

**Title: Metals by ICP/MS
EPA Method 200.8 / 6020**

Approvals (Signature/Date):			
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1.0 SCOPE AND APPLICATION

1.1 EPA Method 200.8 is used to determine trace metals in the following matrices: water, drinking water, and wastewater; EPA Method 6020 is used to determine trace metals in ground waters, surface waters, industrial wastes, soil and sludge samples.

1.2 Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation method (EPA 200.2, 3005A, 3050). 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. For any samples submitted under an NPDES permit, digestion is required regardless of turbidity.

1.3 If silver (Ag) analysis is requested, the sample must be digested in all cases. When silver concentrations exceed 0.1ppm the dilution must be performed by redigestion, rather than diluting the digestate at the instrument.

1.4 EPA 200.8 / 6020 may be performed in Collision Cell mode, an instrument analytical technique to remove interferences. **NOTE: Collision cell is not approved for drinking water samples.**

1.5 Drinking Water Maximum Contaminant Levels (MCL) and California Detection Limits for reporting purposes (DLR) are found below. Standard Reporting limits for all metals, attached at the end of this SOP, are either equal or less their corresponding DLRs.

Analyte	DLR (mg/L)	MCL (mg/L)	Analyte	DLR (mg/L)	MCL (mg/L)
Aluminum*	0.050	1.0/0.2	Lead	0.0050	0.015
Antimony	0.006	0.006	Manganese	0.020	0.05
Arsenic	0.002	0.050	Nickel	0.010	0.10
Barium	0.10	1.0	Selenium	0.0050	0.050
Beryllium	0.0010	0.0040	Silver	0.010	0.10
Cadmium	0.0010	0.0050	Thallium	0.0010	0.0020
Chromium (total)	0.010	0.050	Uranium	1 pCi/L	CA: 20 pCi/L Federal: 30 pCi/L
Copper	0.050	1.0	Zinc	0.050	5.0
Iron	0.1	0.3			

* Aluminum has both a primary MCL (1mg/L) and a Secondary MCL (0.2 mg/L)

1.6 See Attachment 5 for quantitation masses and associated internal standard elements.

1.7 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.

1.8 See attached Analysis codes for applicable analyte list and reporting limits.

2.0 SUMMARY OF METHOD

2.1 For non-collision cell analysis, sample material in solution is introduced by pneumatic nebulization into radio frequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a

quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by an electron multiplier detector and the ion information processed by a data handling system.

- 2.2** For collision cell analysis, a reaction system is used to eliminate interferences arising from plasma and sample matrix. This is an instrument mode employing Helium or Hydrogen so side reactions that would create unpredictable, new interferences are eliminated when analyzing trace metals on the ICP-MS.

3.0 DEFINITIONS

There are no specific definitions associated with this test. See the laboratory QA manual, EPA Method 200.8, and EPA Method 6020 for general definitions.

4.0 INTERFERENCES

- 4.1** Isobaric interferences occur when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., Mo 98 and Ru 98). Most commonly used corrections are already present as default interference equations in the instrument software.
- 4.2** Carbon species may cause a positive interference chromium signal at mass 52 due to the formation of Ar^{40} and C^{12} . The level of interference is dependent upon the concentration of available carbon species. Digestion of the sample will remove the volatile organics through heating and remove the inorganic carbon by conversion to CO_2 . Semi-volatile organics will be at least partially oxidized by the use of HNO_3 and heat.
- 4.3** Physical interferences are effects associated with the sample nebulization and transport process. 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. Physical interference effects can be minimized through the use of a peristaltic pump and internal standard.
- 4.4** Molecular interferences are effects associated with the sample nebulization and transport process. 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. Physical interference effects can be minimized through the use of a peristaltic pump, internal standard, and dilution.
- 4.5** Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Optimizing the rinse time between samples and using a rinse solution with the proper acid strength can minimize memory effects. 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.
- 4.6** For collision cell, inert gas (Helium or Hydrogen) introduced into the cell collides with the interfering ions with larger diameters, reducing their kinetic energy so they can be removed through Kinetic Energy Discrimination.

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 material, operations and equipment. This SOP does not purport to address all of the safety

problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Personal Protective Equipment Required: Safety Glasses, Labcoat, Nitrile Gloves.

5.2 Primary Materials Used

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

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 Mass Spectroscopy (ICP-MS) - Model Elan 6100 and Agilent 7700
- 6.1.2 Autosampler – Cetac ASX-500 with ADX-500 or equivalent
- 6.1.3 Autosampler- ESI SC-4 DX Fast

6.2 Supplies

- 6.2.1 Sample tube racks
- 6.2.2 15 mL sample tubes
- 6.2.3 Pipettes and pipettors
- 6.2.4 Pipettor tips
- 6.2.5 Small plastic containers
- 6.2.6 50 mL graduated sample tubes

- 6.2.7 Plastic Erlenmeyer flasks
- 6.2.8 100 mL glass beakers
- 6.2.9 50 mL centrifuge tubes
- 6.2.10 100 mL volumetric flasks, class A.
- 6.2.11 500 mL volumetric flasks, class A.

7.0 REAGENTS AND STANDARDS

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

7.1 Reagents

- 7.1.1 Argon, industrial grade
- 7.1.2 Concentrated Nitric Acid (trace metal grade)
- 7.1.3 Concentrated Hydrochloric acid (trace metal grade)
- 7.1.4 Laboratory Reagent Grade Water (RGW)
- 7.1.5 Helium, industrial grade

7.2 Standards

- 7.2.1 SPEX Metals Stock Standards (or equivalent): 10 ppm
- 7.2.2 Accustandard Metals Stock Standards (or equivalent): 10 ppm
- 7.2.3 Single element internal standard solutions (Ge, Sc, Tb, In, Li, Y, and Bi):1000ppm
- 7.2.4 O2si tuning solution (or equivalent): 10 ppm
- 7.2.5 Agilent PA tuning 1 solution
- 7.2.6 Agilent PA tuning 2 solution
- 7.2.7 O2si Interference Check Standard A (or equivalent): 10000ppm Cl, 2000ppm C, 1000ppm Al, Fe, Mg, Ca, Na, K, P, S and 20ppm Ti, Mo.
- 7.2.8 O2Si CRI standard

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, 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
Water	Polyethylene bottle	500 mL	Preserved with nitric acid to a pH<2	6 months	40 CFR Part 136.3
Soil	Glass Jar or Sampling Sleeve	4 oz	None	6 months	EPA SW-846

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, in order to verify the failure wasn't due to a physical or mechanical problem

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 at \geq RL.

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

- The samples are not detected (ND).
- The sample result is $> 10x$ the blank level.

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

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

9.1.2 Laboratory Control Sample (LCS).

Prepare and analyze a laboratory control sample (LCS) for every batch of 20 samples or less per matrix. The recovery must be within **85-115% for EPA 200.8** and **80-120% for EPA 6020**.

If the LCS is outside of the limit for a requested metal, re-prepare and re-analyze 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 non-conformance memo (NCM) written.
- All positive samples must be re-analyzed with the LCS if the LCS is re-analyzed.

9.1.3 LCS Duplicate (LCSD)

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

9.1.4 Samples

All samples and QC are analyzed with three replicate integrations.

9.1.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

- For 200.8: Analyze a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) for every batch of 10 samples or less per matrix.
- For 6020: Analyze a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) for every batch of 20 samples or less per matrix.
- A typical batch of 10 (or 20) samples will include MB, LCS, MS1, MSD1, MS2 and MSD2
- The LCS spike solution is used to spike the MS/MSD.
- The MS and MSD recoveries must be within **70-130% for EPA Method 200.8** or **75-125% for EPA Method 6020** of the actual value and the RPD must be within 20%.

9.1.6 Dilution Test

As specified in EPA 200.8, a dilution test is not required if an internal standard calibration is used. However, if required for specific client projects, the following procedures are followed:

- If the analyte concentration is sufficiently high (a factor of at least 10 times above the RL after dilution or 50 times without dilution), an analysis of a 1:5 dilution must agree within $\pm 10\%$ of the original determination.
- If the two samples differ by more than $\pm 10\%$, chemical or physical interference effect must be suspected.
- If the regular sample is reported at a dilution, then the diluted sample should be performed at 5 times that dilution (e.g., if reporting at 5X, the dilution test samples should be run at 25X)
- One dilution test must be included for each 20 samples (or less) of each batch.

9.1.7 Post Digestion Spike for 6020

If a 6020 MS and/or MSD recovery fails, a Post Digestion Spike must be performed. A spike is added to portion of a sample or its dilution and should have a recovery of within **75 to 125 %**. The spike addition should be based on the concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, the sample must be diluted and re-analyzed to compensate for the matrix effect. Results must agree to within 10% of the original determination.

9.1.8 Method of Standard Additions (MSA)

The method of standard additions is a special case of the post digestion spike and may be a special project requirement. A spike is added to a portion of sample, generally at 50%, 100%, and 150% of the native sample concentration. These spiked samples are analyzed and their known concentration is plotted against the measured concentration using least-squares linear regression. The intercept of this regression is the MSA result.

9.1.9 Method Reporting Limit (MRL) for drinking water samples

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:

- If the MRL is still below the acceptance limit, the affected samples must be re-digested and re-analyzed.
- If the MRL is above the acceptance limits and samples are ND or greater than 10X the level in the MB, the results may be reported. The results must be flagged and a non-conformance memo (NCM) written.
- All positive samples must be re-analyzed with the MRL if the MRL is re-analyzed.

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 any batch QC or client samples are analyzed.

9.2.1 Initial Calibration Verification (ICV)

Immediately after the initial calibration, analyze the secondary source. Verify that that its recovery is within **90-110%** and the RSD between replicate exposures $\leq 10\%$.

- If the recovery for any requested analyte varies from the expected values by more than $\pm 10\%$ or the RSD is $> 10\%$, re-analyze the ICV using a freshly made standard.
- If the ICV is still below the acceptance limit (90%) for the requested analyte, re-calibrate and re-analyzed the ICV.
- If the ICV is above the acceptance limits (110%) and samples are ND, the results may be flagged and a non-conformance memo (NCM) written.

9.2.2 Initial Calibration Blank (ICB)

Analyze the initial calibration blank (ICB) immediately after the ICV. The ICB should be:

- $< \pm RL$ for samples $\geq 2x RL$ or
- $\leq \pm \frac{1}{2} RL$ or MDL (whichever is greater) for samples $< 2x RL$

If any element exceeds the limit

- Check to see if it is a requested metal; if it is not required the analysis can proceed as is.
- If the element is required, reanalyze the ICB and if it is still out of limits, re-calibrate and re-analyze ICV and ICB.
- Reanalysis is not required if the sample results are $> 10x$ the blank result or the samples are not detected (ND).

9.2.3 Interference Check Solutions (ICSA/ICSAB) - Required for EPA method 6020 only

Analyze the interference check solutions (ICSA and ICSAB) immediately after the ICB to verify the inter-element corrections function properly.

Target Element	ICSA Requirement (ug/L)	ICSAB Requirement (%Difference from True)
As	≤5	±30
Cd	≤5	±30
Cr	≤5	±30
Co	≤5	±30
Cu	≤5	±30
Mn	≤5	±30
Ni	≤5	±30
Ag	≤5	±30
Zn	≤15	±50

If any analyte is outside a limit, then:

- Dilute the ICSA to a level that does not cause interference. If the interfering analyte (e.g. Fe) is present in a sample greater than its concentration in ICSA, dilute the sample so the interfering element concentration is below the ICSA concentration. OR
- Calculate the maximum concentration of the interfering element that can be present and not have an adverse effect on the sample results.
 [e.g. ICSA - Fe = 190mg/L, Se = 0.008mg/L; RL=0.005 (190/0.008) x 0.005= 118 mg/L Fe. If Fe exceeds this level in any sample, the sample must re-analyzed at a dilution].

Analyze the interference check solutions (ICSA and ICSAB) at the beginning of an analytical run or once every 12 hours, whichever is more frequent.

9.2.4 Internal Standard

Internal standard is added to every injection using instrument peristaltic pump.

The internal standard intensities are required to meet the following criteria:

Sample Type	EPA 200.8	EPA 6020
ICV & ICB	±20% of ICAL Standard	±20% of ICAL Standard
Batch QC	60-125%	30-120%
Client Samples	60-125%	30-120%

- If the ICV and the ICB do not meet this requirement, investigate the source of the problem before proceeding.
- If the batch QC samples do not meet these requirements, rerun once and recalibrate if necessary.
- Monitor the internal standard intensity in samples. If these criteria are not meet, dilute the sample and reanalyze. Repeat the dilution process until the IS meets the criteria.

9.2.5 Continuing Calibration Verification (CCV)

Verify the calibration curve by analyzing a CCV after the analysis of every batch of 10 samples. The recovery of the CCV must be between **90% - 110%** of the expected value and the RSD between replicate exposures **≤10%**. If the CCV is outside of acceptance limits:

- Re-prepare the CCV and re-analyze once.
- If it is acceptable, continue with the next 10 samples.
- If it is still unacceptable, re-calibrate the instrument and re-analyze the previous 10 samples.
- If a CCV fails during an overnight or unattended run, re-analyze all samples not bracketed by acceptable CCVs.
- If the CCV is out high, any ND samples may be reported.

9.2.6 Continuing Calibration Blank (CCB)

Follow every CCV with a CCB. The QC criteria for CCB are the same for ICB.

Analyze a continuing calibration blank (CCB) immediately after every CCV. The CCB should be:

- $< \pm RL$ for samples $\geq 2x RL$ or
- $\leq \pm \frac{1}{2} RL$ or MDL (whichever is greater) for samples $< 2x RL$

If any element exceeds the limit:

- check to see if it is a requested metal; if it is not required the analysis can proceed as is.
- If CCB is still out for that element, all samples that are not detected (ND) or detected higher than 10x the contaminated CCB can be reported.
- If the element is required, reanalyze the CCB and if it is still out of limits, All positive samples bracketed by the failed CCB must be re-analyzed.

9.2.7 Calibration Acceptance Summary

Refer to the "Calibration Curves" SOP and the "Selection of Calibration Points" SOP for more information on calibrating the instrument.

Prepare and analyze a calibration curve daily for each element with 3 standards and a blank. The Correlation Coefficient must be ≥ 0.995 or the calibration must be repeated.

9.2.8 Linear Dynamic Range (LDR)

- Analyze once a year or whenever there is a change in analytical performance caused by either a change in instrument hardware or operating conditions. The upper limit of the linear calibration range should be established for each analyte by determining the signal responses from a minimum of three different concentration standards; the highest standard is close to the upper limit of the linear range.
- For every element, calibrate the instrument as normal. Analyze standards for each element at two additional concentrations, the highest concentration being defined as the

Linear Dynamic Range (LDR) of the instrument. Results of each of the standard must be within $\pm 10\%$ of the true value in order for the calibration range to be established.

- If $\pm 10\%$ cannot be achieved, a fresh standard may be prepared and reanalyzed and/or the instrument must be recalibrated and/or a lower standard may be used. The standard levels for the LDR will vary by element and must be documented after the range has been established.
- Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and analyzed.

9.2.9 Instrument Detection Limits (IDLs)

- Instrument Detection Limit studies can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of reagent blank solutions.
- IDLs should be determined at least every three months.

10.0 PROCEDURE

10.1 Reagent Preparation

10.1.1 Acidified Reagent Grade Water:

- Acidified RGW is used in the preparation of all subsequent standards.
- Acidified RGW is also used as rinse solution.
- Prepare the 1% (v/v) HNO₃ and 0.5% (v/v) HCl by adding 10 mL HNO₃ and 5 mL of HCL to 1L RGW in a 1L bottle.

10.2 Standard Preparation

All working standards are prepared daily in 100 mL class A volumetric flasks. Internal Standard is added at instrument by peristaltic pump.

Source	Blank	S1 0.2 ppb	S2 1 ppb	S3 10 ppb	S4 100 ppb	ICV 25 ppb	CCV 50 ppb	ICB/CCB
First Source Std					1 mL		2.5 mL	
S2		2 mL	10 mL					
S3				10 mL				
Second Source Std						250 μ L		
Final Volume with Acidified Reagent Grade Water	100 mL	100 mL	100 mL	100 mL (45 mL*)	100 mL (45 mL*)	100 mL	500 mL	100 mL

*Final volume for S2 and S3 are 45 mL since 5 mL of standard is used for the preceding standard

10.2.1 Standard 1 Concentration (S1): 1 ppb Metals, 10 ppb Fe, 25 ppb K, Mg, Na, 50 ppb Ca.

10.2.2 Standard 2 Concentration (S2): 10 ppb Metals, 100 ppb Fe, 250 ppb K, Mg, Na, 500 ppb Ca.

10.2.3 Standard 3 Concentration (S3): 100 ppb Metals, 1000 ppb Fe, 2500 ppb K, Mg, Na, 5000 ppb Ca.

10.2.4 Calibration Blank, ICB, CCB: acidified Reagent Grade water

10.2.5 Second source ICV: purchased

10.2.6 Interference Check Solution A (ICSA): 1000 ppm of Cl; 200 ppm of C; 100 ppm Al, Ca, Fe, Mg, P, K, Na, S and 2ppm of Mo, Ti. Prepare the ICSA by adding 1 mL of ICSA standard to 9 mL acidified Reagent Grade water in a 15mL sample tube. Prepare daily.

10.2.7 Interference Check Solution AB (ICSAB): 1000 ppm of Cl, 200 ppm of C, 100 ppm Al, Ca, Fe, Mg, P, K, Na S, 2 ppm of Mo, Ti; It also contains 20 ppb of each of As, Cd, Cr, Co, Cu, Mn, Ni, Ag, and Zn. Prepare ICSAB by adding 1 mL of ICSA standard, 20 μ L of 10ppm stock standard to 9 mL of acidified Reagent Grade water. Prepare daily.

10.2.8 Reporting Limit Checks

Note: Internal standard is added using instrument peristaltic pump.

Reagent/Standard	0.2 ppb	1.0 ppb	2.0 ppb
100 ppb standard	20 μ L	100 μ L	200 μ L
Acidified Reagent Grade Water to Final Volume	10 mL	10 mL	10 mL

10.2.9 Tuning check solution for both Perkin Elmer ELAN and Agilent EPA Tune Check:

The tuning solution is prepared at 10ppb from the O2Si (or equivalent) tuning solution (10 ppm). Prepare the tuning solution with 1% (v/v) nitric acid in Reagent Grade water. Add 1mL of tuning solution (10ppm) to 1L of 1% (v/v) HNO₃ solution.

10.2.10 1ppb Agilent Tuning Solution

Make a 1:10 dilution of the stock 10ppm Agilent Tune Solution to have 1ppm working standard in 1% (v/v) HNO₃. Add 1mL of 1ppm standard to 1L of 1% (v/v) HNO₃ solution.

10.2.11 Agilent PA Solution

Add 1mL of Agilent PA Solution 1 and 1 mL of Agilent PA Solution 2 to 100mL of 1%HNO₃ and 0.5% HCl solution.

10.2.12 Internal standard solution mix:

For ELAN: The online internal standard solution mix is prepared at 200 ppb (Sc, Tb, In, Ge and Li) from the 1000 ppm single element stock solutions. Add 200 ul of each of the 1000 ppm stock solutions to 1 L of acidified Reagent Grade water.

For Agilent: The online internal standard solution mix is prepared at 1 ppm (Sc, Tb, In, Ge, Y and Li) from the 1000 ppm single element stock solutions. Add 500 ul of each of the 1000 ppm stock solutions to 500mL of acidified Reagent Grade water.

10.2.13 Linear Dynamic Range Standards

Prepare 7 standard solutions as follows: 10ppm is the stock standard

Source	1000 ppb	1500 ppb	2000 ppb	2500 ppb	3000 ppb	3500 ppb	4000 ppb
First Source 10 mg/L std (mls)	1.0	1.5	2.0	2.5	3.0	3.5	4.0
Final Volume with Acidified (RGW) (mls)	10	10	10	10	10	10	10

10.3 Sample Preparation

Batch QC samples (MB, MRL, LCS, MS/MSD) are prepared in the same manner as client samples but are spiked in accordance with the table below.

Container Matrix	QC Type	Matrix	Spike (uL)	Final Volume
Digested Water	MB	Digested RGW, prepared by Metals Prep Group		
	LCS	Digested & spiked RGW, prepared by Metals Prep Group		
	MRL			
	MS/MSD	Digested & spiked source sample, prepared by Metals Prep Group		
Digested Soil/Wipe	MB	PTFE chips or wipe digested in RGW, prepared by Metals Prep Group		
	LCS			
	MRL	PTFE chips or wipe spiked & digested in RGW, prepared by Metals Prep Group		
	MS/MSD	Source sample spiked & digested in RGW, prepared by Metals Prep Group*		
STLC	MB	0.5mL STLC Blank	N/A	10mL with acidified RGW
	LCS	0.5mL STLC Blank	80µL of 10ppm stock standard	10mL with acidified RGW
	MS/MSD	0.5mL source sample	80µL of 10ppm stock standard	10mL with acidified RGW
TCLP/SPLP	MB	Digested TCLP/SPLP Blank, prepared by Metals Prep Group		
	LCS	Digested & spiked TCLP/SPLP Blank, prepared by Metals Prep Group		
	MS/MSD	Digested TCLP/SPLP MS, prepared by Metals Prep Group		
Leachate (DI WET)	MB	DI WET Blank	N/A	10mL DI WET Blank
	LCS	DI WET Blank	80µL of 10ppm stock standard	10mL DI WET Blank
	MS/MSD	Source sample	80µL of 10ppm stock standard	10mL source sample

*MS/MSD not applicable to wipe samples.

NOTE: Internal standard is added using instrument's peristaltic pump.

10.3.1 Digested Water:

- Transfer samples into labeled corresponding centrifuge tubes.

10.3.2 Soil/Wipe

- Soil samples are diluted 20x after digestion and before analysis.
- Pipet 0.5 mL of the sample into a centrifuge tube.
- Add 9.5 mL of acidified Reagent Grade water.

10.3.3 STLC

- STLC samples (after rotation and filtration) are diluted 20x after digestion and before analysis.
- Pipet 0.5 mL of the sample into a centrifuge tube.
- Add 9.5 mL of acidified Reagent Grade water.

10.3.4 TCLP /SPLP

- TCLP / SPLP samples are diluted 10x after digestion and before analysis.
- Pipet 1.0 mL of the sample into a centrifuge tube.
- Add 9.0 mL of acidified Reagent Grade water.

10.3.5 Leachate (DI WET)

- Pour the sample into a 15mL centrifuge tube.

10.4 Instrument Initialization: Pre-calibration routine: instrument tuning and mass calibration. Follow instrument manual for start-up for both ELAN and Agilent.

- Warm-up the instrument for at least 30 minutes for ELAN, 20 minutes for Agilent.
- Analyze the tuning solution 5 times and achieve a relative standard deviation for all analytes of <5%.
- Conduct mass calibration and resolution checks using the tuning solution. Peak width should be 0.700 ± 0.010 amu at 10% peak height.
- Follow the instructions provided by the instrument manufacturer for operating conditions (see attached flow chart). For the following optimizations aspirate the tuning solution (10ppb) containing Ba, Be, Ce, Co, In, Li, Mg, Pb, Rh, Ti, U, Y.
- Run a mass tune. Measured masses should be within 0.05 atomic units of the actual masses.
- Conduct an autolens calibration for the ELAN where the slope of the curve must be positive. When the lens voltage range gets to between 10 and 11, the lens should be cleaned or replaced. Autolens calibration is not needed for the Agilent.
- Then conduct a Daily performance check.

	Perkin-Elmer ELAN		Agilent	
	Mass Tune Requirement (± 0.5 au)	Daily Performance Check	Mass Tune Requirement (± 0.1 au)	Daily Performance Check
He	2.966-3.066			
Mg	23.935-24.035	>20,000 cps	23.9-24.10	>2,000 cp 1/10s
Rh	102.855-102.955	>150,000 cps	102.90-103.10	>15,000 cp 1/10s
Pb	207.927-208.027	>100,000 cps	207.90-208.10	>10,000 cp 1/10s
In		>150,000 cps	114.90-115.10	>15,000 cp 1/10s
CeO/Ce		≤ 0.03 net intensity		$\leq 1\%$
Ba ⁺⁺ /Ba ⁺		≤ 0.03 net intensity		$\leq 3.0\%$
Mass 220		≤ 30 cps		
U			237.90-238.10	

10.5 Calibration

Calibrate the instrument daily using a blank, S1, S2 and S3

10.6 Sample Analysis

10.6.1 Analyze the ICV, ICB at the start of the analytical run.

10.6.2 Analyze ICSA and ICSAB (only required for EPA Method 6020).

10.6.3 Analyze RL Check standards.

10.6.4 Analyze a method blank (MB) and LCS for each batch of 20 samples or less per matrix.

10.6.5 Analyze up to 8 samples after the LCS and MB.

10.6.6 For 200.8: Analyze a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) for every batch of 10 samples or less per matrix.

10.6.7 For 6020: Analyze a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) for every batch of 20 samples or less per matrix.

10.6.8 Analyze a CCV and CCB for every set of 10 client samples and at the end of the analytical run.

10.6.9 Analyze RL Checks at the end (if required by client)

10.6.10 Analyze ICA and ICSAB at the end (if required by client)

A typical daily run sequence is listed below:

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Sample Name	Criteria
Blank	
S1	$r^2 \geq 0.995$
S2	
S3	
ICV	%Rec=90-110; RSD \leq 10
ICB	< \pm RL for samples \geq 2x RL OR $\leq \pm\frac{1}{2}$ RL or MDL (whichever is greater) for samples <2x RL
ICSA*	(As, Cd, Cr, Co, Cu, Mn, Ni, Ag) \leq 5 ug/L, Zn \leq 15ug/L
ICSAB*	(As, Cd, Cr, Co, Cu, Mn, Ni, Ag) \pm 30%, Zn \pm 50%
RL Check standards	%Rec= \square 50
MB1	<RL
LCS1	%Rec=85-115 for 200.8 %Rec=80-120 for 6020
Source sample for MS1/MSD1	RSD \leq 25**
MS1	%Rec=70-130 for 200.8; RPD \leq 20
MSD1	%Rec=75-125 for 6020; RPD \leq 20
Samples	RSD \leq 25 %**
CCV	%Rec=90-110; RSD \leq 10
CCB	< \pm RL for samples \geq 2x RL OR $\leq \pm\frac{1}{2}$ RL or MDL (whichever is greater) for samples <2x RL
Source sample for MS2	RSD \leq 25*
MS2 (200.8 only)	%Rec=70-130; RPD \leq 20
MSD2 (200.8 only)	
CCV	%Rec=90-110; RSD \leq 10
CCB	< \pm RL for samples \geq 2x RL OR $\leq \pm\frac{1}{2}$ RL or MDL (whichever is greater) for samples <2x RL
Samples	RSD \leq 25 %*

Sample Name	Criteria
CCV	%Rec=90-110; RSD≤10
CCB	<±RL for samples ≥2x RL OR ≤ ±½ RL or MDL (whichever is greater) for samples <2x RL
Closing ICSA (if req'd)***	(As, Cd, Cr, Co, Cu, Mn, Ni, Ag) ≤5 ug/L, Zn ≤15 ug/L
Closing ICSAB (if req'd)***	(As, Cd, Cr, Co, Cu, Mn, Ni, Ag) ±30 %, Zn ±50 %
Closing RL check (if req'd)***	%Rec=±50

* Only required for EPA Method 6020

** For sample results >2X the RL, otherwise RSD must be ≤50%. (unless specified otherwise by client)

*** For special project

10.7 Preventative Maintenance

10.7.1 Daily

- Inspect peristaltic pump tubing and replace when needed.
- Inspect waste level and empty when necessary.
- Replace rinse solution.
- Inspect torch and injector for signs of clogging, misalignment, or deposits.
- Clean cones
- Clean or change nebulizer tips.

10.7.2 As Needed

- Clean or replace ion lens.
- Replace O-rings on torch.
- Check gas filters for moisture and replace when necessary.
- Change rough pump oil (about monthly).

10.7.3 Record all performed maintenance in the instrument maintenance logbook.

10.7.4 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 and verification of proper operation into the instrument maintenance logbook.

11.0 CALCULATIONS / DATA REDUCTION

11.1 Accuracy

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

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{known concentration}} \times 100$$

spiked concentration

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 Calibration

$$y = mx + b \qquad C = \frac{(y - b)}{m}$$

Where y is the instrument response ratio

m is the slope

x is the concentration

b is the y-intercept

C is the raw sample concentration (instrument reading)

11.4 Concentration

$$C_f = C_i \times \text{PF} \times \text{DF}$$

Where C_f = Final concentration in $\mu\text{g/L}$

C_i = Concentration in $\mu\text{g/L}$ from instrument

PF = Preparation (digestion) factor

DF = any additional bench Dilution Factor

11.5 Calculation / Reporting of Uranium

Uranium may be reported either as mass ($\mu\text{g/L}$) or activity (pico-Curies per liter [pCi/L]):

$$\mu\text{g/L} \times \text{conversion factor} = \text{pCi/L}$$

conversion factor from mass to activity is 0.67

The CA DLR for uranium is 1 $\mu\text{g/L}$ or 2 pCi/L.

The CA MCL for uranium is 20 pCi/L.

The Federal MCL for uranium is 30 $\mu\text{g/L}$

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 MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 **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.

12.3 **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, 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 kept in the metal shelves for 30 - 60 days. After the 30 -60 days analyst transfer the digestates to the main waste storage area. Sample archive technicians will store this waste on the shelves designated for metals for 30-60 days. After this time Sample archive technicians will bulked this waste as nitric acid waste w/RCRA metals.
- **Acid Waste** (Hydrochloric, Nitric Acid). This waste is generated by ICP /ICP MS machines. This waste is collected behind the instruments into 4 gal Carboy satellite container. Sample archive technicians will bulk this waste with the nitric acid waste w/RCRA metals.
- **Unused standards**. If the standard is hazardous and can not be collected with one of the waste streams generated in the method, it must be labeled as hazardous waste with the date the material became waste. The analyst or technicians take this standard and placed it on the shelves labeled "hazardous waste" in the main waste storage area. The standard will be lab packed (example: mercury standard).
- If the standard 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 EPA Method 200.8, Revision 5.4, May 1994
- 15.2 EPA Method 200.2, Revision 2.8, May 1994
- 15.3 EPA Method 6020 SW-846, Revision 0, September 1994
- 15.4 Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth Edition, EPA, January 2005, EPA 815-R-05-004
- 15.5 CA-Q-S-005, Calibration Curves (General)
- 15.6 CA-T-P-002, Selection Of Calibration Points

16.0 METHOD MODIFICATIONS

Item	Method Ref	Modification
1	EPA 200.8 Sec. 10.2.1	For mass tuning, peak width of approximately 0.70 amu at 10% peak height is calibrated instead of 0.75 amu at 5% peak height for better performance per instrument manufacturer.
2	EPA 200.8 Sec. 7.4.1, 7.6.1	All calibration standards and calibration blank are prepared in 1% (v/v) HNO ₃ and 0.5% (v/v) to match with sample matrix.
3	EPA 200.8 Sec. 7.5	Germanium is used as internal standard in lieu of Yttrium and Bismuth
4	EPA 200.8 Sec 9.3.1	ICB/CCB acceptance criteria modified from 2.2 x MDL or >10% of any sample concentration, whichever is greater, to $\leq \pm RL$ for samples $\geq 2x RL$ and $\leq \pm \frac{1}{2} RL$ (or $< MDL$, whichever is greater) for samples $< 2x RL$.
5	EPA 200.8 6020 Sec 2.	The new technique, collision cell, is added to remove interferences and improve RL due to less dilution.

17.0 ATTACHMENTS

- 17.1 **Attachment 1:** Analysis Information for 200.8
- 17.2 **Attachment 2:** Analysis Information for 6020
- 17.3 **Attachment 3:** Elan Data Review Checklist
- 17.4 **Attachment 4:** Agilent Data Review Checklist
- 17.5 **Attachment 5:** Quantitation Ions and Associated Internal Standards

18.0 REVISION HISTORY

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files.

18.1 **Revision 2, dated 14 August 2012**

- This revision supersedes IR-MET-ICPMS, revision 1 (04/04/2011)
- Removed Qualifier Summary attachment
- Revised Matrix Spike and Matrix Spike Duplicate section
- Added MSA section
- Added Analysis Information for 6020
- Revised data review checklists
- Revised by DT and LH

18.2 Revision 3, dated 30 August 2013

- This revision supersedes IR-MET-ICPMS, revision 2 (08/16/2013)
- Added digested Reporting Limit Verification (MRL) for drinking water samples that required digestion.
- Added Bismuth in the list of internal standards used in the method.
- Added standard 0.2ppb to the calibration curve
- All working standards are prepared in 100 mL class A volumetric flasks
- Added Instrument Detection Limits study
- Added Rubidium, Thorium, Tin and Strontium to the list of elements analyzed.
- Unused standards must be labeled as hazardous waste with the date the material became waste
- Added Wipe Sample Preparation
- Revised by NH, LH and DD

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Attachment 1
Analysis Information for 200.8

TestAmerica Irvine

Analytical Method Information

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	RPD	Blank Spike / LCS %R	RPD
ICP/MS-200.8 in Water (EPA 200.8)								
Preservation: 4 C, HNO ₃								
Container: 500 mL Poly								
Amount Required: 100 ml								
Hold Time: 180 days								
Aluminum	5.0	10 ug/l			70 - 130	20	85 - 115	20
Antimony	0.50	2.0 ug/l			70 - 130	20	85 - 115	20
Arsenic	0.50	1.0 ug/l			70 - 130	20	85 - 115	20
Barium	0.50	1.0 ug/l			70 - 130	20	85 - 115	20
Beryllium	0.25	0.50 ug/l			70 - 130	20	85 - 115	20
Cadmium	0.25	1.0 ug/l			70 - 130	20	85 - 115	20
Chromium	0.50	2.0 ug/l			70 - 130	20	85 - 115	20
Cobalt	0.50	1.0 ug/l			70 - 130	20	85 - 115	20
Copper	0.50	2.0 ug/l			70 - 130	20	85 - 115	20
Iron	8.0	20 ug/l			70 - 130	20	85 - 115	20
Lead	0.50	1.0 ug/l			70 - 130	20	85 - 115	20
Manganese	0.50	1.0 ug/l			70 - 130	20	85 - 115	20
Molybdenum	0.50	2.0 ug/l			70 - 130	20	85 - 115	20
Nickel	0.50	2.0 ug/l			70 - 130	20	85 - 115	20
Selenium	0.50	2.0 ug/l			70 - 130	20	85 - 115	20
Silver	0.50	1.0 ug/l			70 - 130	20	85 - 115	20
Thallium	0.50	1.0 ug/l			70 - 130	20	85 - 115	20
Vanadium	0.50	2.0 ug/l			70 - 130	20	85 - 115	20
Zinc	5.0	20 ug/l			70 - 130	20	85 - 115	20

Attachment 2
Analysis Information for 6020

TestAmerica Irvine									
Analytical Method Information									
Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix Spike %R	Matrix Spike RPD	Blank Spike %R	Blank Spike /LCS RPD	
ICP/MS-6020 in Water (EPA 6020)									
Preservation: 4 C, HNO3									
Container: 500 mL Poly									
Amount Required: 100 ml									
Hold Time: 180 days									
Aluminum	5.0	10 ug/l			75 - 125	20	80 - 120	20	
Antimony	0.50	2.0 ug/l			75 - 125	20	80 - 120	20	
Arsenic	0.50	1.0 ug/l			75 - 125	20	80 - 120	20	
Barium	0.50	1.0 ug/l			75 - 125	20	80 - 120	20	
Beryllium	0.25	0.50 ug/l			75 - 125	20	80 - 120	20	
Cadmium	0.25	1.0 ug/l			75 - 125	20	80 - 120	20	
Chromium	0.50	2.0 ug/l			75 - 125	20	80 - 120	20	
Cobalt	0.50	1.0 ug/l			75 - 125	20	80 - 120	20	
Copper	0.50	2.0 ug/l			75 - 125	20	80 - 120	20	
Iron	8.0	20 ug/l			75 - 125	20	80 - 120	20	
Lead	0.50	1.0 ug/l			75 - 125	20	80 - 120	20	
Manganese	0.50	1.0 ug/l			75 - 125	20	80 - 120	20	
Molybdenum	0.50	2.0 ug/l			75 - 125	20	80 - 120	20	
Nickel	0.50	2.0 ug/l			75 - 125	20	80 - 120	20	
Selenium	0.50	2.0 ug/l			75 - 125	20	80 - 120	20	
Silver	0.50	1.0 ug/l			75 - 125	20	80 - 120	20	
Thallium	0.50	1.0 ug/l			75 - 125	20	80 - 120	20	
Vanadium	0.50	2.0 ug/l			75 - 125	20	80 - 120	20	
Zinc	5.0	20 ug/l			75 - 125	20	80 - 120	20	

Attachment 3
Elan Data Review Checklist

DATA CHECKLIST

EPA 6020 / 200.8 – Trace Metals by ICPMS - ELAN

Analyst: _____ Analysis Date: _____ ICPMS #: _____ Data Set #: _____	2 nd Level Review: _____ Date: _____ Method used: EPA 200.8 _____ 6020 _____
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<u>Analyst Rev</u>	<u>2nd Level Rev</u>
--------------------	---------------------------------

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
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_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Pre-calibration tuning and performance:

Precision: RSD (absolute signals) < 5% from 4 to 5 runs of tuning solution

Mass tune He: 2.966 – 3.066 Rh: 102.855 – 102.955
 Mg: 23.935 – 24.035 Pb: 207.927 – 208.027

Performance In: >150,000 cps CeO/Ce: ≤ 0.03 net intensity
 Rh: >150,000 cps Ba²⁺/Ba: ≤ 0.03 net intensity
 Mg: >20,000 cps Backgrnd: < 30cps at Mass 220
 Pb: >100,000 cps

Daily calibration: 3-point, 3-replicate run, linear and forced through zero. $r \geq 0.995$

2nd source ICV (25ppb) / 1st source CCV (50ppb):

- ICV immediately after calibration, CCV every 10 samples and at the end of analysis
- %REC = 90 – 110
- %RSD ≤ 10

Reporting Limit (RL) Check: 50-150%

ICS: for 6020, run before sample analysis **ICSA** **ICSAB**

- As,Cd,Cr,Co,Cu,Mn,Ni,Ag: ±5ppb ±30%
- Zn: ±15ppb ±50%

Analyze ICSA/ICSAB once every 12 hours

ICB/CCB/MB:

- Run after ICV/CCV
- ICB/CCB: < ±RL for samples ≥ 2x RL or
 ≤ ±½ RL or MDL (whichever is greater) for samples < 2x RL
- MB: < RL

LCS/LFB: every batch of 20 samples or less

- EPA 6020: %REC = 80 – 120 (or in-house)
- EPA 200.8: %REC = 85 – 115

MS/MSD: EPA 6020 : every 20 samples or less
 EPA 200.8: every 10 samples or less

- EPA 6020: %REC = 75 - 125 (or in-house) and %RPD (MS/MSD) ≤ 20
- EPA 200.8: %REC = 70 – 130 and %RPD (MS/MSD) ≤ 20

Post-Digestion Spike (EPA 6020 only): Analyze if MS or MSD fail. %REC = 75 - 125

Dilution Test: (for special project)

Internal Standard:

- EPA 200.8: 60 – 125% (80-120% in ICB/ICV)
- EPA 6020B: 30 – 120% (80-120% in ICB/ICV)

All samples checked for replicates (3), precision (RSD if applicable), accuracy, dilution, linear range (≤90% of LDR), correct bracket (≤10 samples) between compliant CCV/CCB and transcription errors

Comments: _____

Attachment 4
Agilent Data Review Checklist

DATA CHECKLIST	
EPA 6020 / 200.8 – Trace Metals by ICPMS - AGILENT	
Analyst: _____	2 nd Level Review: _____
Analysis Date: _____	Date: _____
ICPMS #: _____	Method used: EPA: 200.8
Data Set #: _____	6020

<u>Analyst Rev</u>	<u>2nd Level Rev</u>	Pre-calibration tuning and performance:
_____	_____	Precision: RSD (absolute signals) < 5% from 4 to 5 runs of tuning solution
_____	_____	Mass tune In: 114.90 – 115.10 Rh: 102.90 – 103.10
_____	_____	Mg: 23.9 – 24.10 Pb: 207.90 – 208.10
_____	_____	U: 237.90 – 238.10
_____	_____	Performance In: >15,000 cp(1/10)s Oxide: <= 1%
_____	_____	Rh: >15,000 cp(1/10)s Ba ²⁺ /Ba ⁺ : <= 3%
_____	_____	Mg: >2,000 cp(1/10)s mz/ RSD: < 10%
_____	_____	Pb: >10,000 cp(1/10)s
_____	_____	Daily calibration: 3-point, 3-replicate run, linear and forced through zero. r ² ≥ 0.995
_____	_____	2nd source ICV (25ppb) / 1st source CCV (50ppb):
_____	_____	• ICV immediately after calibration. CCV every 10 samples and at the end of analysis
_____	_____	• %REC = 90 – 110
_____	_____	• %RSD ≤ 10
_____	_____	Reporting Limit (RL) Check: 50-150%
_____	_____	ICS: for 6020, run before sample analysis
_____	_____	• As,Cd,Cr, Co,Cu,Mn,Ni,Ag: ICSA ±5ppb ICSAB ±30%
_____	_____	• Zn: ±15ppb ±50%
_____	_____	Analyze ISCA/ICSAB once every 12 hours
_____	_____	ICB/CCB/MB:
_____	_____	• Run after ICV/CCV
_____	_____	• ICB/CCB : <=RL for samples ≥2x RL or
_____	_____	<= ±½ RL or MDL (whichever is greater) for samples <2x RL
_____	_____	• MB: <RL
_____	_____	ICS/LFB: every batch of 20 samples or less
_____	_____	• EPA 6020: %REC = 80 – 120 (or in-house)
_____	_____	• EPA 200.8: %REC = 85 – 115
_____	_____	MS/MSD: EPA 6020: every 20 samples or less
_____	_____	EPA 200.8: every 10 samples or less
_____	_____	• EPA 6020: %REC = 75 - 125 (or in-house) and %RPD (MS/MSD) ≤ 20
_____	_____	• EPA 200.8: %REC = 70 – 130 and %RPD (MS/MSD) ≤ 20
_____	_____	Post-Digestion Spike (EPA 6020 only): Analyze if MS or MSD fail. % REC = 75 - 125
_____	_____	Dilution Test: (for special project)
_____	_____	Internal Standard:
_____	_____	• EPA 200.8: 60 – 125% (80-120% in ICB/ICV)
_____	_____	• EPA 6020B: 30 – 120% (80-120% in ICB/ICV)
_____	_____	All samples checked for replicates (3), precision (RSD if applicable), accuracy, dilution, linear range (<=90% of LDR), correct bracket (<=10 samples) between compliant CCV/CCB and transcription errors
_____	_____	Comments: _____
_____	_____	_____

ICPMS_CHKLIST_AGILENT_CHK1.doc
 Rev. 08/14/12

Attachment 5

Quantitation Ions and Associated Internal Standards

Element Name	Quant Ion Mass	Internal Standard
Beryllium (Be)	9	Germanium (Ge) and/or Lithium(Li)
Aluminum (Al)	27	Germanium(Ge) and/or Scandium(Sc)
Vanadium (V)	51	Germanium(Ge) and/or Scandium(Sc)
Chromium (Cr)	52	Germanium(Ge) and/or Scandium(Sc)
Manganese (Mn)	55	Germanium(Ge) and/or Scandium(Sc)
Cobalt (Co)	59	Germanium(Ge) and/or Scandium(Sc)
Nickel (Ni)	60	Germanium(Ge) and/or Scandium(Sc)
Copper (Cu)	63	Germanium(Ge) and/or Scandium(Sc)
Copper (Cu)	65	Germanium(Ge) and/or Scandium(Sc)
Zinc (Zn)	66	Germanium (Ge)
Arsenic (As)	75	Germanium (Ge)
Selenium (Se)	82	Germanium (Ge)
Krypton (Kr)	84	No IS. Monitoring purposes only*
Molybdenum (Mo)	98	Germanium (Ge) and/or Indium(In)
Silver (Ag)	107	Germanium (Ge) and/or Indium(In)
Cadmium (Cd)	114	Germanium (Ge) and/or Indium(In)
Antimony (Sb)	121	Germanium (Ge) and/or Indium(In)
Barium (Ba)	135	Germanium (Ge) and/or Indium(In)
Thallium (Tl)	205	Terbium (Tb) and/or Bismuth (Bi)
Lead (Pb)	208	Terbium (Tb) and/or Bismuth (Bi)
Uranium (U)	238	Terbium (Tb)
Sodium (Na)	23	Scandium (Sc)
Magnesium (Mg)	24	Scandium (Sc)
Potassium (K)	39	Scandium (Sc)
Calcium (Ca)	44	Scandium (Sc)
Iron (Fe)	54	Scandium (Sc)
Rubidium	85	Indium (In)
Thorium	232	Terbium (Tb)
Tin	118	Indium (In)
Strontium	88	Indium (In)
Cesium (Ce)	140	Indium (In)

*Krypton should read > 5000 – 10000 counts as daily monitoring response