POMONA COLLEGE	Environmental Analysis Teaching and Research Laboratory	Date: 3/11/2017	Number: 35
	Standard Operating Procedure	Title: Microwave Assisted Acid Digestion of Soils for Trace Metal Analysis	
	Approved By: Los Huertos	Revision Date: October 6, 2017	

1. Scope and Application

1.1 This method provides a quick and effective digestion procedure for the extraction of trace metals from soil samples using a Microwave. Prepared samples can be analyzed for their trace metal content by flame atomic absorption spectrometry (FAA), inductively coupled plasma mass spectrometry (ICP-MS) or other suitable analysis methods.

1.2 This procedure was modified from the EPA test method 3051A and provides students with guidance on preparing samples, running a microwave acid digestion, filtering and extracting the sample for analysis.

1.3 The SOP can be applied to the preparation of sediments, sludges, and soil samples.

2. Summary of Method

Digestion of soil samples begins by oven (or air) drying the sample (approximately 3-5 grams) at 60 ℃ overnight. The oven dried samples are grinded with a mortar and pestle and passed through a sieve. Next, 0.5 g of the dried samples are transferred into microwave vessels, sealed and placed into a MARS 5 laboratory microwave. The microwave runs a 10 minute program which meets the heat and pressure qualifications outlined in the EPA method. After the microwave program the vessels are allowed to cool for at least 5 minutes inside the microwave and then for into a fume hood to cool down for another

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3. Definitions

4. Interferences

5. Health and Safety

5.1 The method relies on extracting soils in hot, i.e. boiling concentrated acid. Thus, maintaining a safe laboratory environment and diligience to properly use PPE is key to the success of this SOP.

6. Personnel & Training Responsibilities

6.1 To be qualified on for this SOP students must be appropriately trained.
The technician must be approved for the following SOPs:

- SOP 01 Laboratory Safety
- SOP 02 Handling of Hazardous Material

6.1. Equipment and Supplies

1. Mesh sieves -(Sizes: No.10; Locations: entry room of the lab; Cabinet: Ziploc Bags & Notebooks) total amount: 4 (order 2 more)
2. balance -(Mettler Toledo Precision Balance, Model MS1602TS)
3. Filter paper Whatman No. 41 or equivalent.
4. Disposable Aluminum Crinkle Dishes with Tabs 332.60 25433-008 VWR 60 mL VWR Disposable Aluminum Crinkle Dishes with Tabs
5. Fisherbrand 25mm syringe filter 0.45 um, Cat no 09-719D MF-Millipore Membrane Filters
6. Tongs -(Cabinet 9: Mattle Tins, Plastic Bottle)
7. Hot plate -(Thermo Scientific Cimarec S88857104 Stirrer, 7x7" Aluminum; 120 VAC, Assembled in China, inside the Fumehoods)
8. Funnel -(64mm, 10500, 12PK, Made in Mexico, The White table with water sinks; the top drawer by the wall)
9. Glass wool CAS Number 65997-17-3 Sigma Aldrich Catalog
10. 250ml Erlenmeyer flasks
11. 100ml volumetric flasks

12. 100mL beaker
13. 50mL Centrifuge tubes
14. 15mL Centrifuge tubes
15. Stop watches
16. Gloves

6.2. Reagents and Standards

6.2 Reagent grade[1] chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is questionable, analyze the reagent to determine the level of impurities. The reagent blank must be less than the MDL in order to be used.

6.3 Reagent Water. Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified. Refer to Chapter One for a definition of reagent water.

6.4 Nitric acid (concentrated), HNO . Acid should be analyzed to determine level of 3 impurities. If method blank is ¡ MDL, the acid can be used.

7. Estimated Time

7.1 Digesting soil, extracting Pb, and filtering supernatant generally requires 120 minutes to complete the digestion and filter samples.

8. Procedure

You must wear safety goggles or eyeglasses Plastic Gloves are available to protect your hands from acid. Wash off any acid, even on the gloves.

8.1. Start with Clean Glassware

- All vessels and volumetricware must be acid washed and rinsed with reagent water
- SOP XX describes a procedure for cleaning microwave vessels using hot HCl and HNO3 (Chuck has special soap that he uses to wash the microwave vessels instead of acid washing)

[1] metal grade!

- Polymeric or glass volumetric ware and storage containers should be cleaned by leaching with more dilute acids (10% V/V) appropriate for the specific material used then rinsed with reagent water and dried in a clean environment

8.2. Homogenize the Samples

1. Weigh out approximately 5 grams of your soil sample into a clean beaker. Try to avoid things that are obsviously not soil such as larger rocks, plant material (such as twigs, leafs, large pieces of grass, etc.)

2. oven dry the samples at 60 ℃overnight

3. Using a clean mortar and pestle, grind the samples and pass them through a XX mm sieve

8.3. Sample Digestion

1. Weigh 0.500 g (to the nearest 0.001 g) of the sieved soil into a clean and appropriate microwave vessel. For oil contaminated samples use no more than 0.250 g. Be sure to record the weight of each sample in a laboratory notebook

2. Add 10 mL ($\pm$ 0.1 mL) concentrated nitric acid (trace metal grade) in a fume hood. Be sure to wear protective gloves and face mask.
   Note: The addition of acid to samples containing volatile or easily oxidized organic species may cause a vigorous reaction. If you notice an immediate reaction it is okay to allow the sample some time for pre-digestion in the fume hood. Leave the vessel in the fume hood for a couple of hours with the cap loosely fitted on top. You want to ensure that any gases escape into the hood. Also, using an acid mixture here instead (9 mL nitric acid to 3 mL of hydrochloric acid) has been shown to improve the recoveries of some metals including: Aluminum, Antimony, Barium, Beryllium, Chromium, Iron, Magnesium, Silver, and Vanadium but can increase interference with some analysis methods (see EPA method 3051A).

3. Seal the vessels with the proper caps and according to the manufacturers directions

4. Place the sealed vessel into a slot on the vessel rack containing a carbon fiber sleeve. DO NOT PLACE THE VESSEL INTO A SLOT WITHOUT A SLEEVE as this can result in the vessel exploding due to buildup of pressure.

5. Secure the rack with the vessels into the MARS 5 microwave

6. *If applicable, connect the appropriate temperature and pressure sensors to the vessels (ask Prof. Taylor about this step)*

7. Select the correct program for the microwave to perform. The MARS 5 in the Advanced Laboratory in Seaver North has already been pre-programmed with a method that will follow the specifications for a 10 minute acid digestion described in the EPA method 3051A for up to 24 vessels. With guidance from an instructor double check

that all vessels are sealed and the rack is correctly placed in the closed microwave. Then run the program.

Note: You can create a new program if you need to digest more vessels. The microwave should follow the basic heating guidelines below. For full information turn to the EPA method 3051A.

- Each sample should reach 175 5 C in 5.5 0.25 mins
- Should remain at 175 C for 4.5 min (or the remainder of the 10 min digestion)
- The pressure to peak between 5 and 10 minutes for most samples.
- If pressure exceeds the limits of the vessels it should be safely and controllably reduced by the built in pressure relief mechanism of the vessel.

8. After the microwave process has finished, allow the vessels to cool for at least 5 minutes or until they are cool enough to be moved into a fume hood or some other well-ventilated area to cool down further

8.4. Filtering and Extraction Process

1. Once the microwave vessels are sufficiently cool enough to handle, and working under a fume hood uncap and vent the samples for 2-3 minutes. This is to avoid a rush of acid vapor that may still be in the headspace.

2. Quantitatively (i.e. with minimal loss of sample) transfer the sample(s) into clean 50 mL graduated centrifuge tubes

3. Carefully dilute the samples up to the 25 mL mark with reagent water. Be careful not to go past the 25 mL mark and keep all samples as close to the same volume as possible for accuracy.

4. Secure the caps back onto the centrifuge tubes

5. Centrifuge the sample tubes at 2,000 - 3,000 rpm for 10 min or until clear.

   Note: sometimes when undissolved materials such as SiO2, TiO2, or other refractory oxides, remain this may cause the supernatant not to look clear even after centrifugation. Allowing the sample to stand overnight will give time for things to settle further and can improve clarity.

6. Now, using a clean syringe and XX filter withdraw 10 mL of the supernatant and filter into a 15 mL centrifuge tube. Be careful not withdraw any of the centrifuge debris at the bottom of the centrifuge tube and use a new filter and clean syringe for each sample. Ask your instructor to demonstrate how to use the syringes and filters as this step can be difficult to accomplish without prior experience.

8.5. Dispose of sample and solutions as directed by instructors. Clean up your bench space.

9. Quality Control and Quality Assurance

9.1 Blank Solutions: Blank Solutions Explanation

9.2 Standard Solutions: Standard Solutions Explanation

9.3 Spiked Solutions: Spiked Solutions Explanation

10. References

References

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10.1 Glass Wool 18421. 2017. Sigma-Aldrich. Accessed April 4. Sigma Aldrich.

10.2 VWR Disposable Aluminum Crinkle Dishes with Tabs — VWR. 2017. Accessed April 4. VWR.

10.3 MF-Millipore Membrane Filters - Filter Discs and Membranes. 2017. Accessed April 4. Filters.

10.4 Analytical Methods for Atomic Absorption Spectroscopy Methods for AA Spectroscopy

10.5 Concepts; Instrumentation and Techniques in Atomic Absorption Spectrophotometry Techniques in AA Spectrophotmeter