MBMG ANALYTICAL LABORATORY

QUALITY ASSURANCE MANUAL

Jacqueline Timmer

Montana Bureau of Mines and Geology Open-File Report 729
June 2020

Cover photo: Instruments in the MBMG Analytical Laboratory in 2020. Top row: Thermo iCAP 6000 ICP-OES, Picarro G2131-I $\delta^{13}C$ High-Precision Isotopic carbon dioxide (CO_2) analyzer. Middle row: Picarro L2130-I Isotopic Water Analyzer, Thermo iCAP Q ICP-MS, and HIDEX 300SL Auto TDCR Radon Analyzer. Bottom row: Metrohm 882 IC Plus Anion Chromatograph, Metrohm 855 Robotic Titrator. Photos by Jacqueline Timmer.

TABLE OF CONTENTS

FIGURES

TABLES

LIST OF ACRONYMS

‰	per mille
CCV	Continuing calibration verification
COC	Chain of custody
CRDS	Cavity ring-down spectroscopy
DIC	Dissolved inorganic carbon
DOC	Dissolved organic carbon
DOT	Department of Transportation
EPA	Environmental Protection Agency
ERA	Environmental Resource Associates
FA	Filtered Acidified
FU	Filtered Unpreserved
GCMS	Gas chromatograph mass spectroscopy
GWIC	Groundwater Information Center
H_2SO_4	Sulfuric acid
HCl	Hydrochloric acid
HDPE	High-density polyethylene
HNO_3	Nitric acid
IC	Ion chromatograph
ICP-MS	Inductively coupled plasma mass spectroscopy
ICP-OES/AES	Inductively coupled plasma-optical/atomic emission spectroscopy
ICV	Initial calibration verification
ID	Identification
LCS	Laboratory control sample
LIMS	Laboratory Information Management System
LSC	Liquid scintillation counter
MBMG	Montana Bureau of Mines and Geology
MDL	Method detection limit
mg/L	milligrams per liter (parts per million)
MRL	Method reporting limit
MTDPHHS	Montana Department of Public Health and Human Services
MUS	Montana University System
pCi/L	picocuries per liter
QA/QC	Quality assurance/quality control
RA	Raw Acidified
RPD	Relative percent difference
RU	Raw Unpreserved
SAP	Sampling and analysis plan
SOP	Standard operating procedure
SPE	Solid phase extraction
SRS	Standard resource sample
SVOC	Semi-volatile organic compounds
TOC	Total organic carbon
USGS	United States Geological Survey
VOA	Volatile organic analysis
µg/L	micrograms per liter (parts per billion)

INTRODUCTION

The Montana Bureau of Mines and Geology (MBMG) Analytical Laboratory is located on the first floor of the Natural Resources Building (NRB) on the Montana Tech campus (1300 West Park Street) in Butte, MT. The MBMG Analytical Lab provides a wide array of analytical support to MBMG professionals and their projects, as well as to research from professors and graduate and undergraduate students from Montana Tech and other Montana University System (MUS) schools.

The MBMG analytical lab is committed to quality assurance and strives to ensure all data produced by the lab is high quality and meets or exceeds minimum QA/QC standards. The laboratory is currently certified by the Montana Department of Public Health and Human Services (MTDPHHS) Laboratory Certification Program and is licensed to analyze public drinking water supplies.

This Quality Assurance Manual is intended to meet the requirement of a QA plan outlined in the MTDPHHS Laboratory Certification Program, as well as be useful as a training tool to laboratory personnel. It should also be useful to field personnel/samplers within the MBMG.

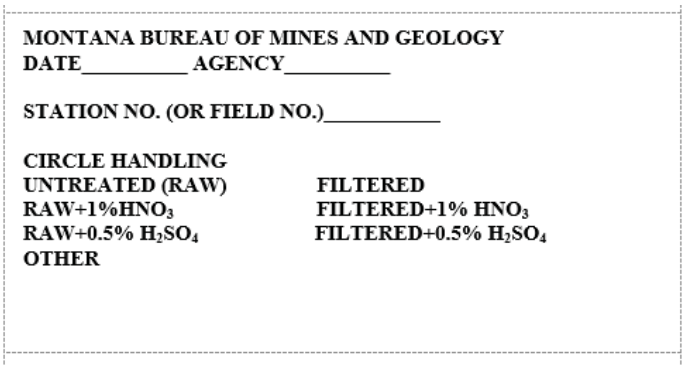
In addition to analyzing for pH, conductivity, alkalinity, major anions (ion chromatograph, IC), and cations (inductively coupled plasma-optical/atomic emission spectroscopy, ICP-OES/AES), the MBMG Analytical Lab also has the capability of analyzing water samples for trace elements, including some rare earth elements (inductively coupled plasma mass spectroscopy, ICP-MS), organic and inorganic carbon (TOC analyzer), 222Radon (liquid scintillation counter, LSC), stable water isotopes and carbonate-carbon isotopes (cavity ring-down spectroscopy, CRDS), and project-specific organic compounds (gas chromatogram mass spectroscopy, GCMS).

SAMPLE HANDLING

Sample Handling for Inorganic Constituents

The adherence to sampling protocols greatly contributes to data quality. As outlined below, these procedures are meant to ensure that data from sample analyses reflect the actual composition of the water source.

Samples should be taken using project-specific standard operating procedures (SOP). All inorganic samples that will be submitted to the MBMG Laboratory are collected in high-density polyethylene (HDPE) bottles obtained from the laboratory. If samplers are planning on collecting 20 or more samples, it is important they let the lab know as soon as possible (at least 7 to 10 days before needed). This not only gives the lab time to get bottles and acid ready, but also ensures there are enough bottles available. Labels are supplied with the bottles and have spaces for the appropriate information concerning the sample (i.e., sample ID, date, time, filtration, preservation, etc.; see fig. 1). All bottles should be filled to a zero headspace capacity when possible. NOTE: When acid is being added for preservation, some headspace is needed to allow for mixing of the acid.


MONTANA BUREAU OF MINES AND GEOLOGY
DATE_____ AGENCY_____

STATION NO. (OR FIELD NO.)_____

CIRCLE HANDLING

UNTREATED (RAW)	FILTERED
RAW+1%HNO_3	FILTERED+1% HNO_3
RAW+0.5% H_2SO_4	FILTERED+0.5% H_2SO_4
OTHER	

Figure 1. Sample bottle label.

Filtration and preservation of samples will depend on what analyses are needed (table 1). The samples collected for the MBMG suite of inorganic parameters are:

<u>Raw Unpreserved</u> (RU):

An unfiltered, unpreserved 500 mL aliquot of the sample, used for the determination of specific conductance, pH, and alkalinity in the laboratory.

Circle **UNTREATED (RAW)** on the label. Samples should be kept at or below 4°C and submitted to the laboratory as soon as possible. While alkalinity and specific conductance have a holding time of 14 to 28 days, respectively, pH needs to be analyzed as soon as possible.

<u>Filtered Acidified</u> (FA):

1. A 500 mL aliquot of sample filtered through a 0.45 µm pore diameter membrane filter and preserved with 5 mL of nitric acid (HNO_3) to

Table 1. Sample container, preservation, and hold time.

Parameter	Method	Volume (mL)	Container	Preservation	Filter		Holding Time (Days)
GENERAL CHEMISTRY							
Acidity	EPA 305.1	500	HDPE	Cool, 4°C	N	RU	14
Alkalinity	EPA 310.1	500	HDPE	Cool, 4°C	N	RU	14
Chloride	EPA 300	250	HDPE	Cool, 4°C	Y	FU	28
Conductivity	EPA 120.1	500	HDPE	Cool, 4°C	N	RU	28
Fluoride	EPA 300	250	HDPE	Cool, 4°C	Y	FU	28
Nitrate-N (IC)	EPA 300	250	HDPE	Cool, 4°C	Y	FU	28
Nitrite-N (IC)	EPA 300	250	HDPE	Cool, 4°C	Y	FU	48 hours
Nitrate/Nitrite-N (nutrient)	HACH	250	HDPE	H_2SO_4,Cool, 4°C	Y	FU	28
pH	EPA 150.1	500	HDPE	Cool, 4°C	N	RU	ASAP
Ortho-Phosphate-P	EPA 300	250	HDPE	Cool, 4°C	Y	FU	28
Radon	EPA 913.0 Mod	125	Glass	Cool, 4°C	N	RU	ASAP
Sulfate	EPA 300	250	HDPE	Cool, 4°C	Y	FU	28
Water Isotopes	Picarro	25	HDPE		Y	FU	NONE
METALS/TRACE METALS							
ICP-OES/ICP-MS-Dissolved	EPA 200.7/ 200.8	500	HDPE	HNO_3	Y	FA	180
ICP-OES/ICP-MS-Total Recoverable	EPA 200.7/ 200.8	500	HDPE	HNO_3	N	RA	180
ORGANICS							
Pentachlorophenol	EPA 528.0 Mod	1000	Glass	HCl, 4°C	N	RA	14 to extract 14 to analysis
Organic Carbon Dissolved / Total	EPA 415.3	40	Glass	H_2SO_4, 4°C	Y/N	FA/ FU	28

a pH <2. This is used for the determination of major metals and trace elements by EPA Methods 200.7 (ICP-OES) and 200.8 (ICP-MS).

Circle **FILTERED + 1% HNO_3** on the label. Once filtered and preserved, samples have a holding time of 6 months.

2. A 250 mL sample aliquot filtered through a 0.45 µm pore diameter membrane filter and preserved with 2.5 mL of 5% sulfuric acid (H_2SO_4) to a pH <2. These are used for the determination of nitrite plus nitrate (and total nitrogen, when requested), often referred to as 'Nutrients' by HACH Methods 10206 and 10242, respectively.

Circle **FILTERED + 0.5% H_2SO_4** on the label. Samples should be kept at or below 4°C. Once filtered and preserved, samples have a holding time of 28 days.

Filtered Unpreserved (FU):

A 250 mL aliquot of sample filtered through a 0.45 µm pore diameter membrane filter. No preservative is added. Samples are used for the determination of anions by EPA Method 300.0 (IC).

Circle **FILTERED on the label**. Samples should be kept at or below 4°C. Once filtered and preserved, samples have a holding time of 28 days.

Water Isotope:

25 mL of filtered, unpreserved sample is also needed for water isotopes. Samples are collected in 25 mL plastic vials with caps that have conical inserts to ensure no headspace. There is no holding time for water isotopes.

Raw Acidified (RA):

A 500 mL aliquot of unfiltered sample preserved with 5 mL of nitric acid for the determination of total recoverable metals and trace elements by EPA Methods 200.7 (ICP-OES) and 200.8 (ICP-MS).

Circle **RAW + 1% HNO_3** on the label. Once preserved, samples have a holding time of 6 months.

Sample Handling for Organic (Inorganic) Carbon

See project-specific SOP for proper sample collection and filtration. Field filtration of the sample, performed by the sampler, will depend upon the sampler's request for total organic carbon (TOC) or dissolved organic carbon (DOC). TOC samples do not require filtration, whereas DOC samples do. NOTE: If filtration is required, ensure filters used are 0.45 µm and appropriate for organic carbon (example: polyethersulfone filter). Organic carbon samples are collected in 40 mL VOA vials and are preserved with two drops of H_2SO_4. Vials should be filled leaving no headspace and need to be kept at or below 6°C. Labels should be clearly marked with preservation and filtration. Samples that have been properly preserved and stored have a holding time of 28 days. TOC/DOC are analyzed by EPA Method 415.3 (TOC analyzer).

While dissolved inorganic carbon (DIC) is not an organic carbon, it falls under this section as it is analyzed with the DOC/TOC method. Inorganic carbon is defined as the carbon in any carbon-containing compound that purges from water that has been acidified. This carbon purges as carbon dioxide (CO_2) and includes metal carbonates, metal bicarbonates, and dissolved carbon dioxide. DIC samples are filtered through a 0.45 µm pore diameter membrane filter and collected in 40 mL VOA vials. Vials should be filled leaving no headspace, however; these samples are NOT preserved with acid. Vials should be kept at or below 6°C. Samples that have been properly sampled and stored have a holding time of 28 days. DIC are analyzed by EPA Method 415.3 (TOC analyzer).

Sample Handling for Radon

See project-specific SOP for proper purging of the well. Radon samples are collected in a 125 mL glass bottle with a Teflon-lined cap. The sample is taken from a non-aerated connection to the well and should come from a source under laminar flow conditions. It is important to ensure there are no air bubbles in the sample bottle. The sample must be labeled with the date and time of collection. Because of radon's short half-life (3.8 days), it is imperative to submit the sample to the laboratory as soon as possible, but no longer than 48 hours after sampling. Samples need to be kept on ice until they are submitted to the laboratory. Radon is analyzed by EPA Method 913.0 modified (LSC).

Sample Handling for Semi-Volatile Organic Constituents

Collect sample as outlined by the project-specific SOP. One liter amber bottles are used to collect samples from the field. The sample is preserved by acidification to pH <2 with 6N hydrochloric acid (HCl). After preservation, the samples should be stored at a temperature between 6°C and 0°C, but must not be allowed to freeze. Semi-volatile organic samples have a 14 day holding time to extraction and 28 days to analysis. These samples are used for the determination of semi-volatiles and chlorophenols by EPA Method 528 (GCMS).

SAMPLE ACCEPTANCE

Prior to sample delivery, samples should be entered into the MBMG Groundwater Information Center data entry portal (DATAGWIC). All data collected in the field are entered, along with the analytical services requested. All sites from which samples are collected are assigned a GWIC identification number (GWIC ID) which is used to track every sample collected from that site (contact GWIC staff for assistance to register your site if it does not have a GWIC ID). Each sample collected during a particular sampling campaign is assigned a unique sample number by the GWIC database (the GWIC sample ID). This information is then passed on to the Laboratory Information and Management System (LIMS by Systat Software, Inc.). The group of samples is assigned an order number and the samples are sequentially assigned a number within the order. The numbering scheme is YY-XXXXX-ZZZ, where YY is the last two digits of the fiscal year, XXXXX is a sequential number assigned to the orders within a fiscal year, and ZZZ is a sequential sample number within the order.

Samples should be delivered to the MBMG Laboratory, Natural Resources Building (NRB) room 103,

located on the Montana Tech campus, 1300 W. Park St., Butte, MT 59701. MBMG Analytical Services are only available to researchers within the MBMG, researchers from other State and Federal agencies collaborating on MBMG projects, or faculty and student researchers within the MUS system. Instructions for submitting samples are given at the time sampling supplies are picked up. Field personnel are made aware that the samples should arrive in a timely manner, packed in coolers with ice to maintain a temperature of less than 4°C throughout the storage time, and protected from damage and freezing. Further, any material shipped by common carrier must comply with DOT rules regarding hazardous substances. When shipping coolers, it is preferable to use coolers that do not have a drain to avoid leakage from melting ice during shipping. If the cooler has a drain, tape should be placed over the drain on both the inside and the outside to attempt to prevent leakage during shipping.

Samples requiring chain of custody documentation must be delivered in person with a completed MBMG COC Form, a chain of custody form identical to its Environmental Protection Agency (EPA) counterpart. The samples must be released into the custody of laboratory personnel by obtaining a signature from a laboratory representative. The laboratory will retain the white copy of the COC form and return the yellow and pink copies to the submitter as proof of transfer. The majority of MBMG programs/projects do not require a COC; the information entered into DATAGWIC is usually sufficient. Samplers should refer to the project SOP/SAP for specifics.

SAMPLE LOGIN

Inorganic

After samples are received (and have been entered into DATAGWIC and LIMS), they are sorted into sets of bottles corresponding to the samples within the order. Labels with the LIMS order and sample number are attached and samples are stored in the designated, refrigerated sample bank. The analytical parameter request is automatically populated in the LIMS and the appropriate methods activated.

Organic

The organic login is also initiated electronically in DATAGWIC. A job order is generated listing the information about each sample that will be submitted for analysis. The field identifier of the site location (GWIC ID), time and date of sampling, and identity of the sampler are all included.

Organic samples do not go into LIMS and are tracked using the GWIC sample ID within the given order number of a job. Job numbers are sequentially incremented.

When the samples are logged in, a separate label is affixed to the bottle indicating the GWIC sample ID. A sample log is generated from DATAGWIC and an extraction sheet is created in Excel. The extraction sheet is used to document the date of each step in the operation (extraction, evaporation, and analysis).

SAMPLE PREPARATION

Inorganic

Determination of dissolved concentrations requires no additional preparation prior to introduction to the instrument(s), other than dilution (when necessary). The dilution schedule is based on the specific conductance of the sample and dilutions are performed by mass using disposable tubes and pipette tips.

Radon samples need to dark-equilibrate prior to analyzing. This simply consists of adding an aliquot of sample to the ‘cocktail’ and allowing it to sit in the instrument (in the dark) for 3 hours before starting analysis. Due to radon’s short half-life (3.8 days), this 3 hour equilibration time needs to be taken into account when calculating the holding time.

Samples that are to be analyzed for water isotopes should be filtered in the field. If for some reason they were not, and/or there is visible debris, they will need to be filtered in the laboratory prior to introduction to the instrument.

Total recoverable metals and trace elements require digestion in accordance with the specifications of EPA Method 200.2 (Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements). The same digestion is used for EPA Method 200.7 (Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry) and EPA Method 200.8 (Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry). The digestions are carried out in a block digester using acid-washed disposable poly-

propylene tubes. In accordance to EPA Method 200.2, a method blank, a laboratory control sample (LCS), a method duplicate, and a laboratory fortified sample (spike) are included for each batch of 20 samples.

Organic

Water samples (1L, acidified with HCl) containing semi-volatile organic compounds are subjected to solid phase extraction (SPE) cartridges. Once the sample has been 'loaded' on the cartridge, the cartridges are dried by pulling air through them with a vacuum. The cartridges are then eluted with dichloromethane and run through a drying cartridge of sodium sulfate. Samples are then evaporated to 1.0 mL. The dichloromethane eluate is spiked with internal standards and analyzed by Method 528.

Glassware, such as evaporator tubes, are washed in a Labconco dishwasher, run through a second cycle on a 'rinse' setting that has a 30 minute dry time, and then rinsed with pesticide-grade acetone.

SHORT-TERM SAMPLE STORAGE

Inorganic

Acidified samples (FA and RA) for metals are stored in the secure storage area in NRB 103A of the Natural Resources Building.

Raw Unacidified (RU), Filtered Unacidified (FU), and Filtered Acidified Nutrients samples are stored under refrigeration at 4°C until analysis is complete. Samples are stored in rooms 103A, 104A, and 106, respectively. These samples may be retained for up to 2 months beyond the method-specific holding time before they are disposed of.

Organic

Preserved samples are stored (refrigerated at 4°C) in NRB 106. The entire aliquot of sample for SVOC is used during extraction. Empty sample bottles are rinsed and disposed of. DOC samples have limited sample remaining after analyses due to the number of replicates required by the method. The remaining sample is not suitable to analyze again due to the large volume of headspace.

LONG-TERM STORAGE

The only samples that are entered into long-term storage are metals (FA and RA) samples. These samples are archived for a period of 5 years in secure storage in NRB 113.

SAMPLE ANALYSIS

Samples are analyzed by EPA-approved methods for parameters listed in table 2. Method detection limits are determined quarterly.

DATA ACQUISITION AND REPORTING

Methods are set up in instrument software such that the reports are produced in formats compatible with importation into spreadsheets. Data from every instrument are exported to an Excel spreadsheet where the data can then be imported into the LIMS software. Quality assurance review is conducted to ensure that QA samples (continuing calibration verification, CCV; initial calibration verification, ICV; LCS; blanks, etc.) meet acceptable recoveries within the spreadsheet. In cases where analytical runs have QA samples that are outside the acceptable limits, samples are designated for reanalysis.

Final qualified analytical results are exported to the GWIC database by LIMS. Reports can be obtained on the web at http://mbmggwic.mtech.edu/. The user must know the specific identifier number (GWIC ID) or the location of the site to access the report.

Raw data files are archived electronically on the MBMG lab server, which is backed up daily.

OUTSIDE EVALUATION PROGRAMS

The MBMG Laboratory maintains certification under the drinking water program administered by the MTDPHHS Environmental Laboratory. The laboratory also participates in the water supply performance evaluation programs administered by Environmental Resource Associates (ERA), and in the United States Geological Survey (USGS) Standard Reference Sample Project (SRS).

PREVENTIVE MAINTENANCE

The analytical equipment employed by the MBMG are given in table 2. The spectrometric equipment items are covered under service contracts (table 2). Other equipment is maintained in-house according to the manufacturer's recommendations. Consumable items, such as gas filters, are changed annually or

Table 2. Equipment routinely used by MBMG Laboratory.

Instrument	Service Contract
Thermo iCAP 6000 ICP-OES	Yes
Thermo iCAP Q ICP-MS	Yes
Metrohm 882 IC Plus Anion Chromatograph a	Yes
Metrohm 882 IC Plus Anion Chromatograph b	Yes
Metrohm 855 Robotic Titrator	Yes
Thermo ISQ 7000 GC-MS	Yes
Agilent 6890N GC/ECD	No
Agilent 6890N/5973N GC/MS with SIM/SCAN	No
Picarro L2130-i Isotopic Water Analyzer	No
Picarro G2131-i $\delta^{13}C$ High-Precision Isotopic Carbon Dioxide (CO_2) analyzer	No
Costech Combustion Module (for ^{13}C analyses of solid samples)	No
Hidex 300SL Auto TDCR Radon Analyzer	No
Hach DR 3800 Spectrophotometer	No

sooner if use of the equipment is higher than normal. Analytical balances are calibrated annually by Quality Control Services, Inc. of Portland, OR.

Waste Disposal

All wastes, including samples, standards, and diluents are disposed of in accordance with the Chemical Hygiene Plan for Laboratories (Montana Tech, March 2010). Inorganic wastes are processed by in-lab chemical management procedures consisting of neutralization and addition of metal precipitants. The liquid is decanted and analyzed prior to disposal in the sanitary sewer. Solids are reposited in an approved metal solid waste holding landfill. Organic liquids are held in approved drums in a storage facility until an annually scheduled pick-up by a waste disposal company occurs.

Chemical Inventory

The MBMG uses the Montana Tech campus inventory database to track chemicals. All incoming chemicals are bar coded and entered into the database. When the material is used up, the bar coded label is removed from the container and the reagent is marked in the database as being disposed.

Certified Analytes

The MBMG Analytical Laboratory maintains certification under the drinking water program administered by the MTDPHHS. Laboratories that hold this certification have met strict quality requirements and have the authority of the MTDPHHS to analyze and submit data that can be used to determine compliance with State and Federal regulations. Certification is maintained through the MTDPHHS Environmental Laboratory for the analytes listed in table 3.

Quality Control Protocol

All analyses are subject to the quality assurance guidelines of the EPA or USGS method under which the samples are analyzed. Table 4 summarizes the QA samples that are included in every analytical procedure and their respective acceptance criteria. Second-source standards (purchased from a different vendor than those used to make the calibration standards) are used as ICV in all methods. Samples used as LCS originate with the USGS Reference Sample Program or ERA Reference Materials. The quality assurance review process is carried out in a spreadsheet as discussed above. The method detection limits (MDL) for the spectroscopic methods are determined for every analytical run and are summarized quarterly. The estimated MDL and method reporting limit (MRL) are then set as limits for reporting analytes in the LIMS. For MBMG work, the MRL is considered to be five times the MDL. Qualifiers are assigned to reported concentrations in the final reports according to QA guidelines of the EPA.

The parameters of pH, specific conductance, and alkalinity are determined by automated methods; therefore, it is not realistic to determine MDLs for these analytes. The sensors are calibrated prior to collection of data and rechecked after every 10 samples.

NOTE: It is important for samplers to understand that the quality control protocol discussed here is in reference to the laboratory and the instrumentation used within it. Project-specific QA/QC is defined within the SOP/SAP for each project. This is where the sampler will find the number/frequency of field blanks, duplicate/triplicate samples, trip blanks, equipment blanks, etc. that are required based on the specific project they are sampling for.

Table 3. Analyte parameters and associated methods.

Parameter	EPA Method	Method Detection Limit	Units	EPA Method	Method Detection Limit	Units
Inorganic: Metals and trace elements						
Aluminum	200.7	0.049	mg/L	200.8	7.56	µg/L
Antimony				200.8	0.05	µg/L
Arsenic				200.8	0.06	µg/L
Barium	200.7	0.031	mg/L	200.8	0.08	µg/L
Beryllium			mg/L	200.8	0.18	µg/L
Boron	200.7	0.032	mg/L	200.8	0.47	µg/L
Calcium	200.7	0.256	mg/L			
Cadmium				200.8	0.05	µg/L
Chromium	200.7	0.032	mg/L	200.8	0.04	µg/L
Copper	200.7	0.036	mg/L	200.8	0.39	µg/L
Iron	200.7	0.043	mg/L	200.8	2.02	µg/L
Lead				200.8	0.15	µg/L
Magnesium	200.7	0.029	mg/L			
Manganese	200.7	0.031	mg/L	200.8	0.05	µg/L
Molybdenum	200.7	0.031	mg/L	200.8	0.04	µg/L
Nickel	200.7	0.032	mg/L	200.8	0.11	µg/L
Potassium	200.7	0.315	mg/L			
Selenium				200.8	0.1	µg/L
Silver				200.8	0.04	µg/L
Sodium	200.7	0.141	mg/L			
Thallium				200.8	0.03	µg/L
Vanadium	200.7	0.06	mg/L	200.8	0.08	µg/L
Zinc	200.7	0.036	mg/L	200.8	0.11	µg/L
Inorganic: Anions						
Bromide	300	0.01	mg/L			
Chloride	300	0.1	mg/L			
Fluoride	300	0.01	mg/L			
Nitrite as N	300	0.01	mg/L			
Nitrate as N	300	0.01	mg/L			
Ortho-Phosphate as P	300	0.02	mg/L			
Sulfate	300	0.5	mg/L			
Inorganic: Other						
pH	150.1					
Alkalinity	310.1					
Specific conductance	120.1					
Organic						
Pentachlorophenol	528.0 modified	0.1	µg/L			
Organic carbon Dissolved / total	415.3	0.25	mg/L			
Other: Isotope						
Radon	913.0	20.0	pCi/L			
Stable water isotopes	Picarro CRDS	NA	‰			

Table 4. Quality control check samples for analytical work.

QC CHECK TYPE		EPA 200.7	EPA 200.8
Metals and Trace Elements	**Frequency**	**Tolerance**	
Continuing Calibration Verification (CCV)	10%	±10%	±10%
Continuing Calibration Blank (CCB)	10%	<MDL	No spec
Initial Calibration Verification (ICV)	After Cal	±5%	±10%
Initial Calibration Blank	After Cal	<MDL	<MDL
Linear Range Check	Once per	±10%	±10%
Interference Check Sample-Interferents	24 hr	No spec	No spec
Interference Check Sample-Analytes	24 hr	No spec	No spec
Laboratory Fortified Blank[b]	24 hr	±15%	±15%
Duplicate Sample	10%	20% RPD	20% RPD
Laboratory Fortified Matrix[c]	10%	±25%	±25%
Serial Dilution	10%	±10%	±10%
Laboratory Control Sample	Batch[a]		
Laboratory Reagent Blank	Batch[a]	<2.2MDL	No spec
EPA CRDL	24 hr	±20%	
Internal Standards	Every Sample		60–125%
Anions		**EPA 300**	
	Frequency	**Tolerance**	
Continuing Calibration Verification	10%	±10%	
Continuing Calibration Blank	10%	<MDL	
Initial Calibration Verification	After Cal	±10%	
Initial Calibration Blank	After Cal	<MDL	
Laboratory Fortified Blank[b]	24 hr.	±10%	
Duplicate Sample	10%	10% RPD	
Laboratory Fortified Matrix[c]	10%	±10%	
Organic Pentachlorophenol	**Frequency**	**Mid-Level**	**Low Level**
Continuing Calibration Check	10%	±50%	
Continuing Calibration Blank	10%	<MDL	
Initial Calibration Verification	After Cal	±15%	±30%
Initial Calibration Blank	After Cal	<MDL	
Matrix Spike	5%	±30%	
Laboratory Control Sample	Batch[a]		
Surrogate	Every Sample	60–130%	

[a]Batch: 20 or fewer samples

[b,c]Laboratory Fortified Blank and Matrix. Figure cited as deviation from 100% recovery