC checks.

**10.6.3** If an instrument is unusable or has limitation to its use, it must be tagged accordingly until such a time the problem has been corrected. Record the problem, solution, date, and verification of proper operation into the instrument maintenance logbook.

## 11.0 CALCULATIONS / DATA REDUCTION

### 11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{Observed concentration}}{\text{Known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{Spiked sample}) - (\text{Unspiked sample})}{\text{Spiked concentration}} \times 100$$

## 11.2 **Precision (RPD)**

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

## 11.3 **Concentration**

11.3.1 Analytical results as measured from the instrument are referred to as initial results.

11.3.2 Initial results, and any additional dilution (in conjunction with information from the preparation bench sheet), are uploaded into the data system for final result calculation.

11.3.3 Alternatively, final results can be calculated as follows.

$$\text{fResults (ppm)} = \text{iResults} \times \text{DF} \times \frac{\text{iSample (mL or mg)}}{\text{fSample}}$$

Where:

fResults (ppm) = final results (mg/L or mg/Kg)  
DF = additional Dilution Factor  
iSample = initial sample volume (mL) or weight (g)  
fSample = final sample digestate volume (mL)

11.3.4 Results calculated from filter or wipe for air sampling (ug/m<sup>3</sup>):

$$\text{calResults (ug/m}^3\text{)} = \frac{\text{fResults (ug/filter)}}{\text{L (m}^3\text{)}}$$

Where:

calResults (ug/ m<sup>3</sup>) = calculated results for air sampling (ug/ m<sup>3</sup>)  
fResults (ug/filter) = final results for filter or wipes (ug/filter or wipe)  
L (m<sup>3</sup>) = volume of air used in air sampling (m<sup>3</sup>)

## 11.4 **Calculation of IEC offset**

IEC corrections factors are the ratios of the apparent concentration for each analyte to the concentration measured for the IEC reference element. The IEC table is typically done, and saved every six months by referring to instrument software manual (e.g. Winlab32 software guide).

On a daily basis, the automatically calculated and predetermined IEC factors from the table can be offset by a manual calculation, if needed, to improve the accuracy in analyzing a solution of a known concentration. For example, a reference solution of an interfering solution, 180 ppm Fe, yields an apparent concentration of -0.0123 ppm As and results in 186.6 ppm Fe. The IEC offset is calculated as:

$$\frac{-0.0123 \text{ Asppm}}{186.6 \text{ Feppm}} \times 1000 = -0.06592 \text{ ppbAs} / \text{ ppmFe}$$

This result (positive or negative) is then algebraically added to the predetermined factor in the IEC table, supposedly equal to 0.19372:

$$0.19372 - 0.06592 = 0.13077$$

The newly calculated result, 0.13077, is updated in the IEC table for the interferent, Fe, and on the element, As. The IEC is saved as a new file and/or printed out for documentation.

## **12.0 METHOD PERFORMANCE**

### **12.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure as described in laboratory's SOP, IR-QA-MDL. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains annual MDL studies and verifications for 200.7/6010B analyses. MDL analyses must also be performed before sample analysis if any major (non-routine) changes have been made to the instrumentation of a specific ICP.

### **12.2 Instrument Detection Limit (IDL)**

The instrument detection limit (IDL) is the lowest concentration that can be detected for a given analyte on a specific instrument independent of matrix and sample preparation. It is used to determine instrument sensitivity as opposed to the method sensitivity for which the MDL is used. IDLs reflect a statistical value for instrument sensitivity and may not be achievable in actual samples. The laboratory maintains semiannual IDL studies for each instrument. These studies are compared to the MDLs and should be at or below the MDL levels.

### **12.3 Demonstration of Capabilities**

Every analyst must perform an Initial Demonstration of Capability (IDOC) before performing analyses on any client samples.

An IDOC consists of 4 consecutive LCS samples at 1 to 4 times the RL with an average recovery and RSD within laboratory acceptance limits

An on-going DOC must be performed annually. An ODOC can be 4 consecutive LCSs at mid-level or a passing PT.

### **12.4 Training Requirements**

The analyst must have documented training, including reading of the SOP and source methods, conducted by the department manager, senior chemist, or other analyst with training, documentation, and a passing DOC.

## **13.0 POLLUTION CONTROL**

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, and preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in the "Waste Management and Pollution Prevention" section of the Corporate Environmental Health and Safety Manual (CW-E-M-001).

## 14.0 WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the laboratory's Waste Disposal SOP (IR-EHS-WASTE). The following waste streams are produced when this method is carried out:

- **Metals digestates** (50 ml polytubes). Once the samples have been analyzed, they are stored on in the metal shelves for 30 - 60 days. After the 30 -60 days, analysts transfer the digestates to the main waste storage area. Sample archive technicians will store this waste on the shelves designated for metals for another 30-60 days. After this time, Sample archive technicians will bulk this waste as nitric acid waste w/RCRA metals.
- **Acid Waste** (Hydrochloric, Nitric Acid). This waste is generated by ICP /ICP-MS instruments. This waste is collected behind the instruments into 4 gal Carboy satellite container. Analysts in the metals department remove the individual Carboy satellite containers and the 2.5 L waste bottles from the metals area to the main waste storage area twice a week. Analysts will bulk this waste with the nitric acid w/RCRA metals.
- **Unused standards or reagents.** If the standard or reagent is hazardous and can not be collected with one of the waste streams generated in the method, then the analysts and technicians will take this standard or reagent and place it on the shelves labeled "hazardous waste" in the main waste storage area. The waste material must be labeled with the words "Hazardous Waste", contents and the date taken to the waste storage area. The waste material will be lab packed (example: mercury standard).
- If the waste material can be collected in the satellite waste container for one of the waste streams of the method, then pour the standard in the right satellite container, rinse the original container, and collect the rinsate in the satellite container. The original container can be placed in the regular trash. (Example, buffer solutions pH 4). Sample archive technicians will bulk this waste with the nitric acid waste w/RCRA metals.

## 15.0 REFERENCES / CROSS-REFERENCES

15.1 **Reference 1:** EPA Method 200.7, EMMC Version, Revision 4.4 May 1994

15.2 **Reference 2:** EPA SW-846 Final Update III, December 1996, EPA Method 6010B.

## 16.0 METHOD MODIFICATIONS

Item	Method	Modification
1	200.7 (9.3.1)	ICB/CCB acceptance criteria modified from 2.2 x MDL or >10% of any sample concentration, whichever is greater, to <±RL for samples ≥ 2x RL and ≤ ±½ RL (or <MDL, whichever is greater) for samples <2x RL.
2	200.7 (1.6)	Samples containing Silver are diluted only when the concentration exceeds the LCS level of 0.25 ppm rather than 0.1 ppm. Historical laboratory data (filed with SOP*) indicates that effective silver recoveries can still be attained

Item	Method	Modification
		at this level
3	200.7 (9.3.4)	The RSD of two replicates of the ICV has been set at 5%. The method only states the RSD be <3% if 4 or more replicates are analyzed

\*SOP support document "IR-MET-ICP\_r0-Ag LCS Recoveries\_12mar2009"

## 17.0 ATTACHMENTS

- 17.1 **Attachment 1a:** Analysis Information for 200.7
- 17.2 **Attachment 1b:** Analysis Information for 6010B - Water
- 17.3 **Attachment 1c:** Analysis Information for 6010B - Soil
- 17.4 **Attachment 2:** Data Review Checklist
- 17.5 **Attachment 3:** Daily ICP Standards IDs

## 18.0 REVISION HISTORY

### 18.1 Revision 0, dated 13 March 2009

- This revision supersedes ICP.SOP, revision 0 (06/15/07)
- Prepared by TV

### 18.2 Revision 1, dated 01 September 2010

- This revision supersedes IR-WET-ICP-r0, revision 0 (03/13/09)
- Added table of element wavelengths.
- Revised Sample Preparation.
- Revised Daily Run Sequence
- Changed blank acceptance criteria to <RL
- Addition of analysis for bismuth, gallium and scandium.
- Revised Demonstration of Capabilities.
- Addition of Instrument Detection Limit.
- Revised by DT and LH.

### 18.3 Revision 2, dated 23 October 2011

- This revision supersedes IR-WET-ICP, revision 1 (09/01/2010)
- Added Soil samples Special Low Level Preparation.
- Added Soil samples digested per EPA 3051A Preparation.
- Added Cd, Mn and Ni to the Routinely Analyzed Element list.
- Revised Instrument Initialization.
- Revised by DT and LH.

### 18.4 Revision 3, dated 18 January 2013

- This revision supersedes IR-WET-ICP, revision 2 (10/28/2011)
- Added Tungsten to Special Request Elements list.
- Revised 200.7 %RSD requirements for ICV.
- Revised ICSA/ICSAB section.
- Revised Demonstration of Capabilities.

- Revised Waste Management section.
- Added OCSD Sample Preparation Procedure
- Added Daily ICP Standards IDs
- Revised by DT, DD, FR and LH.

Uncontrolled Document

**Attachment 1a**  
**Analysis Information (200.7)**

<b>TestAmerica Irvine</b>							<b>10/09/2012</b>	
<b>Analytical Method Information</b>								
<b>Analyte</b>	<b>MDL</b>	<b>Reporting Limit</b>	<b>Surrogate %R</b>	<b>Duplicate RPD</b>	<b>Matrix Spike %R</b>	<b>RPD</b>	<b>Blank Spike / LCS %R</b>	<b>RPD</b>
<b>200.7-ICP (Full List) in Water (EPA 200.7)</b>								
<b>Preservation:4 C, HNO3</b>								
<b>Container:500 mL Poly</b>								
<b>Amount Required:500 ml</b>								
<b>Hold Time:180 days</b>								
Aluminum	0.040	0.050 mg/l			70 - 130	20	85 - 115	20
Antimony	0.0092	0.010 mg/l			70 - 130	20	85 - 115	20
Arsenic	0.0070	0.010 mg/l			70 - 130	20	85 - 115	20
Barium	0.0060	0.010 mg/l			70 - 130	20	85 - 115	20
Beryllium	0.00090	0.0020 mg/l			70 - 130	20	85 - 115	20
Boron	0.020	0.050 mg/l			70 - 130	20	85 - 115	20
Cadmium	0.0020	0.0050 mg/l			70 - 130	20	85 - 115	20
Calcium	0.050	0.10 mg/l			70 - 130	20	85 - 115	20
Chromium	0.0020	0.0050 mg/l			70 - 130	20	85 - 115	20
Cobalt	0.0020	0.010 mg/l			70 - 130	20	85 - 115	20
Copper	0.0030	0.010 mg/l			70 - 130	20	85 - 115	20
Iron	0.015	0.050 mg/l			70 - 130	20	85 - 115	20
Lead	0.0030	0.0050 mg/l			70 - 130	20	85 - 115	20
Lithium	0.030	0.050 mg/l			70 - 130	20	85 - 115	20
Magnesium	0.012	0.020 mg/l			70 - 130	20	85 - 115	20
Manganese	0.0070	0.020 mg/l			70 - 130	20	85 - 115	20
Molybdenum	0.0020	0.020 mg/l			70 - 130	20	85 - 115	20
Nickel	0.0020	0.010 mg/l			70 - 130	20	85 - 115	20
Phosphorus	0.020	0.50 mg/l			70 - 130	20	85 - 115	20
Platinum	0.068	1.0 mg/l			70 - 130	20	85 - 115	20
Potassium	0.37	0.50 mg/l			70 - 130	20	85 - 115	20
Selenium	0.0080	0.010 mg/l			70 - 130	20	85 - 115	20
Silicon	0.013	0.050 mg/l			70 - 130	20	85 - 115	20
Silver	0.0060	0.010 mg/l			70 - 130	20	85 - 115	20
Sodium	0.19	0.50 mg/l			70 - 130	20	85 - 115	20
Strontium	0.0050	0.020 mg/l			70 - 130	20	85 - 115	20
Thallium	0.0080	0.010 mg/l			70 - 130	20	85 - 115	20
Tin	0.012	0.10 mg/l			70 - 130	20	85 - 115	20
Titanium	0.0020	0.0050 mg/l			70 - 130	20	85 - 115	20
Tungsten	0.50	1.0 mg/l			70 - 130	20	85 - 115	20
Vanadium	0.0030	0.010 mg/l			70 - 130	20	85 - 115	20
Zinc	0.0060	0.020 mg/l			70 - 130	20	85 - 115	20

**Attachment 1b**  
**Analysis Information (6010B-Water)**

		<b>TestAmerica Irvine</b>				<b>10/09/2012</b>	
<b>Analytical Method Information</b>							
<b>Analyte</b>	<b>MDL</b>	<b>Reporting Limit</b>	<b>Surrogate %R</b>	<b>Duplicate RPD</b>	<b>Matrix Spike</b>		<b>Blank Spike / LCS</b>
					<b>%R</b>	<b>RPD</b>	<b>%R RPD</b>
<b>6010B-ICP (Full List) in Water (EPA 6010B)</b>							
<b>Preservation:4 C, HNO3</b>							
<b>Container:500 mL Poly</b>							
<b>Amount Required:500 ml</b>							
<b>Hold Time:180 days</b>							
Aluminum	0.040	0.050 mg/l			75 - 125	20	80 - 120 20
Antimony	0.0092	0.010 mg/l			75 - 125	20	80 - 120 20
Arsenic	0.0070	0.010 mg/l			75 - 125	20	80 - 120 20
Barium	0.0060	0.010 mg/l			75 - 125	20	80 - 120 20
Beryllium	0.00090	0.0040 mg/l			75 - 125	20	80 - 120 20
Boron	0.020	0.050 mg/l			75 - 125	20	80 - 120 20
Cadmium	0.0020	0.0050 mg/l			75 - 125	20	80 - 120 20
Calcium	0.050	0.10 mg/l			75 - 125	20	80 - 120 20
Chromium	0.0020	0.0050 mg/l			75 - 125	20	80 - 120 20
Cobalt	0.0020	0.010 mg/l			75 - 125	20	80 - 120 20
Copper	0.0030	0.010 mg/l			75 - 125	20	80 - 120 20
Iron	0.015	0.040 mg/l			75 - 125	20	80 - 120 20
Lead	0.0030	0.0050 mg/l			75 - 125	20	80 - 120 20
Lithium	0.030	0.050 mg/l			75 - 125	20	80 - 120 20
Magnesium	0.012	0.020 mg/l			75 - 125	20	80 - 120 20
Manganese	0.0070	0.020 mg/l			75 - 125	20	80 - 120 20
Molybdenum	0.0020	0.020 mg/l			75 - 125	20	80 - 120 20
Nickel	0.0020	0.010 mg/l			75 - 125	20	80 - 120 20
Phosphorus	0.020	0.50 mg/l			75 - 125	20	80 - 120 20
Platinum	0.068	1.0 mg/l			75 - 125	20	80 - 120 20
Potassium	0.37	0.50 mg/l			75 - 125	20	80 - 120 20
Selenium	0.0080	0.010 mg/l			75 - 125	20	80 - 120 20
Silicon	0.013	0.050 mg/l			70 - 130	20	85 - 115 20
Silver	0.0060	0.010 mg/l			75 - 125	20	80 - 120 20
Sodium	0.19	0.50 mg/l			75 - 125	20	80 - 120 20
Strontium	0.0050	0.020 mg/l			75 - 125	20	80 - 120 20
Thallium	0.0080	0.010 mg/l			75 - 125	20	80 - 120 20
Tin	0.012	0.10 mg/l			75 - 125	20	80 - 120 20
Titanium	0.0020	0.0050 mg/l			75 - 125	20	80 - 120 20
Tungsten	0.50	1.0 mg/l			70 - 130	20	85 - 115 20
Vanadium	0.0030	0.010 mg/l			75 - 125	20	80 - 120 20
Zinc	0.0060	0.020 mg/l			75 - 125	20	80 - 120 20
Zirconium	0.012	1.0 mg/l			75 - 125	20	80 - 120 20
Calcium as CaCO3	0.12	0.25 mg/l			75 - 125	20	80 - 120 20
Magnesium as CaCO3	0.050	0.082 mg/l			75 - 125	20	80 - 120 20



**Attachment 1c**  
**Analysis Information (6010B-SOIL)**

<b>TestAmerica Irvine</b>								<b>10/09/2012</b>
<b>Analytical Method Information</b>								
<b>Analyte</b>	<b>MDL</b>	<b>Reporting Limit</b>	<b>Surrogate %R</b>	<b>Duplicate RPD</b>	<b>Matrix Spike %R</b>	<b>Matrix Spike RPD</b>	<b>Blank Spike / LCS %R</b>	<b>Blank Spike / LCS RPD</b>
<b>6010B-ICP (Full List) in Soil (EPA 6010B)</b>								
<b>Preservation: 4 C, Cool</b>								
<b>Container: 4 oz Jar/Brass Sleeve</b>		<b>Amount Required: 100 g</b>			<b>Hold Time: 180 days</b>			
Aluminum	5.0	10 mg/kg			75 - 125	20	80 - 120	20
Antimony	1.15	10 mg/kg			75 - 125	20	80 - 120	20
Arsenic	0.81	2.0 mg/kg			75 - 125	20	80 - 120	20
Barium	0.80	1.0 mg/kg			75 - 125	20	80 - 120	20
Beryllium	0.20	0.50 mg/kg			75 - 125	20	80 - 120	20
Boron	2.1	5.0 mg/kg			75 - 125	20	80 - 120	20
Cadmium	0.20	0.50 mg/kg			75 - 125	20	80 - 120	20
Calcium	6.2	15 mg/kg			75 - 125	20	80 - 120	20
Chromium	0.30	1.0 mg/kg			75 - 125	20	80 - 120	20
Cobalt	0.30	1.0 mg/kg			75 - 125	20	80 - 120	20
Copper	0.38	2.0 mg/kg			75 - 125	20	80 - 120	20
Iron	1.5	5.0 mg/kg			75 - 125	20	80 - 120	20
Lead	0.40	2.0 mg/kg			75 - 125	20	80 - 120	20
Lithium	3.8	5.0 mg/kg			75 - 125	20	80 - 120	20
Magnesium	1.0	10 mg/kg			75 - 125	20	80 - 120	20
Manganese	0.80	1.0 mg/kg			75 - 125	20	80 - 120	20
Molybdenum	0.20	2.0 mg/kg			75 - 125	20	80 - 120	20
Nickel	0.20	2.0 mg/kg			75 - 125	20	80 - 120	20
Phosphorus	2.5	50 mg/kg			75 - 125	20	80 - 120	20
Potassium	19	50 mg/kg			75 - 125	20	80 - 120	20
Selenium	1.0	2.0 mg/kg			75 - 125	20	80 - 120	20
Silicon	1.6	10 mg/kg			75 - 125	20	80 - 120	20
Silver	0.80	1.0 mg/kg			75 - 125	20	80 - 120	20
Sodium	24	50 mg/kg			75 - 125	20	80 - 120	20
Strontium	0.70	5.0 mg/kg			75 - 125	20	80 - 120	20
Thallium	0.80	10 mg/kg			75 - 125	20	80 - 120	20
Tin	1.3	10 mg/kg			75 - 125	20	80 - 120	20
Titanium	0.20	2.0 mg/kg			75 - 125	20	80 - 120	20
Vanadium	0.30	1.0 mg/kg			75 - 125	20	80 - 120	20
Zinc	0.50	5.0 mg/kg			75 - 125	20	80 - 120	20
Zirconium	1.5	25 mg/kg			75 - 125	20	80 - 120	20

**Attachment 2**  
**Data Review Checklist**

**DATA CHECKLIST**  
**EPA 6010B / 200.7 – Trace Metals by ICP**

Analyst: _____	2 <sup>nd</sup> Level Review: _____
Analysis Date: _____	Date: _____
ICP #: _____	Method used: <u>EPA 200.7 &amp; 6010B</u>
Data Set #: _____	

<u>Analyst Rev</u>	<u>2<sup>nd</sup> Level Rev</u>	
_____	_____	<b>Calibration:</b> Daily. Minimum Blank + 2 Standards Correlation of linearity $R \geq 0.995$
_____	_____	2 <sup>nd</sup> source <b>ICV:</b> <u>EPA 6010B:</u> %Recovery = 90-110 - %RSD $\leq \pm 5$ <u>EPA 200.7:</u> %Recovery = 95-105 - %RSD $\leq \pm 5$
_____	_____	<b>ICB/CCB:</b> $< \pm RL$ for samples $> 2xRL$ ; $\leq \pm \frac{1}{2} RL$ (or MDL) for samples $\leq 2xRL$
_____	_____	<b>Reporting Limit Check:</b> For drinking water, at the RL (detected for CA, 50-150% for AZ) Special projects have RL check levels and recoveries within required limits
_____	_____	<b>ICSA/ICSAB</b> Before samples (and at the end if required by special project) %Recovery: 80 – 120 (for spiked elements) $\leq \pm 2x RL$ (for interfered elements) Reduced strength ICSA (if needed)
_____	_____	<b>CCV:</b> every 10 aliquots/end of run ; at different concentration than ICV; %R = 90-110
_____	_____	<b>Method blank:</b> $< RL$
_____	_____	<b>LCS:</b> every digestion batch of 20 samples or less <u>EPA 6010B:</u> %Recovery = 80-120 <u>EPA 200.7 :</u> %Recovery = 85-115
_____	_____	<b>MS/MSD:</b> every digestion batch of 20 samples or less for 6010B and 10 samples or less for 200.7 <u>EPA 6010B:</u> 75-125 % Recovery; $\pm 20$ % RPD (or flag accordingly) <u>EPA 200.7 :</u> 70-130 % Recovery; $\pm 20$ % RPD (or flag accordingly)
_____	_____	<b>Internal Standard:</b> <u>EPA200.7:</u> %Recovery = 60 – 140 (Yttrium) <u>EPA 6010B:</u> %Recovery = 60 – 140 (Yttrium)
_____	_____	All samples checked for dilutions, accuracy, precision (RSD if applicable), linear range, IS, RSD, transcription errors and bracketed between compliant CCV/CCB
Comments:		_____
		_____
		_____

G:\DATA\_REV\CHECKLIST\METALS\ICP\ICP\_CHK3.doc  
Rev 12/03/12

**Attachment 3**  
**Daily ICP Standards IDs**

*DAILY ICP STANDARD IDs*

<b>ICP ID#:</b> _____	<b>DATE:</b> _____
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<b>STD 0</b> _____ _____ <b>STD 1</b> _____ _____ <b>STD 2</b> _____ _____ <b>STD 3</b> _____ _____	<b>ICV</b> _____ _____ <b>ICS A</b> _____ _____ <b>ICS AB</b> _____ _____	<b>CRI 2</b> _____ _____ <b>CRI 3</b> _____ _____
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<b>CCV</b> _____ _____ <b>CCB</b> _____ _____
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**ICV Failures:** \_\_\_\_\_

**ICS A Failures:** \_\_\_\_\_

**ICS AB Failures:** \_\_\_\_\_

**CRI Failures:** \_\_\_\_\_

icpstandards\_r0  
rev. 12/10/2012