



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L410

Hot-Plate Acid Digestion of Soils for Trace Metal Analysis

Standard Operating Procedure

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1. Purpose

This procedure describes the open-vessel, hot-plate acid digestion of soil, sediment, and sludge samples for near-total extraction of lead (Pb) and other trace metals, following US EPA Method 3050B (U.S. Environmental Protection Agency, 1996). The digested extract can be analyzed by ICP-OES, ICP-MS, or flame/ graphite furnace AAS. This is a near-total-dissolution method; silicates and some refractory oxides are not fully dissolved, but environmentally available metals are recovered.

For microwave-assisted digestion using the MARS 5 system, see [SOP-L411](#). The microwave method is faster but processes fewer samples per batch; the hot-plate method described here is slower but requires only standard fume-hood equipment.

2. Scope

- **Applies to:** Soils, sediments, and sludge samples requiring trace metal analysis (Pb, Cd, Cu, Zn, Cr, As, Ni, Fe, Mn, and others), including urban soil lead surveys and photovoltaic panel site characterizations
- **Does not apply to:** Water samples (direct acidification or separate digestion); samples requiring total silicate dissolution (HF-based methods); microwave digestion (see [SOP-L411](#))
- **Location:** EA Program laboratory, SGM 133

3. Responsibilities

Role	Responsibility
Lab PI	Approve SOP, ensure training compliance
Lab Manager	Maintain equipment, stock reagents
Trained Personnel	Perform digestion per this SOP
New Students	Complete training before independent use; supervised for first digestion

4. Definitions

- **Aqua regia:** A 3:1 (v/v) mixture of HCl and HNO₃; dissolves noble metals (gold, platinum) and many sulfide minerals not attacked by HNO₃ alone; more aggressive than single-acid digestion but rarely necessary for soil and sediment.

- **Certified reference material (CRM):** A soil or sediment with certified elemental concentrations from an accredited producer (NIST, USGS); run alongside every digestion batch to verify accuracy.
- **Digestion blank (method blank):** A vessel containing only acid reagents and no soil, processed through the entire digestion procedure; detects contamination from acids, vessels, or the hot plate environment.
- **EPA Method 3050B:** The US EPA hot-plate acid digestion procedure for soils, sediments, and sludges; uses repeated refluxes with HNO_3 and H_2O_2 to dissolve environmentally available trace metals; the reference method for this SOP.
- **Matrix spike:** A sample to which a known concentration of analyte has been added before digestion; used to assess matrix-specific recovery and method accuracy.
- **Partial digestion:** Dissolution of the environmentally available or acid-soluble metal fraction of a sample (EPA 3050B); does not dissolve silicate minerals; appropriate for assessing bioavailability and regulatory compliance; distinguished from total digestion (which requires HF).
- **Reflux:** Heating an acid-sample mixture such that acid vapors condense and return to the vessel; increases digestion efficiency for resistant matrices.

5. Safety and Hazards

5.1. Critical Safety Warning

This procedure uses concentrated mineral acids (HNO_3 , HCl) and hydrogen peroxide (H_2O_2) at elevated temperatures in open vessels. All acid handling and digestion must be performed in a certified chemical fume hood.

5.2. Hazards

- **Chemical:** Concentrated HNO_3 (15.8 M) is a severe oxidizer and corrosive. Concentrated HCl (12 M) is severely corrosive. 30% H_2O_2 is a reactive oxidizer that can cause exothermic reactions with organic matter.
- **Physical:** Hot plate operates at approximately 95 °C; digestion vessels and watch glasses are hot. Burns from contact with heated glassware or splashing acid.
- **Inhalation:** HNO_3 digestion produces toxic NO_x fumes. HCl produces irritating HCl vapor. All heating must occur in the fume hood.

5.3. Required PPE

- ☐ Chemical splash goggles or face shield
- ☐ Lab coat
- ☐ Acid-resistant gloves (neoprene or heavy-duty nitrile, long-cuffed)
- ☐ Closed-toed shoes

5.4. Emergency Procedures

For acid splashes to skin or eyes, flush immediately at the eyewash station or safety shower for at least 15 minutes. For inhalation of fumes, move to fresh air. For acid spills, neutralize small spills with sodium bicarbonate; for large spills, evacuate and use the spill kit. Refer to [SOP-L003 Hazardous Materials Handling](#).

! Important

Never add water to concentrated acid. Add acid slowly to samples. Never allow digestion vessels to go to dryness; this can volatilize analytes and damage vessels.

6. Equipment and Materials

6.1. Equipment

Item	Notes
Hot plate with temperature control	Capable of maintaining 95 C; placed inside fume hood
Digestion vessels	Teflon (PFA) beakers, glass beakers, or borosilicate digestion tubes
Watch glasses	Sized to cover the digestion vessels
Analytical balance (0.001 g)	See SOP-L102
Fume hood	Required for all steps involving acid and heating
Volumetric flasks (50 mL, Class A)	For final dilution
Graduated pipettes or dispensers	For acid delivery
Filter funnels and Whatman No. 41 filter paper	For clarifying digests
Tongs or heat-resistant clamps	For handling hot vessels

6.2. Reagents

Reagent	Grade / Notes
Nitric acid (HNO ₃), concentrated (15.8 M)	Trace Metal grade
Hydrochloric acid (HCl), concentrated (12 M)	Trace Metal grade
Hydrogen peroxide (H ₂ O ₂), approximately 30%	ACS reagent grade
Deionized water (Type I)	From SOP-L11

i Note

All reagents must be Trace Metal grade or equivalent. Analyze a reagent blank before each batch; the blank must read below the method detection limit (MDL) for all target analytes.

7. Procedure

7.1. Sample Preparation

1. Air-dry the soil at room temperature or at 60 C until constant mass
2. Pass through a No. 10 sieve (2 mm) per [SOP-F400 Soil Sampling](#)
3. Homogenize thoroughly using a mortar and pestle
4. Determine gravimetric moisture on a companion subsample per [SOP-L400](#)

7.2. Glassware Cleaning

Acid-wash all vessels, volumetric flasks, and watch glasses per [SOP-L101 Glassware Cleaning](#), Section 6.3 (10% HNO₃ acid bath). Rinse with reagent water and dry in a clean environment.

7.3. Weighing

1. Weigh 0.50–1.00 g of prepared soil into a clean digestion vessel
2. Record the exact mass to the nearest 0.001 g in the lab notebook

7.4. Initial Nitric Acid Digestion

1. Working in the fume hood, add approximately 10 mL of concentrated HNO₃ to the vessel
2. Cover with a watch glass
3. Place on the hot plate set to approximately 95 C. Do not allow the solution to boil.
4. Reflux gently until the initial vigorous reaction subsides and the volume begins to reduce. Do not allow the sample to go to dryness.
5. Continue heating for approximately 30–60 minutes, periodically lifting the watch glass to rinse the underside with DI water back into the vessel

7.5. Oxidation with H₂O₂

1. Remove the vessel from the hot plate and allow to cool slightly
2. Add 1–2 mL of 30% H₂O₂ dropwise. Wait for effervescence to subside between drops.
3. Return to the hot plate and resume heating at 95 C
4. Repeat the H₂O₂ additions until the reaction is minimal (typically 2–4 additions) and the digest appears light-colored

 Warning

Add H₂O₂ very slowly. The reaction with organic matter is exothermic and can cause violent spattering if added too quickly, especially to hot solutions. Always cool the vessel first.

7.6. HCl Treatment

1. Add approximately 5 mL of concentrated HCl to the digest
2. Cover and heat at approximately 95 C for 15–30 minutes
3. This step dissolves residual metal precipitates and improves recovery of metals such as Fe, Al, and Pb

7.7. Volume Reduction

1. Remove the watch glass and continue heating at approximately 95 C
2. Reduce the volume to approximately 5 mL
3. Monitor closely and do not allow the sample to go to dryness

 Important

Evaporation to dryness will cause loss of volatile analytes (As, Se, Hg) and can bake residues onto the vessel walls, making quantitative recovery impossible.

7.8. Dilution and Filtration

1. Remove from the hot plate and allow to cool to room temperature
2. Rinse the vessel walls and watch glass with small portions of DI water
3. If undissolved material is present, filter through Whatman No. 41 filter paper into a clean 50 mL volumetric flask, rinsing the filter and vessel quantitatively
4. If the solution is clear, transfer directly to the volumetric flask
5. Bring to 50.0 mL with DI water
6. Mix thoroughly
7. Transfer to a clean, labeled polyethylene or polypropylene storage bottle. Store at 4 C until analysis.

8. Quality Control and Acceptance Criteria

8.1. QC Requirements

- **Method blank:** Include one vessel containing reagents only (no soil) in each digestion batch. The blank must read below the MDL for all target analytes.
- **Certified reference material (CRM):** Digest a CRM with known metal concentrations in each batch. Recovery: 80–120%.

- **Matrix spike:** Spike a duplicate sample with a known concentration of the target analyte(s). Recovery: 75–125%.
- **Duplicate digestions:** At least 10% of samples. Relative percent difference (RPD) less than 20%.
- **Calibration verification:** Verify with a mid-range check standard every 10 samples during instrumental analysis.

8.2. Calculations

Metal concentration in soil (mg/kg dry weight):

$$C_{\text{soil}} = \frac{C_{\text{digest}} \times V_{\text{final}}}{M_{\text{soil}}}$$

where C_{digest} = concentration in the digest (mg/L), V_{final} = final volume (mL, typically 50), and M_{soil} = mass of dried soil (g).

For soil dried at 60 C, apply a moisture correction to report on a 105 C oven-dry basis (see [SOP-L400](#)):

$$C_{\text{soil, OD}} = C_{\text{soil}} \times \frac{100 + \theta_{60}}{100 + \theta_{105}}$$

9. Waste Disposal

Waste Type	Disposal Method
Acid digest waste (all liquids from digestion)	Collect in labeled acid waste carboy; EH&S hazardous waste pickup
Used filter paper	Acid waste stream (contaminated with metals and acid)
Digested soil residue	Acid waste carboy
Spent H ₂ O ₂	Small volumes may go into acid waste; large volumes, consult EH&S

Acid Waste

All waste from this procedure contains concentrated mineral acids and dissolved metals. **Do not pour down the drain.** Collect in a labeled polyethylene carboy for EH&S hazardous waste pickup.

10. References

- USEPA. (1996). Method 3050B: Acid digestion of sediments, sludges, and soils. Revision 2. In: *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (SW-846). U.S. Environmental Protection Agency, Washington, DC.

- APHA, AWWA, WEF. (2012). *Standard Methods for Examination of Water and Wastewater*. 22nd ed. American Public Health Association (Eds.). Washington. 1360 pp.

U.S. Environmental Protection Agency. (1996). *Method 3050B: Acid digestion of sediments, sludges, and soils* (SW-846, Revision 2). U.S. Environmental Protection Agency. <https://www.epa.gov/sites/production/files/2015-06/documents/epa-3050b.pdf>

11. Training Record

Before performing hot-plate acid digestion independently, personnel must:

1. Read this SOP, [SOP-L002 Laboratory Safety](#), and [SOP-L003 Hazardous Materials Handling](#) in full
2. Review the SDS for concentrated HNO₃, HCl, and 30% H₂O₂
3. Observe a trained operator perform a complete digestion cycle
4. Perform at least one complete digestion batch under direct supervision, including blank and CRM, with acceptable QC results
5. Be signed off by the PI or lab manager

12. Appendix A: Example Calculation

```
# Trace metal concentration from hot-plate digest
# EPA 3050B

C_digest_mg_L <- 1.24      # Pb concentration from ICP-MS (mg/L)
V_final_mL <- 50.0        # Final dilution volume (mL)
M_soil_g <- 0.752         # Mass of dried soil digested (g)

C_soil_mg_kg <- (C_digest_mg_L * V_final_mL) / M_soil_g

cat(sprintf("Digest concentration: %.3f mg/L Pb\n", C_digest_mg_L))
cat(sprintf("Soil mass: %.3f g\n", M_soil_g))
cat(sprintf("Final volume: %.1f mL\n", V_final_mL))
cat(sprintf("Soil Pb: %.1f mg/kg\n", C_soil_mg_kg))

# Screening levels
cat(sprintf("\nCA DTSC residential screening level: 80 mg/kg\n"))
cat(sprintf("EPA RSL residential: 400 mg/kg\n"))
```

13. Appendix B: Method Comparison – Hot Plate vs. Microwave

Feature	Hot Plate (EPA 3050B, this SOP)	Microwave (EPA 3051A, SOP-L411)
Vessel type	Open (reflux under watch glass)	Sealed (pressurized)
Temperature	Approximately 95 C	175 C
Digestion time	2–4 hours (hands-on)	10 minutes (automated)
Sample throughput	Limited by hot plate space	Up to 24 vessels per run
Equipment required	Hot plate + fume hood	MARS 5 microwave system
Acid volume per sample	Approximately 15 mL total	10 mL
Organic matter handling	H ₂ O ₂ oxidation step	Sealed vessel; pre-digest if vigorous
Metal recovery	Comparable for most elements	Comparable; slightly higher for volatile elements due to sealed system
Best for	Small batches; no access to microwave	Routine batches; time-sensitive work