

DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES
 BY INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY
 EPA Method 200.8 (Revision 5.4, 1994)

Table 1A. Summary of Holding Times and Preservation for Trace Elements by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation
Trace Elements in Water	<u>Technical without mercury:</u> 180 days from collection; <u>Technical with mercury:</u> 28 days from collection; <u>Contract without mercury:</u> 35 days from receipt at laboratory; <u>Contract with mercury:</u> 26 days from receipt at laboratory;	HNO ₃ to pH <2; Cool to 4EC ±2EC
Trace Elements in Soil	<u>Technical without mercury:</u> 180 days from collection; <u>Technical with mercury:</u> 28 days from collection; <u>Contract without mercury:</u> 35 days from receipt at laboratory; <u>Contract with mercury:</u> 26 days from receipt at laboratory;	Cool to 4EC ±2EC

^a Individual target elements are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the concentration of individual elements according to the equation specified in Section 12.0 of Method 200.8. Report water sample results in concentration units of micrograms per liter (Fg/L).

Report soil sample results on a dry-weight basis in milligrams per kilogram (mg/kg). Report percent solid and percent moisture to the nearest whole percentage point.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Elements List, CAS Numbers, and Contract Required Detection Limits (CRDL) for Trace Elements by ICP-MS

COMPOUND	CAS No.	CRDL for Water (µg/L)	CRDL for Soil (mg/Kg)
Aluminum	7429-90-5	30	15
Antimony	7440-36-0	2.0	1.0
Arsenic	7440-38-2	1.0	0.5
Barium	7440-39-3	10	5.0
Beryllium	7440-41-7	1.0	0.5
Cadmium	7440-43-9	1.0	0.5
Chromium	7440-47-3	2.0	1.0
Cobalt	7440-48-4	0.5	0.25
Copper	7440-50-8	2.0	1.0
Lead	7439-92-1	1.0	0.5
Manganese	7439-96-5	0.5	0.25
Mercury	7439-97-6	0.2	0.1
Molybdenum	7439-98-7	1.0	0.5
Nickel	7440-02-0	1.0	0.5
Selenium	7782-49-2	5.0	2.5
Silver	7440-22-4	1.0	0.5
Thallium	7440-28-0	1.0	0.5
Thorium	7440-29-1	1.0	0.5
Uranium	7440-61-1	1.0	0.5
Vanadium	7440-62-2	1.0	0.5
Zinc	7440-66-6	1.0	0.5

Table 2. Summary of Calibration Procedures for Trace Elements by EPA Method 200.8

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
ICP-MS Precalibration Routine ^a	Daily prior to instrument calibration	Criteria specified in Section 10.2 of Method 200.8	1. Identify the problem. 2. Tuning criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 1 calibration standard) (ICAL)	Initially, Daily; whenever required, due to failure of IPC, QCS, or CRDL	Acceptable IPC, QCS, and CRDL standards	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Instrument Performance Check (IPC)(mid range calibration standard)	Following the calibration and prior to sample analysis; after every 10 samples; and end of run	90-110% of expected value	1. Recalibrate and verify 2. Reanalyze samples back to last good QCS
Quality Control Sample (QCS) (Separate source from ICAL standards)	Following the IPC and prior to sample analysis	90-110% of expected value or limits listed in Table 8 of Method 200.8, whichever is greater	1. Recalibrate and verify 2. Reanalyze samples back to last compliant IPC
Calibration Blank Verification	After ICAL; every IPC; and end of the analytical sequence	< CRDL	1. Terminate analysis 2. Identify and document the problem 3. Recalibrate, verify and reanalyze all associated samples
Contract Required Detection Limit (CRDL) Verification Standard	After QCS, but before sample analysis	65-135% of expected concentration	1. Reprep and reanalyze standard 2. Recalibrate and verify
Internal Standards (IS) ^b	In all samples, standards, and blanks	IS area within 60-125% of the IS area in the calibration blank	1. Reanalyze all samples analyzed while system was out-of-control

^a Perform precalibration routine specified in Section 10.2 of Method 200.8. Instrument stability must be demonstrated by running the tuning solution a minimum of five times with resulting relative standard deviations (RSD) of absolute signals for all analytes of less than 5%.

^b Section 10.3 of Method 200.8 identifies Table 3 for a list of acceptable internal standards. A minimum of three internal standards must be used.

Table 3. Summary of Internal Quality Control Procedures for Trace Elements by EPA Method 200.8

QC Element	Frequency	Acceptance Criteria	Corrective Action
Laboratory Reagent Blank (LRB)	One per Batch or SDG ^a (1 per 20 samples minimum)	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all associated samples must be prepared again with another method blank and reanalyzed
Laboratory Fortified Blank (LFB)	One per batch or SDG (1 per 20 samples minimum)	85-115% of expected value	1. Terminate analysis 2. Identify and document the problem 3. Reanalyze all associated samples
Laboratory Fortified Matrix (LFM) ^b	A minimum of 10% of the field samples	70-130% of expected value	1. Flag associated data with an "N"
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each seven (7) calendar day period during which field samples in a case are received.

^b If the LFM sample exceeds the calibration range, the sample must be diluted appropriately, re-spiked, and reanalyzed. LFM recovery calculations are not required if the concentration of the analyte added is <30%.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.