



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L411

Microwave-Assisted Acid Digestion of Soils for Trace Metal Analysis

Standard Operating Procedure

SOP Number	SOP-L411
Revision	0.2
Author(s)	Marc Los Huertos, Isaac Medina
Date	2026-06-26
Organization	EA Program, Pomona College

FOR INTERNAL USE ONLY

This document is the property of the Environmental Analysis Program at Pomona College.
Unauthorized distribution is not permitted.

Table of contents

1 Purpose	2
2 Scope	2
3 Responsibilities	2
4 Definitions	2
5 Safety and Hazards	3
5.1 Critical Safety Warning	3
5.2 Hazards	3
5.3 Required PPE	3
5.4 Emergency Procedures	3
6 Equipment and Materials	4
6.1 Equipment	4
6.2 Consumables	4
6.3 Reagents	4
7 Procedure	5
7.1 Glassware and Vessel Cleaning	5
7.2 Sample Preparation	5
7.3 Microwave Digestion	5
7.4 Critical	6
7.5 Filtration and Extract Preparation	6
7.6 Disposal and Cleanup	7
8 Quality Control and Acceptance Criteria	7
8.1 QC Requirements	7
8.2 Calculations	7
9 Waste Disposal	8
10 References	8
11 Training Record	8

12 Appendix A: Example Calculation 9

13 Appendix B: Troubleshooting 9

1. Purpose

This procedure describes the microwave-assisted acid digestion of soil, sediment, and sludge samples for trace metal analysis, following US EPA Method 3051A (U.S. Environmental Protection Agency, 2007). The digested extract can be analyzed by flame atomic absorption spectrometry (FAAS), inductively coupled plasma mass spectrometry (ICP-MS), or other suitable methods. This is a near-total-dissolution method; silicates and some refractory oxides (SiO_2 , TiO_2) may not fully dissolve but environmentally available metals are recovered.

For non-microwave hot-plate acid digestion, see *SOP-L65*.

2. Scope

- **Applies to:** Soils, sediments, and sludge samples requiring trace metal analysis (Pb, Cd, Cu, Zn, Cr, As, Ni, Fe, Mn, and others) at all EA Program sites, including urban soil lead surveys and photovoltaic panel site characterizations
- **Does not apply to:** Water samples (direct acidification or separate digestion per EPA 3005A); samples requiring complete silicate dissolution (use HF-based methods); organic tissue digestion
- **Location:** Advanced Laboratory, Seaver North (MARS 5 microwave); EA Program laboratory, SGM 133 (sample preparation and filtration)

3. Responsibilities

Role	Responsibility
Lab PI	Approve SOP, ensure training compliance
Lab Manager	Maintain MARS 5 microwave, stock reagents
Trained Personnel	Perform digestion and filtration per this SOP
New Students	Complete training before independent use; must be supervised for first digestion run

4. Definitions

- **Closed-vessel digestion:** Digestion performed in sealed vessels under pressure; higher temperatures are achievable (up to 240 °C) compared to open-vessel hot-plate digestion; reduces loss of volatile elements and acid consumption.

- **EPA Method 3051A:** The US EPA microwave-assisted acid digestion method for soils, sediments, and sludges; uses HNO_3 (with optional HCl) in closed PTFE vessels at controlled temperature and pressure; the reference method for this SOP.
- **MARS system (Microwave Accelerated Reaction System):** The CEM Corporation microwave digestion system used in the EA Program lab; controls temperature and pressure in each vessel during digestion.
- **Maximum allowable working pressure (MAWP):** The pressure limit of the digestion vessel specified by the manufacturer; exceeding this limit risks vessel rupture; monitored by the MARS control software.
- **PTFE (polytetrafluoroethylene):** A chemically inert fluoropolymer used to fabricate digestion vessels; resistant to concentrated acids and temperatures up to $\sim 260^\circ\text{C}$; must be pre-cleaned with HNO_3 before use to remove trace metal contamination.
- **Ramp time:** The programmed time over which the microwave system increases temperature from ambient to the target; too-rapid ramping can generate excessive pressure and risk vessel failure.

5. Safety and Hazards

5.1. Critical Safety Warning

This procedure uses concentrated nitric acid (HNO_3) under high temperature and pressure in sealed vessels. Improper handling can cause severe acid burns, toxic fume exposure, or vessel rupture. Read the entire procedure and the MARS 5 operator's manual before beginning.

5.2. Hazards

- **Chemical:** Concentrated HNO_3 (15.8 M) is severely corrosive and produces toxic NO_x fumes. Optional HCl (12 M) is also severely corrosive. Both cause severe burns on skin and eye contact.
- **Physical:** Microwave vessels reach 175°C and develop significant internal pressure. Residual pressure and acid vapor remain in the headspace after digestion. Hot vessels can cause burns.
- **Inhalation:** NO_x and HCl fumes are toxic. All acid handling and vessel venting must occur in the fume hood.

5.3. Required PPE

- ☐ Chemical splash goggles (not safety glasses)
- ☐ Face shield (when opening hot vessels)
- ☐ Lab coat
- ☐ Neoprene or heavy-duty acid-resistant gloves (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 and seek medical attention if

symptoms persist. For vessel rupture or spill, evacuate the area and notify the lab manager. Refer to [SOP-L003 Hazardous Materials Handling](#).

6. Equipment and Materials

6.1. Equipment

Item	Notes
MARS 5 Microwave Accelerated Reaction System	CEM Corporation; with pre-programmed EPA 3051A method
Microwave digestion vessels with carbon fiber sleeves	Must be compatible with the MARS 5; pressure-relief mechanism required
Temperature and pressure sensors (if applicable)	Consult instrument supervisor
Fume hood	Required for all acid handling and vessel venting
Analytical balance (0.001 g)	See SOP-L102
Centrifuge	Capable of 2,000–3,000 rpm; see <i>SOP-L109</i> or <i>SOP-L108</i>
Mortar and pestle	For grinding dried soil
No. 10 sieve (2 mm mesh)	For sieving ground soil
Drying oven	Set to 60 C; see SOP-L105

6.2. Consumables

Item	Cat. No. / Notes
50 mL graduated centrifuge tubes	For dilution and centrifugation
15 mL centrifuge tubes	For filtered digest storage
Disposable syringes (10 mL)	Acid-resistant polypropylene
Syringe filters, 0.45 μ m, 25 mm	Fisherbrand Cat. No. 09-719D
100 mL beakers or aluminum crinkle dishes	For drying samples

6.3. Reagents

Reagent	Grade / Notes
Nitric acid (HNO_3), concentrated (15.8 M)	Trace Metal grade; Fisher A509P500 or equivalent
Hydrochloric acid (HCl), concentrated (12 M) (optional)	Trace Metal grade; for improved recovery of Al, Sb, Ba, Be, Cr, Fe, Mg, Ag, V (U.S. Environmental Protection Agency, 2007)

Reagent	Grade / Notes
Reagent water (Type I)	From SOP-L11 ; must be interference-free at target MDLs

i Note

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

7. Procedure

7.1. Glassware and Vessel Cleaning

1. Acid-wash all vessels, centrifuge tubes, and volumetric ware per [SOP-L101 Glassware Cleaning](#), Section 6.3 (10% HNO₃ acid bath)
2. Rinse with reagent water and dry in a clean environment
3. For microwave vessels, follow the manufacturer's cleaning protocol (CEM recommends a cleaning cycle with dilute HNO₃ between batches)

7.2. Sample Preparation

1. Weigh approximately 3–5 g of soil into a clean beaker or aluminum dish. Avoid obviously non-soil material (large rocks, twigs, leaves).
2. Dry at 60 C overnight in the drying oven. Do not exceed 60 C; higher temperatures can volatilize mercury and other semi-volatile metals.
3. Allow to cool. Using a clean mortar and pestle, grind the dried soil and pass through a No. 10 sieve (2 mm).

7.3. Microwave Digestion

1. Weigh 0.500 g (+/- 0.001 g) of the sieved, dried soil into a clean microwave vessel. For oil-contaminated or high-organic samples, use no more than 0.250 g. Record the exact mass.
2. Working in the fume hood, add 10.0 mL (+/- 0.1 mL) of concentrated HNO₃ (Trace Metal grade) to the vessel.

⚠ Vigorous Reaction

Adding acid to samples with high organic matter may cause immediate effervescence. If a vigorous reaction occurs, leave the vessel uncapped in the fume hood for 1–2 hours to allow pre-digestion before sealing.

3. *Optional:* For improved recovery of Al, Sb, Ba, Be, Cr, Fe, Mg, Ag, and V, substitute 9 mL HNO₃ + 3 mL concentrated HCl (aqua regia variant). Note: HCl can increase interferences with some ICP-MS analyses; consult the analyst.

4. Seal the vessels according to the manufacturer's instructions (CEM Corporation, 2015)
5. Place each sealed vessel into a slot on the vessel rack **with a carbon fiber sleeve in place**

7.4. Critical

Never place a vessel into a rack slot without its carbon fiber sleeve. The sleeve provides essential containment in the event of a vessel failure. Operating without the sleeve risks vessel explosion.

6. Secure the rack into the MARS 5 microwave
7. If applicable, connect the temperature and pressure sensors to the designated control vessel(s)
8. Select the pre-programmed EPA 3051A method. Verify the following parameters:

Parameter	Specification (EPA 3051A)
Ramp to 175 C	5.5 +/- 0.25 minutes
Hold at 175 +/- 5 C	4.5 minutes (remainder of 10 min total)
Maximum pressure	Per vessel rating; relief mechanism must activate before failure

9. With instructor verification that all vessels are sealed and the rack is properly installed, close the microwave door and start the program
10. After the program finishes, allow the vessels to cool inside the microwave for at least 5 minutes, then transfer to the fume hood for further cooling

7.5. Filtration and Extract Preparation

1. Working in the fume hood, carefully uncap each vessel and vent for 2–3 minutes to release residual acid vapor and pressure
2. Quantitatively transfer the digest into a clean 50 mL graduated centrifuge tube, rinsing the vessel walls with small portions of reagent water to ensure complete transfer
3. Dilute to the 25.0 mL mark with reagent water. Record the final volume. Keep all samples as close to the same volume as possible.
4. Cap and centrifuge at 2,000–3,000 rpm for 10 minutes or until the supernatant is clear

Note

Refractory materials (SiO_2 , TiO_2 , Al_2O_3) may remain undissolved and cause persistent turbidity. If the supernatant is not clear after centrifugation, allow the sample to stand overnight before filtering.

5. Using a clean 10 mL syringe, carefully withdraw 10 mL of supernatant without disturbing the pellet
6. Attach a fresh 0.45 μm syringe filter (Fisherbrand 09-719D) and filter the supernatant into a clean 15 mL centrifuge tube
7. Use a new syringe and filter for each sample
8. Cap and label the filtered extract. Store at 4 C until analysis.

7.6. Disposal and Cleanup

1. Collect all acid digestion waste (vessel rinses, supernatant residues, centrifuge pellets in acid) in a labeled acid waste carboy in the fume hood. EH&S pickup.
2. Rinse and clean microwave vessels per the manufacturer's protocol
3. Dispose of used syringe filters and syringes in the acid waste stream (they are contaminated with concentrated HNO_3 digest)
4. Wipe down all bench surfaces with DI water
5. Remove and properly dispose of gloves; wash hands thoroughly

8. Quality Control and Acceptance Criteria

8.1. QC Requirements

- **Reagent blank:** Include one vessel containing only 10 mL HNO_3 (no soil) in each digestion batch. The blank must read below the MDL for all target analytes.
- **Laboratory control sample (LCS):** Digest a certified reference material (CRM) or laboratory reference soil with known metal concentrations in each batch. Recovery should be 75–125% of the certified value.
- **Matrix spike:** Spike a duplicate sample with a known concentration of the target analyte(s). Recovery should be 75–125%.
- **Duplicate digestions:** Process at least 10% of samples in duplicate. Relative percent difference (RPD) should be less than 20%.
- **Calibration verification:** After instrument analysis, verify the calibration with a mid-range check standard every 10 samples.

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 from ICP-MS or FAAS), V_{final} = final digest volume (mL, typically 25), and M_{soil} = mass of dried soil (g).

Since the soil was dried at 60 C (not 105 C), apply a moisture correction if reporting on a 105 C oven-dry basis:

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

where θ_{60} and θ_{105} are the moisture contents at 60 C and 105 C respectively (determined on a companion subsample per [SOP-L400](#)).

9. Waste Disposal

Waste Type	Disposal Method
Acid digest waste (vessels, rinses, pellets)	Collect in acid waste carboy; EH&S hazardous waste pickup
Used syringe filters and syringes	Acid waste stream (contaminated with HNO ₃)
Centrifuge tube residues	Acid waste carboy
Digested soil pellets	Acid waste carboy (contain concentrated acid)

Acid Waste

All waste from this procedure contains concentrated HNO₃ and dissolved metals. **Do not pour down the drain.** Collect in a labeled polyethylene carboy for EH&S hazardous waste pickup.

10. References

- CEM Corporation. (2015). *MARS 5 microwave accelerated reaction system: Operator's manual*. <https://www.cem.com>
- U.S. Environmental Protection Agency. (2007). *Method 3051A: Microwave assisted acid digestion of sediments, sludges, soils, and oils* (SW-846, Revision 1). U.S. Environmental Protection Agency. <https://www.epa.gov/sites/production/files/2015-12/documents/3051a.pdf>

11. Training Record

Before performing microwave 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 and HCl
3. Review the MARS 5 operator's manual (vessel loading, program selection, safety interlocks)(CEM Corporation, 2015)
4. Observe a trained operator perform a complete digestion cycle (vessel loading through filtration)
5. Perform at least one complete digestion batch under direct supervision, including a blank and a CRM, with acceptable QC results
6. Be signed off by the PI or lab manager

Important

First-time users must be directly supervised for their entire first digestion run. Independent operation is permitted only after sign-off.

12. Appendix A: Example Calculation

```
# Trace metal concentration from microwave digest
# EPA 3051A, analyzed by ICP-MS

C_digest_mg_L <- 0.452      # Pb concentration in filtered digest (mg/L)
V_final_mL <- 25.0         # Final volume after dilution (mL)
M_soil_g <- 0.503          # Mass of dried soil digested (g)

# Concentration in soil (mg/kg)
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))

# Compare to screening levels
cat(sprintf("\nCalifornia DTSC residential screening level for Pb: 80 mg/kg\n"))
cat(sprintf("EPA RSL for Pb (residential): 400 mg/kg\n"))
if (C_soil_mg_kg > 400) {
  cat("WARNING: Exceeds EPA residential screening level\n")
} else if (C_soil_mg_kg > 80) {
  cat("NOTE: Exceeds CA DTSC residential screening level\n")
} else {
  cat("Below both screening levels\n")
}
```

13. Appendix B: Troubleshooting

Symptom	Probable Cause	Corrective Action
Vigorous reaction when acid added	High organic matter or carbonite content	Pre-digest uncapped in fume hood for 1–2 hours before sealing
Vessel pressure exceeds limit during run	Sample mass too large; excessive organic content	Reduce sample mass to 0.250 g; extend pre-digestion time
Supernatant not clear after centrifugation	Refractory oxides (SiO ₂ , TiO ₂) remain undissolved	Allow to stand overnight; filter through 0.45 um; this is normal for EPA 3051A
Blank reads above MDL	Contaminated acid, vessels, or water	Use fresh Trace Metal grade acid; re-clean vessels; check DI water quality

Symptom	Probable Cause	Corrective Action
CRM recovery below 75%	Incomplete digestion; sample loss during transfer	Verify temperature reached 175 C; rinse vessels quantitatively; consider HNO ₃ /HCl mixture
CRM recovery above 125%	Contamination; calibration error	Check blank; recalibrate instrument; re-analyze
High RPD between duplicates	Soil heterogeneity; inconsistent weighing	Grind and sieve more thoroughly; weigh to 0.001 g precision