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Method 200.8 Determination of Trace Metals in Waters and Wastes by ICP-MS

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1. Scope and Application

- 1.1 Method 200.8 is used to determine trace metals in ground waters, surface waters, drinking waters, wastewaters, tissues, sludges, and soils by ICP-MS. Samples analyzed by this method must be acceptable for analysis by direct aspiration or prepared by EPA method 200.2 or 200.3.

This method is used to analyze the following metals:

<u>Compound</u>	<u>CAS No.</u>
Aluminum	7429-90-5
Antimony	7440-36-0
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Cadmium	7440-43-9
Chromium	7440-47-3
Cobalt	7440-48-4
Copper	7440-50-8
Iron	7439-89-6
Lead	7439-92-1
Manganese	7439-96-5
Molybdenum	7439-98-7
Nickel	7440-02-0
Selenium	7782-49-2
Silver	7440-22-4
Sodium	7440-23-5
Thallium	7440-28-0
Tin	7440-31-5
Vanadium	7440-62-2
Zinc	7440-66-6
Uranium	7440-61-1

- 1.2 Restricted Procedure

This procedure is restricted to use by an analyst experienced in the operation of

Inductively Coupled Plasma Mass Spectrometers. Additionally, the analyst must complete the requirements of the GaEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GaEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

- 2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions. (See SOP reference 13.7)
- 2.2 Interference Check Solution (INCK): A standard used to correct inter-elemental caused by spectral overlaps. Commonly referred to as IEC and SIC.
- 2.3 Instrument Detection Limit (IDL): The concentration equivalent to analyte signal which is equal to 3 times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the same mass.
- 2.4 MDL standard (ML) = Method Detection Limit Spike Standard (MDL_{spike}): An MDL_{spike} is analyzed with every batch of samples and is carried through the same procedure as the samples being analyzed. One or more MDLspikes may be used per batch if needed.
- 2.5 MDL_{blank} is analyzed with every batch of samples and is carried through the same procedure as the samples being analyzed. One or more MDL_{blank} may be used per batch if needed.

3 Interferences

- 3.1 Isobaric elemental interferences: isotopes of different elements which form singly or doubly charged ions of the same mass-to-charge ratio cause isobaric interferences. Tuning the instrument to generate low abundances of doubly charged ions and oxides minimizes isobaric elemental interferences.
- 3.2 Abundance sensitivity: abundance sensitivity is the contribution by the wings of a mass peak to adjacent peaks. The potential for these interferences is recognized and the spectrometer resolution is adjusted to minimize them by daily tuning procedure according to instrument manufacturer's recommendations.
- 3.3 Isobaric polyatomic ions interferences: isobaric polyatomic interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest and cannot be resolved by the mass quadrupole. Most of the common interferences have been identified. Interference equations are used to correct these interferences. ⁸⁴Kr is also monitored with each run due to its interference with both arsenic and selenium.
- 3.4 Physical interferences: physical interferences are associated with the actual transport of the sample to the plasma, through the plasma, and the transmission of the ions through the mass quadrupole. Internal standards are used to compensate for these interferences.
- 3.5 Memory interferences: memory interferences are caused when isotopes from a previous sample contribute to the signal from another isotope. Rinse and analysis delay times are used to eliminate these interferences. Continuing calibration blanks are used to document the absence of memory effects throughout the run.

4 Safety

Refer to Laboratory Chemical Hygiene Plan, online revision. (See SOP reference 13.8)

5 Apparatus and Equipment

- 5.1 Fully functional ICP-MS.
- 5.2 High purity argon gas supply.
- 5.3 Top loading electronic balance.
- 5.4 Air displacement pipettors capable of delivering volumes between 0.001 ml and 1 ml with an assortment of tips. Air displacement pipettes and auto-pipettors may also be described as mechanical pipettes.
 - 5.4.1 Each day of use, the volume dispensed by each mechanical pipette must be verified for the specific volume for which the pipette is being used.
 - 5.4.1.1 Mechanical pipette volumes are verified by measuring the weight of a volume of water dispensed by the unit. At room temperature, 1 ml of water is equal to 1 g. Pipettes must be capable of $\pm 2.5\%$ accuracy and within 2.5% precision RSD.
 - 5.4.1.2 Auto-pipettors may be verified by measuring the volume dispensed with a graduated cylinder. The volume dispensed must be within $\pm 2.5\%$ of the nominal weight.
 - 5.4.1.3 Air displacement pipettes must be professionally calibrated every 6 months.
- 5.5 Assorted HDPE labware items.
- 5.6 Assorted HDPE Digestion Tubes
- 5.7 Assorted HDPE Centrifuge Tubes
- 5.8 Assorted HDPE labware
- 5.9 Hot block digester capable of maintaining a temperature of 95°C.
- 5.10 Disposable polypropylene ribbed watch glasses (for samples requiring digestion by EPA method 200.2 or EPA Method 200.3).
- 5.11 Auto sampler tubes.
- 5.12 Assorted HPDE or glass volumetric certified labware.

6 Reagents and Standards

- 6.1 All reagents or standards must be logged into the standard log notebook, the container must be labeled with the standard number, expiration date, name, and initials of the person who prepared the solution.
- 6.2 Reagent water: 18M Ω water: Purified water which does not contain any measurable quantities of target elements or interfering elements for each element of interest. Milli-Q water is used by the EPD Metals Lab. Milli-Q water has a resistivity of 18.2 [M Ω .cm] @ 25°C and a TOC of 50 μ g/L or less.
- 6.3 Concentrated Ultra-Pure trace metal grade Nitric acid.
 - 6.3.1 1% Nitric Acid Blank: Inside a vented fume hood, add 1000mL of 18 Ω water to a clean 2L HDPE container. Slowly add 20ml of concentrated Nitric Acid and bring to volume with 18M Ω water.
 - 6.3.2 1:1 Nitric Acid Solution: Inside a vented fume hood, add 400mL of 18 Ω water to a clean 1L HDPE container. Slowly add 500mL of Concentrated Ultra trace metal grade Nitric Acid and bring to volume with 18M Ω water.
 - 6.3.2.1 Acidified Blank Solution: Add 500mL of 18M Ω water to a 1L HDPE

container. Slowly add 5mL of 1:1 Nitric Acid Solution (6.3.2) and bring to final with 18MΩ water.

- 6.4 Concentrated Ultra trace metal grade Hydrochloric Acid.
- 6.4.1 1% Nitric Acid and 0.5% Hydrochloric Acid Blank: Inside a vented fume hood, add 1000ml of 18.2MΩ water to a clean 2L HDPE container. Slowly add 20ml of concentrated nitric acid and 10ml of concentrated hydrochloric and bring to volume with 18.2MΩ.
- 6.5 Tuning Solutions for instruments ELAN 9000 and NexION 1000
- 6.5.1 Tuning Stock Intermediate Solution for ELAN 9000: Add 0.5ml of 1000ug/ml single element vendor stock standards of Magnesium, Barium, Beryllium, Cerium, Cobalt, Indium Lead and Rhodium and bring to volume of 50ml with 1% Nitric Acid Blank Solution (see 6.3.1). The final concentration is 10ug/ml. Prepare solution every two months.
- 6.5.1.1 Tuning Stock Solution for ELAN 9000: Add 0.050ml of the tuning stock intermediate solution and bring to final 50ml with 1% Nitric Acid Blank Solution (see 6.3.1) The final concentration is 0.010ug/ml. Prepare solution every two weeks.
Note: The tuning stock intermediate solution (6.5.1) and tuning stock solution (6.5.1.1) are used for ELAN 9000 ICPMS instruments.
- 6.5.1.2 Tuning Solution Stock for NexION 1000 instruments: Add This solution is a 1ug/L solution of Beryllium (Be), Cerium (Ce), Iron (Fe), Indium (In), Lithium (Li), Magnesium (Mg), Lead (Pb) and Uranium (U) usually purchased from an outside Vendor.
- 6.5.1.2.1 Smart Tuning Solution Stock for NexION 1000 instruments (NexION Set-up Solution): To prepare this solution if not available by Vendor, add 0.5ml of 1000ug/ml single element solution of Be, Ce, Fe, In, Li, Mg, Pb and U to 1% Nitric Acid Blank Solution (see 6.3.1). The final concentration in 10ug/L. The smart tune solution intermediate is prepared every two months.
- 6.5.1.2.2 Smart Tuning Solution for NexION 1000 instruments: Add 0.50mL of smart tuning solution stock (6.5.1.2.1) and bring to final volume of 50mL with Nitric Acid Blank Solution (See 6.3.1) for a final concentration of 1ug/L. Prepare smart tune solution every two weeks.
- 6.6 Internal Standards Solutions (ISTD)
- 6.6.1 ISTD Intermediate Solution #1- Lithium-6 Solution: Dissolve 0.25g of Lithium-6, Li₂ carbonate, 95% atom(6Li₂CO₃) into 20ml of 1% Nitric Acid Blank Solution (see 6.3.1). This solution may take several minutes to dissolve. The final concentration is 2000ug/ml. This solution is prepared every two months.
- 6.6.2 ISTD- Intermediate Solution #2- Add 0.5ml of 1000ug/ml single element vendor stock standards of Germanium (Ge), Indium (In), Lutetium (Lu), Scandium, (Sc) and Lithium-6 (see 6.6.1). Bring to final volume of 50ml with 1% Nitric Acid Blank Solution (see 6.3.1). The final concentration of Ge, In, Lu, Sc is 10ug/ml. The final concentration of Lithium-6 is 20ug/ml. The solution is prepared every two months.
- 6.6.3 (ISTD)- Internal Standard Solution: To a 2L HDPE container, add 2.0ml of ISTD- Intermediate Solution #2, bring to final volume with 1% Nitric Acid Blank Solution (see 6.3.1). This solution is prepared every two weeks.

- 6.6.4 Internal Standard Solution for NexION 1000: To a 500ml clean Teflon volumetric Class A flask, add 0.125ml of 1000ug/ml single element vendor stock standards of Germanium (Ge), Indium (In), Lutetium (Lu) to 1% Nitric Acid Blank solution. In addition, add 2.50ml of 1000ug/ml Scandium (Sc) and 0.0625ml of Lithium-6 (See 6.6.1). Bring to volume with 1% Nitric Acid Blank Solution. The final concentration of Ge, In, Lu and Li is 0.250ug/ml. The final concentration of Sc is 5.0ug/ml.
- 6.7 Interference Check Solution Standards (ICS 1 and ICS 1-2)
- 6.7.1 Interference Check Solution Stock #1: This solution is purchased from an outside vendor.

Table 6.7.1.1 – Interference Check Stock Solution #1	
Aluminum (Al)	1000 mg/L
Calcium (Ca)	1000 mg/L
Iron (Fe)	1000 mg/L
Potassium (K)	1000 mg/L
Magnesium (Mg)	1000 mg/L
Sodium (Na)	1000 mg/L
Phosphorus (P)	1000 mg/L
Sulfur (S)	1000 mg/L
Molybdenum (Mo)	20 mg/L
Titanium (Ti)	20 mg/L
Carbon (C)	2000 mg/L
Chlorine (Cl)	10000 mg/L

- 6.7.2 Interference Check Stock Solution #2: This solution is purchased from an outside vendor.

Table 6.7.2.1 – Interference Check Stock Solution #2	
Silver (Ag)	10 mg/L
Arsenic (As)	10 mg/L
Cadmium (Cd)	10 mg/L
Cobalt (Co)	10 mg/L
Chromium (Cr)	10 mg/L
Copper (Cu)	10 mg/L
Manganese (Mn)	10 mg/L
Nickel (Ni)	10 mg/L
Zinc (Zn)	10 mg/L

- 6.7.3 Interference Check Solution (ICS 1): This solution is made from Interference Check Stock Solution #1 (6.7.1). This solution is prepared every two weeks. (Note: Molybdenum is the only spiked analyte required for this solution with limits of 80-120% of the true concentration. The absolute value of the un-spiked required analytes must be less than the reporting limits.)

Table 6.7.3.1 – Interference Check Solution (ICS 1)	
Aluminum (Al)	50 mg/L

Calcium (Ca)	50 mg/L
Iron (Fe)	50 mg/L
Potassium (K)	50 mg/L
Magnesium (Mg)	50 mg/L
Sodium (Na)	50 mg/L
Phosphorus (P)	50 mg/L
Sulfur (S)	50 mg/L
Molybdenum (Mo)	1 mg/L
Titanium (Ti)	1 mg/L
Carbon (C)	100 mg/L
Chlorine (Cl)	500 mg/L
Total Volume of ICS #1	2.5 ml
Addition of 1% Nitric Acid Blank (see 6.3.1)	47.5 ml
Final Volume of ICS #3	50 ml

- 6.7.4 Interference Check Solution1-2 (ICS 1-2): This check solution is a combination of interference check stock solution #1 and interference check stock solution #2. This solution is prepared every two weeks.

(Note: the recoveries of the spiked analytes must be between 80-120% of the true concentration. The absolute value of the un-spiked analytes must be less than the reporting limits.)

Table 6.7.4.1 – Interference Check Solution (ICS 1-2)

Silver (Ag)	0.02 mg/L
Arsenic (As)	0.02 mg/L
Cadmium (Cd)	0.02 mg/L
Cobalt (Co)	0.02 mg/L
Chromium (Cr)	0.02 mg/L
Copper (Cu)	0.02 mg/L
Manganese (Mn)	0.02 mg/L
Nickel (Ni)	0.02 mg/L
Zinc (Zn)	0.02 mg/L
Molybdenum (Mo)	1 mg/L
Titanium (Ti)	1 mg/L
Aluminum (Al)	50 mg/L
Calcium (Ca)	50 mg/L
Iron (Fe)	50 mg/L
Potassium (K)	50 mg/L
Magnesium (Mg)	50 mg/L
Sodium (Na)	50 mg/L
Phosphorus (P)	50 mg/L
Sulfur (S)	50 mg/L
Carbon (C)	100 mg/L
Chlorine (Cl)	500 mg/L
Total Volume of ICS #1	2.5 mL

Total Volume of ICS #2	0.10mL
Addition of 1% Nitric Acid Blank (see 6.3.1)	47.45 mL
Final Volume of ICS 1,2	50 mL

7 Sample Collection

Water samples and liquid waste samples are collected in narrow and wide mouth plastic (HDPE) bottles. Samples are preserved with sufficient HNO₃ to lower the pH below 2. One to two bottles are required for each sample. Analysis must be preformed within 6 months after preservation.

8 Calibration

8.1 Calibration Curve

The ICP-MS (ELAN 9000 and NexION 1000) is calibrated daily using a multipoint calibration curve. Standards for the ELAN 9000 and NexION 1000 non-digested samples are prepared in 1% Nitric Acid Blank Solution. (See 6.3.1). Samples digested by EPA Method 200.2 and analyzed using the NexION 1000 are prepared in 1% nitric acid and 0.5% hydrochloric acid blank solution. (see 6.4.1). Refer to the calibration standard concentration table for standard concentrations. Minimum acceptable correlation coefficient is 0.995 using linear regression.

Table 8.1.1 - Calibration Standard Names

Name	Use
200.8 Calibration Standard 1 (blank)	Calibration Blank
200.8 Calibration Standard 2,3,4,5,6,7,8,9	Calibration Standard
200.8 ICV	Second Source Calibration
200.8 CCC	Initial and continuing calibration check
200.8 CCB	Initial and continuing calibration blank

Table 8.1.2 - Calibration Standard Concentrations Concentration (ug/L)

Element	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5	Standard 6	Standard 7	Standard 8	Standard 9
Metals	0.00	0.500	1.00	5.00	10.0	25.0	50.0	100	200
As, Sb,	0.00		1.00	5.00	10.0	25.0	50.0	100	200
Zn, V	0.00			5.00	10.0	25.0	50.0	100	200
Ag	0.00	0.100	0.200	1.000	2.00	5.00	10.0	20	40
Se	0.00		5.00	25.0	50.0	125	250	500	1000
Al, Fe, Na, Ca, K, Mg	0.00	10.0	20.0	100	200	500	1000	2000	4000

8.2 Calibration Verification

An ICV, CCC, and CCB are analyzed immediately after calibration. A CCC and CCB are also analyzed before each batch, after every ten samples, after each batch and at the end of an analytical sequence.

Table 8.2.1 - ICV Concentrations

Element	Concentration in ug/L (aqueous matrices)	Concentration in mg/Kg (fish tissue)
Metals	100	0.10
Ag	20	0.020
Se	500	0.50
Al, Fe, Na, Ca, K, Mg	2100	2.1

Table 8.2.2 - CCC Concentrations

Element	Concentration in ug/L (aqueous matrices)	Concentration in mg/Kg (fish tissue)
Metals	100	0.10
Ag	20	0.020
Se	500	0.50
Al, Fe, Na, Ca, K, Mg	2000	2.0

8.3 Laboratory Control Spike, Laboratory Control Spike Duplicate, Matrix Spike, and Matrix Spike Duplicate analyzed with each analytical sample batch

Table 8.3.1 - LCS and LCSD Concentrations (\$PBCU) and (\$PBCU) digested

Element	Concentration in ug/L (aqueous matrices)
Copper	80
Lead	80

Table 8.3.2 - LCS and LCSD Concentrations (\$IMS W)

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	16
Se	80

Table 8.3.3 - LCS and LCSD Concentrations (\$IMSI) and (\$IMSI) digested

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	16
Se	400
Al, Fe, Na	1600

Table 8.3.4 - LCS and LCSD Concentrations (\$IMS SOURCE)

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	16
Se	400
Al, Fe,	1600
Na	4000

Table 8.3.5 - Undigested MS and MSD Concentration (\$PBCU and \$IMSI)

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	16
Se	400
Al, Fe, Na	1600

Table 8.3.6 - Digested MS and MSD Concentrations (\$PBCU and \$IMSI)

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	16
Se	400
Al, Fe	1600
Na	4000

Table 8.3.7 - Digested MS and MSD Concentrations (\$IMS W)

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	16
Se	80

Table 8.3.8 - Digested MS and MSD Concentrations \$IMS_SOURCE

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	16
Se	400
Al, Fe	1600
Na	4000

Table 8.3.9 – LCS/LCSD and MS/MSD Concentrations (\$IMS_F)

Element	Concentration in mg/Kg LCS/LCSD (fish tissue)	Concentration in mg/Kg MS/MSD (fish tissue)
Beryllium	0.080	0.080
Chromium	0.080	0.080
Nickel	0.080	0.080
Copper	0.080	0.080
Zinc	0.080	0.080
Arsenic	0.080	0.080
Selenium	0.40	0.40
Silver	0.016	0.016
Cadmium	0.080	0.080
Antimony	0.080	0.080
Thallium	0.080	0.080
Lead	0.080	0.080

Table 8.3.10 - LCS and LCSD Concentrations (\$IMS_DIS)

Element	Concentration in ug/L (aqueous matrices)
Metals	80
Ag	20
Se	400
Al, Fe,	1600
Na	4000

9 Quality Control

- 9.1 For digested samples: Record all reagents used, volumes, standard or lot numbers, time, temperature, and sample IDs on the digestion log. Fill out a run log with every use of the instrument. The run log must include all samples and standards analyzed in the order they were analyzed.
- 9.2 Verify the pipette calibration by following procedure outlined in section 5.4.1. Record the pipette number, volumes and weights on the digestion sheet.
- 9.3 Refer to Tables 14.1.1-14.1.6 for reporting limits (PQLs) and Table 14.2 for Quality Control Procedures associated with this method. For Quality Control Acceptance Criteria for this method, refer to Appendix A, Table A.1 for SOP 2-012.
- 9.4 Control Limits
 - 9.4.1 The default control limits from EPA Method 200.8 are 85 – 115% recovery for LCS recoveries. The EPD Laboratory applies LCS recovery limits to LCSDs. Note, unless

specified by method, the EPD Laboratory does not validate batch quality based on LCSD recoveries.

- 9.4.2 By default, the EPD Laboratory sets LCS/LCSD precision control limits to be 0 – 15% RPD.
- 9.4.3 10% of all routine samples must be spiked. See Section 9.5 modification below. EPA Method 200.8 requires recovery control limits of 70 – 130% for matrix spikes. The EPD Laboratory applies MS recovery limits to MSDs.
- 9.4.4 By default, the EPD Laboratory sets default MS/MSD precision control limits to be 0 – 15% RPD. MS/MSD precision limits are not dependent on control charting results.
- 9.4.5 Recovery and precision control limits are updated using control charts by Drinking Water. MS/MSD control limits are static at the default limit.
- 9.4.6 See Administrative SOP for Control Charting and Control Limits for further details.
- 9.5 Batch samples in groups of 20. For each batch, analyze a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) for a minimum of 10% of routine samples.
 - 9.5.1 For batches of 1 to 10 routine samples, one MS/MSD pair must be spiked. For batches of 11 to 20 routine samples, two MS/MSD pairs must be spiked using different samples for each pair.
- 9.6 Method Detection Limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.
 - 9.6.1 The actual MDL varies depending on the instrument matrix.
 - 9.6.2 The MDL study must be determined annually for each instrument prior to results being reported for that instrument. The MDL determined for each analyte must be less than the reporting limit for that analyte.
 - 9.6.3 The MDL study for all analytes must be performed initially on a new instrument, after major instrument repairs, changes to the procedures and with Drinking Water Initial Demonstrations (see 9.6.3.1). There are two ways to perform the MDL study. The first is by analyzing seven MDL_{spike} (ML) and seven MDL_{blanks} over 3 separate days. *MDL for new instrument start-up, must be performed over 3 days.* The second way is by analyzing the MDL_{spike} sample (ML_{sample}) with every batch.
 - 9.6.3.1 To perform the seven MDL_{spike} and seven MDL_{blanks} MDL study over three separate days, prepare seven blanks and seven spiked blanks. The seven spiked blanks are spiked at the lowest calibration point on the curve. The seven sets of MDL_{spike} and MDL_{blanks} pairs are analyzed over three separate days. Please note, digested samples must be digested over three days.
 - 9.6.3.2 A continuous MDL study format: One blank is spiked per batch and identified as the ML_{sample}. The ML sample is at the lowest point on the calibration curve. The method blank for each batch is the MDL_{blanks}.
- 9.7 The results of the MDL_{blanks} will be entered into Labworks using the Method Blank test code. The MDL_{spike} result will be entered using the prefix \$ML followed by the test code. The instrument used for the MDL_{sample} and MDL_{blanks}, will be selected using the prefix INSTR followed by instrument number.

Note: The default control limits are presented to assist in defining control limits established with control charts for Drinking water sample analysis and are not used as batch acceptance criteria. All samples from programs other than Drinking water will use method default limits for batch acceptance criteria.

Table 9.4.1. – Default Quality Assurance Criteria for Method EPA 200.8

QC Type	Analyte	Accuracy(%R)		Precision (%RPD)
		LCL	UCL	
LCS/LCSD	All metals	85 – 115		0 – 15
MS/MSD	All metals	70 – 130		0 – 15

10 Procedure:

- 10.1 All drinking water and non-drinking water samples must have the pH checked a minimum of 16 hours after preservation. For drinking water samples (\$PBCU, \$IMSI), the turbidity must also be checked a minimum of 16 hours after preservation. If the pH is <2 and the turbidity is <1 NTU for drinking water samples (\$PBCU, \$IMSI), the sample is poured for analysis by direct aspiration.

Note: If the turbidity for drinking water samples (\$PBCU, \$IMSI) exceeds 1 NTU, the samples must be digested by EPA Method 200.2 or EPA Method 200.3. All other samples must be digested by EPA Method 200.2 or EPA Method 200.3

Note: The following 200.8 test codes are checked for pH only, \$IMS_SOURCE and \$IMS_W.

- 10.2 Dissolved samples (\$IMS_DIS) and an aliquot of acidified blank solution for method blank, LCS, LCSD and MDL_{sample}, must be filtered through a 0.45micron filter before digestion by EPA method 200.2. A minimum of 16 hours after preservation, the pH must be checked.

- 10.3 **Start the instrument and run for at least 30 minutes to warm up the instrument.**

- 10.4 **Under the tuning report method, tune the instrument with tuning stock solution (see section 6.5). Print the tuning report and include in the data pack.** Internal standards are not added to tuning solution. Record the tuning and internal standard numbers on the top of the run log.

- 10.5 Make sure probe is in the internal standard solution to perform calibration and analysis of all samples and standards.

- 10.6 Calibrate the instrument using multiple level standards (see section 8.1). The calibration standards are prepared bi-weekly.

- 10.7 Analyze the ICV immediately after calibration.

- 10.8 Analyze the CCC and CCB. The ICV, CCC and CCB do not count towards the sample total in a batch.

- 10.9 To determine the instrument detection limits (IDLs), change the instrument repetitions from 3 to 10. Aspirate an aliquot of blank solution (see section 6.3.1) for 10 repetitions and calculate the IDL for each element by multiplying the standard deviation by 3. If this result exceeds the Reporting Limit, reanalyze once. If reanalysis fails, end the analysis, correct the problem, and start from step 10.4.

- 10.9.1 Change the number of repetitions back to 3.

- 10.10 Analyze the matrix blank. Some batches (such as TCLP) may have more than one matrix blank per batch. All matrix blanks must be analyzed and used for QC purposes.

- 10.10.1 For \$IMS_DIS, analyze the filter blank.
- 10.11 Analyze the MDL Standard (ML).
- 10.12 Analyze the LCD and LCSD
- 10.13 Begin analyzing samples.
- 10.13.1 A matrix spike must be analyzed for every ten samples in a batch.
- 10.13.2 A matrix spike duplicate must be analyzed for every ten samples in a batch.
- 10.13.3 Dilute any sample above Linear Detection Range with 1% Nitric Acid Blank Solution (see section 6.3.1). The upper LDR limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed.
- 10.13.4 Analyze a CCC and CCB after every 10 samples and at the end of the analytical batch.

11 Calculations

- 11.1 Evaluation of the Linearity of the Initial Calibration
Correlation coefficient of ≥ 0.995 is required.
- 11.1.1 The internal standard calibration is calculated by the instrument software and is documented in the instrument software.

11.2 Mean (\bar{X}):

$$\bar{X} = \frac{X_1 + X_2 + \cdots X_n}{n}$$

- 11.2.1 Where:

$$\begin{aligned} X_1 + X_2 + \cdots X_n &= \text{The sum of a set of values } X_i, i = 1 \text{ to } n \\ n &= \text{The number of values in the set} \end{aligned}$$

11.3 Standard Deviation ($n - 1$) (σ_{n-1}):

$$\sigma_{n-1} = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}}$$

- 11.3.1 Where:

$$\begin{aligned} \bar{X} &= \text{Mean of the values} \\ X_i &= \text{Individual values 1 through } i \\ n &= \text{Number of values} \end{aligned}$$

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11.4 Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{\sigma_{n-1}}{\bar{X}} * 100$$

11.4.1 Where:

 σ_{n-1} = Sample Standard Deviation

 \bar{X} = Mean of the values
11.5 Relative Percent Difference (%RPD or RPD):

$$\%RPD = \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} * 100$$

11.5.1 Where:

 $|X_1 - X_2|$ = Absolute difference between two values

 $\frac{(X_1 + X_2)}{2}$ = Average of two values
11.6 Percent Drift, %Drift:

$$\%Drift = \frac{(\text{Concentration}_{\text{Calculated}} - \text{Concentration}_{\text{Expected}})}{\text{Concentration}_{\text{Expected}}} * 100$$

11.6.1 Where:

 $\text{Concentration}_{\text{Calculated}}$ = Concentration calculated from result

 $\text{Concentration}_{\text{Expected}}$ = Theoretical concentration of the standard
11.7 Percent Recovery:11.7.1 LCS/LCSD:

$$\%Recovery = \frac{\text{Conc}_{\text{spiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.7.1.1 Where:

 $\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

 $\text{Conc}_{\text{expected}}$ = Expected concentration

11.7.2 MS/MSD:

$$\% \text{Recovery} = \frac{\text{Conc}_{\text{spiked}} - \text{Conc}_{\text{unspiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.7.2.1 Where:

$\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

$\text{Conc}_{\text{unspiked}}$ = Concentration found in unspiked sample

$\text{Conc}_{\text{expected}}$ = Expected concentration

11.8 Sample Concentration

$$\text{Concentration} = \frac{CVD_f}{DW}$$

Where C = concentration from instrument in mg/L.

V = final digestion volume in L.

D_f = dilution factor.

DW = sample weight in kg after drying to constant weight at $60^\circ\text{C} \pm 5^\circ\text{C}$.

Use the following formula if there is insufficient sample to dry for digestion:

$$\text{Concentration} = \frac{CVD_f}{WW} \times \frac{100}{P_s}$$

Where C = concentration from instrument in mg/L

V = final digestion volume in L.

D_f = dilution factor.

WW = wet weight of sample (not dried).

P_s = percent solids

$$\text{Percent Solids} = \frac{DW}{WW} \times 100$$

Where DW = sample weight in kg after drying to constant weight at $60^\circ\text{C} \pm 5^\circ\text{C}$

WW = wet weight of sample (not dried).

Waste Concentration Calculation:

$$\text{Concentration} = \frac{CVD_F}{WW}$$

Where C = concentration from instrument in mg/L

V = final digestion volume in L.

D_f = dilution factor

WW = wet weight of sample.

Aqueous sample results are reported in ug/L, biological tissues are reported in mg/kg wet weight, solids are reported in mg/kg dry weight, and wastes in mg/kg wet weight.

12 Waste Management

See GA EPD Laboratory SOP – EPD Laboratory Waste Management Standard Operating Procedure. (See SOP reference 13.4)

13 References

- 13.1 EPA Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry, EMMC Version, Revision 5.4, 1994.
- 13.2 GA EPD Laboratory SOPs – Initial Demonstration of Capability SOP 6-001, online revision and/or Continuing Demonstration of Capability SOP 6-002, online revision.
- 13.3 GA EPD Laboratory SOP – EPD Laboratory Procedures for Control Charting and Control Limits SOP, SOP 6-025, online revision.
- 13.4 GA EPD Laboratory SOP – EPD Laboratory Waste Management SOP, SOP 6-015, online revision.
- 13.5 Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/815-R-05-004, January 2005.
- 13.6 GA EPD Laboratory SOP – Determination of Method Detection Limits SOP, SOP 6-007, online revision.
- 13.7 GA EPD Laboratory Quality Assurance Plan, online revision.
- 13.8 GA EPD Laboratory Safety/Chemical Hygiene Plan & Fire Safety Plan, online revision.
- 13.9 EPA Method 200.2, Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements, U.S. EPA Office of Research and Development: Cincinnati, OH, Revision 2.8, May 1994.
- 13.10 EPA Method 200.3, Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements in Biological Tissues, Revision 1, April 1991; Methods for the Determination of Metals in Environmental Samples Supplement I, US EPA Office of Research and Development, Cincinnati, OH, EPA/600/4-91-010, June 1991.

14 Practical Quantitation Limits PQLs, Precision and Accuracy Criteria, Quality Control Approach, and Instrument Tuning Criteria

Table 14.1.1 Reporting Limits for Method 200.8 \$IMS_DIS			
Parameter/Method	Analyte	Matrix Water Quality	
		RL	Units
200.8	Chromium	5	ug/L
	Nickel	10	ug/L
	Copper	5	ug/L
	Zinc	25	ug/L
	Arsenic	5	ug/L
	Selenium	5	ug/L
	Aluminum	50	ug/L
	Silver	5	ug/L
	Cadmium	0.7	ug/L
	Lead	1	ug/L

Table 14.1.2 Reporting Limits for Method 200.8 \$PBCU			
Parameter/Method	Analyte	Matrix Copper and Lead Drinking Water \$PBCU	
		RL	Units
200.8	Copper	20	ug/L
	Lead	1	ug/L

Table 14.1.3 Reporting Limits for Method 200.8 \$IMSI			
Parameter/Method	Analyte	Matrix IOC Drinking Water	
		RL	Units
200.8	Beryllium	2	ug/L
	*Sodium	1000	ug/L
	Aluminum	50	ug/L
	Chromium	25	ug/L
	Iron	50	ug/L
	Manganese	25	ug/L
	Nickel	40	ug/L
	Zinc	50	ug/L

Table 14.1.3 Reporting Limits for Method 200.8 \$IMSI			
Parameter/Method	Analyte	Matrix IOC Drinking Water	
		RL	Units
	Arsenic	5	ug/L
	Selenium	25	ug/L
	Cadmium	2.5	ug/L
	Antimony	3	ug/L
	Barium	50	ug/L
	Thallium	1	ug/L

*Sodium is not a requirement for this method and will be available upon request.

Table 14.1.4 Reporting Limits for Method 200.8 \$IMS_F			
Parameter/Method	Analyte	Matrix Fish	
		RL	Units
200.8	Beryllium	1	mg/kg
	Chromium	2	mg/kg
	Nickel	2	mg/kg
	Copper	2	mg/kg
	Zinc	5	mg/kg
	Arsenic	2	mg/kg
	Selenium	2	mg/kg
	Silver	1	mg/kg
	Cadmium	1	mg/kg
	Antimony	2	mg/kg
	Thallium	2	mg/kg
	Lead	1	mg/kg

Table 14.1.5 Reporting Limits for Method 200.8 \$IMS_W			
Parameter/Method	Analyte	Matrix WQ Water	
		RL	Units
200.8	Chromium	5	ug/L
	Nickel	10	ug/L
	Copper	5	ug/L

Table 14.1.5 Reporting Limits for Method 200.8 \$IMS_W			
Parameter/Method	Analyte	Matrix WQ Water	
		RL	Units
	Zinc	10	ug/L
	Arsenic	5	ug/L
	Selenium	5	ug/L
	Molybdenum	5	ug/L
	Silver	5	ug/L
	Cadmium	0.7	ug/L
	Tin	30	ug/L
	Antimony	5	ug/L
	Barium	2	ug/L
	Thallium	1	ug/L
	Lead	1	ug/L
	Uranium-upon client request	1	ug/L

Table 14.1.6 Reporting Limits for Method 200.8 \$IMS_SOURCE			
Parameter/Method	Analyte	Matrix Source Drinking Water	
		RL	Units
200.8	Beryllium	2	ug/L
	*Sodium	1000	ug/L
	Aluminum	50	ug/L
	Chromium	25	ug/L
	Iron	50	ug/L
	Manganese	25	ug/L
	Nickel	40	ug/L
	Zinc	50	ug/L
	Arsenic	5	ug/L
	Selenium	25	ug/L
	Cadmium	2.5	ug/L
	Antimony	3	ug/L
	Barium	50	ug/L
	Lead	1	ug/L
	Thallium	1	ug/L
	Copper	20	ug/L
	Silver	5	ug/L
*Sodium is not a requirement for this method and will be available upon request			

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**Table 14.2 Summary of Calibration and QC Procedures for Method
 EPA 200.8**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.8	Metals by ICP-MS	Analyst Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analysis of a QC sample, a method blank and a blind sample. In addition, the analyst must prepare at least one standard. For drinking water samples, an MDL study must be performed. See 9.6.3.1	Once per analyst	matrix blank < RL plus 4 LCS recoveries between 85-115%. Recovery of unknown sample within 85-115%. Perform 3 day initial MDL study drinking water \$PBCU,\$IMSI, \$IMS_SOURCE .	Recalculate results, correct problem, then rerun the initial demonstration for those analytes that did not meet criteria.	

**Table 14.2 Summary of Calibration and QC Procedures for Method
EPA 200.8**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.8	Metals by ICP-MS	Continuing Demonstration: Demonstrate ability to generate acceptable accuracy and precision using a variety of analysis options of a QC sample(s)	Every 6 Months after IDC	matrix blank < RL plus 4 LCS recoveries or Unknown sample between 85%-115% or pass PE sample. For drinking water sample: matrix blank plus 4 LCS recoveries or 1 unknown between control charting limits (See App. A Rev. 0 for SOP 2-012)	Correct the problem. Then rerun the continuing demonstration for those analytes that did not meet criteria	
		MDL study initial	New instrument start-up, major instrument repair and analyst initial demonstration for drinking water analysis	Minimum Detection Limits established shall be <RLs in table 14.1. All spiked MDLs must have a value greater than 0	Re-do MDL study	None
		MDL Study Continuous	Generated yearly over a two-year period	All analyte concentrations must be <RL	Redo Initial MDL Study	Corrective Action Report
		MDL _{spike} For dissolved samples, the MDL _{spike} is the portion filtered through a .45 Micron filter.	Once per analytical batch or as needed to acquire data points per SOPMDL6-007, online revision	All batch QC must meet established criteria. All spiked MDLs must have value greater than 0	Rerun the MDL once and initiate a corrective action. If the MDL fails a second time, do not use MDL data. Update corrective action and use associated sample data	None

**Table 14.2 Summary of Calibration and QC Procedures for Method
EPA 200.8**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.8	Metals by ICP-MS	MDL _{blank} Can be combined with Matrix Blank. For dissolved samples, the MDL _{blank} is the portion filtered through a .45 Micron filter.	Once per analytical batch or as needed to acquire data points per SOPMDL6-007, online version	All batch QC must meet established criteria. All MDL blanks must be <RL. Any result without a positive or negative response, must be entered as "ND"	Rerun once, if still out of limits, correct the problem and reanalyze affected batch if MDL _{blank} is matrix blank	
		Linear Dynamic Range (LDR)	Once every 12 months or whenever major maintenance is performed on the instrument that might affect the instruments detector.	Consecutive levels of increasing concentrations must be within 10% of expected value. See section 10.12.3.		
		Interference Check Solution Standards (ICS 1), (ICS 1-2)	Once every 12 months	Spiked element recoveries between 80-120% recovery, the absolute value of unspiked element concentrations < RL.	Rerun once, if still fails correct the problem; and recalibrate.	
		Analysis of PE sample	Once every 12 months	All analyte results acceptable per the auditing agency.	Correct the problem.	

**Table 14.2 Summary of Calibration and QC Procedures for Method
EPA 200.8**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.8	Metals by ICP-MS	Instrument Tune	Daily before calibration	See Table 14.3 for ELAN 9000 instruments. See Table 14.4 for NexION 1000 instruments.	Correct problem and rerun. If still fails, inspect sample introduction system and connections for possible problems and retune instrument	
		Initial Calibration Using a blank and 9 standards.	Daily initial calibration prior to sample analysis.	Correlation coefficient ≥ 0.995	Correct the problem and recalibrate	
		Initial Calibration Verification (ICV)	Daily Immediately after calibration.	All analyte recoveries between 90% and 110% of the true value.	Rerun once, if still out of control, correct the problem and recalibrate.	
		Instrument Detection Limit (IDL)	After first calibration of the day.	3x Standard deviation of 10 reps < analyte RL	Rerun once, if still out of control, recalibrate	
		Continuing Calibration Blank (CCB).	Daily after calibration, before each batch, after every 10 samples, at the end of each analytical batch.	All analyte concentrations must be <RL.	Rerun once, if still out of control, correct the problem, recalibrate, and reanalyze all samples since the last acceptable CCB.	If unable to reanalyze, flag with "B".
		Continuing Calibration Check (CCC).	Daily after calibration, before each batch, after every 10 samples, at the end of each analytical batch	All analyte recoveries between 90-110%	Rerun once, if still out of control, correct the problem, recalibrate, reanalyze all samples since the last acceptable CCC.	

**Table 14.2 Summary of Calibration and QC Procedures for Method
EPA 200.8**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.8	Metals by ICP-MS	Laboratory Control Sample (LCS). For dissolved samples, the portion filtered through a 0.45 micron filter is used for LCS.	Once per batch.	All analyte recoveries between 85-115%. All drinking water samples between control charting limits. (See App. A, Table A.1 for SOP 2-012).	Rerun one, if still out of control correct the problem, and recalibrate. If still fails, redigest batch if sample amounts permit, and reanalyze all samples in the batch.	If insufficient sample for redigestion, flag with "J".
		Laboratory Control Sample/Laboratory Control Spike Duplicate Precision. For dissolved samples, the portion filtered through a 0.45 micron filter is used for LCSD.	Once per batch.	Relative Percent Difference ≤ 15 . All drinking water samples use control charting limits. (See App. A, Table A.1 for SOP 2-012)	Rerun once, if still out of control correct the problem, and recalibrate. If still fails, redigest batch if sample amounts permit, and reanalyze all samples in batch. The EPD Lab does not validate results based on LCSD recoveries.	If insufficient sample for redigestion, flag with "J".
		Laboratory Control Sample Duplicate Recovery.	Once per batch.	All analyte recoveries between 85%-115%. For drinking water samples use control charting limits. (See App. A, Table A.1, for SOP 2-012).	Comment sample report.	
		Matrix Blank. For dissolved samples, the portion filtered through a 0.45 micron filter is used for Matrix as FB.	Once per batch.	All analyte concentrations must be less than the reporting limit.	Rerun once, correct the problem and recalibrate. If still outside of acceptance criteria, correct the problem, redigest and reanalyze all samples in batch.	Flag data with "B" if unable to reanalyze batch. Comment report if reanalysis has contamination.

**Table 14.2 Summary of Calibration and QC Procedures for Method
EPA 200.8**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
200.8	Metals by ICP-MS	Matrix Spike/Matrix Spike Duplicate Recovery. For dissolved samples, the portion filtered through a 0.45 micron filter is used for Matrix Spike/Matrix Spike Duplicate.	Every 10 samples.	All analyte recoveries between 70-130%.	If recovery exceeds QC limits but CCC, CCB, ICV, LCS, and LCSD are acceptable, matrix effect is suspected.	Comment report.
		Matrix Spike/Matrix Spike Duplicate Precision.	Every 10 samples.	Relative Percent Difference ≤ 15 .		If RPD > 15 inform data user data suspect due to matrix effect. Comment report.
		Internal Standards.	Every sample and standard except tuning solution.	All internal standard recoveries must be between 60% - 125% of the original response of the calibration blank.	Dilute and reanalyze.	None

Table 14.3 Tuning Criteria Method 200.8 For ELAN 9000 Instruments

Mass 220 counts	<100
Cerium Oxide ratio	$\leq 3\%$
Ba ⁺⁺ ratio	$\leq 5\%$
Mass calibration of ²⁴ , ²⁵ , ²⁶ Mg and ²⁰⁶ , ²⁰⁷ , ²⁰⁸ Pb	± 0.1 AMU of unit mass.
RSD of 5 replicates of a 10 ug/L solution of ⁹ Be, ²⁴ Mg, ⁵⁹ Co, ¹¹⁵ In, and ²⁰⁸ Pb	< 5
²⁴ Mg counts of a 10 ug/L solution	>5,000 CPS
¹¹⁵ In counts of a 10 ug/L solution	>10,000 CPS

Table 14.3 Tuning Criteria Method 200.8 For ELAN 9000 Instruments

²⁰⁸ Pb counts of a 10 ug/L solution	>7,500 CPS
Peak width of ²⁴ , ²⁵ , ²⁶ Mg and ²⁰⁶ , ²⁰⁷ , ²⁰⁸ Pb, ¹¹⁵ In	Between 0.6 and 0.8 AMU at 10% peak height.

Table 14.4 Tuning Criteria Method 200.8 For NexION 1000 Instruments

Mass 220 counts	<=3
Cerium Oxide ratio	<=3%
Ce ⁺⁺ ratio	<=5%
Mass calibration of ²⁴ , ²⁵ , ²⁶ Mg and ²⁰⁶ , ²⁰⁷ , ²⁰⁸ Pb	± 0.1 AMU of unit mass.
RSD of 5 replicates of a 1.0 ug/L solution of ⁹ Be, ²⁴ Mg, ⁵⁹ Co, ¹¹⁵ In, and ²⁰⁸ Pb	< 5
⁹ Be counts of a 1.0 ug/L solution	>4500 CPS
²⁴ Mg counts of a 1.0 ug/L solution	>5,000 CPS
¹¹⁵ In counts of a 1.0 ug/L solution	>80,000 CPS
²⁰⁸ Pb counts of a 1.0 ug/L solution	>7,500 CPS
Peak width of ²⁴ , ²⁵ , ²⁶ Mg and ²⁰⁶ , ²⁰⁷ , ²⁰⁸ Pb, ¹¹⁵ In	Between 0.6 and 0.8 AMU at 10% peak height.

**Appendix A for Method 200.8 Determination of Trace Metals in Waters and Wastes by
ICP-MS**

Table A.1 IOC Waters (SIMSI)				
QC Type	Analyte	Accuracy (%R)		Precision (%RPD)
		LCL	UCL	
LCS/LCSD	Aluminum	85	115	15
	Antimony	90	113	10
	Arsenic	85	115	10
	Barium	86	110	10
	Beryllium	85	115	10
	Cadmium	88	115	15
	Chromium	85	115	13
	Iron	85	115	11
	Manganese	88	115	10
	Nickel	90	112	10
	Selenium	89	112	10
	Thallium	85	115	10
	Zinc	89	114	10

MS/MSD	Metals(Aluminum-Zinc) 70	130*	15*
MS/MSD Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.			
Control chart date generated from 1/1/2019-1/1/2021			

Table A.2 WQ Waters (\$IMS_W)				
QC Type	Analyte	Accuracy (%R)		Precision (%RPD)
		LCL	UCL	
LCS/LCSD	Antimony	85	115	15
	Arsenic	85	115	15
	Barium	85	115	15
	Cadmium	85	115	15
	Chromium	85	115	15
	Copper	85	115	15
	Lead	85	115	15
	Molybdenum	85	115	15
	Nickel	85	115	15
	Selenium	85	115	15
	Silver	85	115	15
	Thallium	85	115	15
	Tin	85	115	15
	Zinc	85	115	15
MS/MSD	Metals(Antimony-Zinc) 70	130	15	
Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				

Table A.3 Source Approval Waters (\$IMS_SOURCE)				
QC Type	Analyte	Accuracy (%R)		Precision (%RPD)
		LCL	UCL	
LCS/LCSD	Aluminum	85 -	115	15
	Antimony	90	113	10
	Arsenic	85	115	10
	Barium	86	110	10
	Beryllium	86	115	10
	Cadmium	88	115	15
	Chromium	85	115	13
	Copper	90	112	10
	Iron	85	115	11
	Lead	90	115	10
	Manganese	88	115	10
	Nickel	90	112	10
	Selenium	89	112	10

	Silver	90	115	10
	Thallium	85	115	10
	Zinc	89	114	10
MS/MSD	Metals(Aluminum-Zinc)	70	130*	15*
MS/MSD Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				
Control chart date generated from 1/1/2019-1/1/2021				

Table A.4 PBCU Waters (\$PBCU)				
QC Type	Analyte	Accuracy (%R)		Precision (%RPD)
		LCL	UCL	
LCS/LCSD	Copper	90	- 112	10
	Lead	90	115	10
MS/MSD	Metals(Copper-Lead)	70	130*	15*
MS/MSD Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				
Control chart date generated from 1/1/2019-1/1/2021				

Table A.5 Dissolved Water Quality (\$IMS_DIS)				
QC Type	Analyte	Accuracy (%R)		Precision (%RPD)
		LCL	UCL	
LCS/LCSD	Aluminum	85	115	15
	Arsenic	85	115	15
	Cadmium	85	115	15
	Chromium	85	115	15
	Copper	85	115	15
	Lead	85	115	15
	Nickel	85	115	15
	Selenium	85	115	15
	Silver	85	115	15
	Zinc	85	115	15
MS/MSD	Metals(Aluminum-Zinc)	70	130	15
Control Limits are static by EPA Method/EPD Lab default. Static Limits are generated for trend monitoring purposes.				

Updates to previous version:

Appendix A added.