



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L313

Metals in Water (ICP-MS Prep and Acidification)

Standard Operating Procedure

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1. Purpose

This SOP describes the acidification, trace metal preservation, and digestion protocols for aqueous environmental samples prior to Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analysis. It covers dissolved metals (filtration + acidification) and total recoverable metals (acid digestion) (Creed et al., 1994).

2. Scope

- **Applies to:** Environmental water samples (surface water, groundwater, runoff, wastewater) requiring dissolved or total recoverable metals analysis by ICP-MS.
- **Does not apply to:** Solid matrices (soils, sediments, tissues), total metals by XRF, or organic analyte preparation.
- **Location:** Environmental analysis laboratories.

3. Responsibilities

Role	Responsibility
Lab PI	Approve SOP, ensure training compliance
Trained Personnel	Follow this SOP, report deviations
New Students	Complete training before independent use

4. Definitions

- **Acid digestion (water samples):** The addition of concentrated HNO_3 (typically 0.5–2% v/v final) to preserved water samples to dissolve any particulate metal and stabilize dissolved metals; a prerequisite for ICP-MS analysis (Creed et al., 1994).
- **Blank (method blank):** A reagent water sample carried through the entire preparation procedure; used to assess and correct for metal contamination introduced during sample processing (APHA et al., 2012).
- **Certified reference material (CRM):** A water sample with certified metal concentrations from an accredited producer (e.g., NIST, USGS); used to verify ICP-MS accuracy (Beauchemin, 2017).
- **Digestion blank:** A solution containing only the acid reagents (no sample) processed through the digestion procedure; detects contamination from reagents and vessels.
- **ICP-MS (Inductively Coupled Plasma Mass Spectrometry):** An analytical technique that vaporizes and ionizes a sample in an argon plasma and separates ions by mass-to-charge ratio to identify and quantify elements at parts-per-trillion concentrations (Beauchemin, 2017).
- **Method detection limit (MDL):** The minimum concentration of an analyte that can be detected with 99% confidence that the true concentration is greater than zero; determined from replicate blank analyses per 40 CFR Part 136 (Creed et al., 1994).
- **Preservation (acidification):** Addition of HNO_3 to a water sample at the time of collection to prevent metal adsorption to container walls and to maintain dissolved metals in solution; required for regulatory compliance samples (Creed et al., 1994).
- **Relative percent difference (RPD):** $|A - B| / ((A + B) / 2) \times 100$; used to assess duplicate precision; acceptance criterion is $\text{RPD} \leq 20\%$ for environmental matrices (Creed et al., 1994).
- **Spike recovery:** The percentage of a known added concentration of a metal that is measured by the instrument; acceptable range is typically 85–115%; used to check for matrix interferences (Creed et al., 1994).

- **Total recoverable metals:** Metals determined in an unfiltered sample after acid digestion; includes both dissolved and particulate-bound fractions (Creed et al., 1994).

5. Safety and Hazards

Hazards

- **Chemical burns and corrosive vapors:** Concentrated nitric acid (HNO_3 , 67–70%) is an extremely corrosive oxidizing agent. Contact causes severe chemical burns. Vapors are highly damaging to respiratory tissue.
- **Hydrochloric acid:** Concentrated HCl produces corrosive fumes and can cause burns on contact.

Engineering Controls

All acid dispensing, sample acidification, and hot block heating must be performed inside an active chemical fume hood.

5.1. Required PPE

- ☐ Safety glasses or face shield
- ☐ Laboratory coat
- ☐ Acid-resistant nitrile gloves (double-gloving recommended)

5.2. Emergency Procedures

In case of acid spill or chemical burn, refer to [SOP-L002 Laboratory Safety](#) or call campus safety. For acid contact with skin or eyes, flush immediately with water for at least 15 minutes and seek medical attention.

6. Equipment and Materials

6.1. Equipment

Item	Notes
Automated sample digestion block	Hot block, programmable to 85–95 °C
Chemical fume hood	Required for all acid work
Analytical balance	If gravimetric volume verification is needed

6.2. Reagents and Consumables

Item	Notes
Nitric acid (HNO ₃)	Ultra-pure, trace metal grade
Hydrochloric acid (HCl)	Trace metal grade; used when target analytes include Ag, Sb, or high Fe (Creed et al., 1994)
Certified metal-free membrane filters	0.45 µm DigiFILTERS or syringe filters
Metal-free polypropylene tubes	15 mL or 50 mL autosampler tubes
Ribbed watch glasses or reflux caps	To prevent evaporation loss during digestion
Narrow-range pH strips	To verify pH < 2
High-purity DI water	For volume adjustment and blanks
Multi-element stock solution	Certified reference material for LFB spikes (Beauchemin, 2017)

7. Procedure

7.1. Sample Preservation and Dissolved Metals Fraction

1. **Filtration:** Filter the raw, unacidified sample immediately upon collection or arrival through a certified 0.45 µm metal-free membrane filter. Discard the first 5 mL of filtrate to rinse the filter matrix. Collect remaining filtrate into a clean polypropylene tube.
2. **Acidification:** Add high-purity trace metal grade HNO₃ dropwise to the sample container to achieve a final matrix concentration of 2% (v/v). For example, add 1.0 mL concentrated HNO₃ per 50 mL sample matrix (Creed et al., 1994).
3. Verify pH is < 2 using a narrow-range pH strip. Store acidified samples for at least 16 hours prior to analysis to ensure proper metal desorption from container walls (Creed et al., 1994).

7.2. Total Recoverable Metals Digestion (Hot Block)

1. Transfer a well-mixed 50.0 mL portion of unfiltered sample into a clean digestion tube.
2. Working inside a fume hood, add 1.0 mL of trace metal grade HNO₃ and 0.5 mL of trace metal grade HCl (Creed et al., 1994).
3. Place the digestion tube into the hot block. Fit with a ribbed watch glass or vapor reflux cap to prevent evaporation loss.
4. Program the block heater to 85–95 °C. Heat the sample for 2 hours without allowing the solution to boil (United States Environmental Protection Agency, 2007).
5. Remove from heat, allow to cool to room temperature, and adjust back to the original 50.0 mL volume mark using high-purity DI water. If visible particulates remain, filter the digestate to prevent ICP-MS nebulizer clogging.

 Avoiding Boil-Over

Monitor samples during the first 15–20 minutes of heating. If the solution begins to bump or boil, reduce the block temperature by 5 °C. Loss of sample volume through boiling invalidates the digestion (United States Environmental Protection Agency, 2007).

7.3. Shutdown and Cleanup

1. Allow the hot block to cool before removing tubes.
2. Rinse all non-disposable glassware and equipment with 10% (v/v) HNO₃ followed by three rinses of DI water.
3. Dispose of acid waste per §8 below.

8. Quality Control and Acceptance Criteria

QC Element	Requirement
Method blank	Process 50 mL DI water through full procedure; analyte concentrations must be below the MDL (Creed et al., 1994)
Laboratory fortified blank (LFB)	Spike multi-element stock into DI water and carry through digestion; recovery must be 85–115% (Creed et al., 1994)
Laboratory fortified matrix (LFM)	Spike a field sample; recovery 70–130% (APHA et al., 2012)
Duplicate samples	RPD ≤ 20% (Creed et al., 1994)
Calibration verification	Initial and continuing calibration per instrument SOP

See Creed et al. (1994) and APHA et al. (2012) for additional QC guidance.

9. Waste Disposal

Waste Type	Container	Disposal Method
Acid-preserved samples	Acid waste carboy	EH&S pickup
Spent acid rinses	Acid waste carboy	EH&S pickup
Used filters	Solid chemical waste	Standard hazardous waste
Empty acid bottles	Triple-rinsed, labels defaced	Glass recycling

10. Maintenance

- **Each use:** Wipe down the hot block surface and fume hood sill after acid work.
- **Monthly:** Inspect digestion tubes for etching or discoloration; replace as needed.
- **As needed:** Replace pH strips and membrane filters before expiration dates.

11. References

- Creed et al. (1994)
- United States Environmental Protection Agency (2007)
- Beauchemin (2017)
- APHA et al. (2012)
- [SOP-L002 Laboratory Safety](#)

APHA, AWWA, & WEF. (2012). *Standard methods for the examination of water and wastewater* (22nd ed., p. 1360). American Public Health Association.

Beauchemin, D. (2017). *Inductively coupled plasma mass spectrometry methods*. Elsevier. <https://doi.org/10.1016/B978-0-12-803224-4.00024-0>

Creed, J. T., Brockhoff, C. A., & Martin, T. D. (1994). *Method 200.8: Determination of trace elements in waters and wastes by inductively coupled plasma–mass spectrometry* (EPA Method EPA/600/R-94/111). U.S. Environmental Protection Agency, Office of Research; Development. <https://www.epa.gov/esam/epa-method-2008-determination-trace-elements-waters-and-wastes-inductively-coupled-plasma-mass>

United States Environmental Protection Agency. (2007). *Method 3015A: Microwave assisted acid digestion of aqueous samples and extracts* [SW-846]. U.S. EPA, Office of Solid Waste. <https://www.epa.gov/hw-sw846/sw-846-test-method-3015a-microwave-assisted-acid-digestion-aqueous-samples-and-extracts>

12. Training Record

Training is documented in the lab training log. Before working independently, personnel must:

1. Read this SOP in full
2. Observe a trained operator perform the procedure
3. Perform the procedure under supervision
4. Be signed off by the PI or designated trainer