1. Scope and Application

1.1 This method provides digestion procedures for the preparation of sediments, sludges, and soil samples for analysis by flame atomic absorption spectrometry or inductively coupled plasma optical emission spectrometry (ICP-OES).

1.2 The SOP can be applied to sediments, sludges, and soil samples.

Contents

2. Health and Safety

2.1 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.

3. Personnel & Training Responsibilities

3.1 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

4. Preparation

4.1. Supplies and Equipment

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

- Gloves
- 250ml Erlenmeyer Flask- (Back cabinet: erlenmeyer flasks & beakers; there are 21)

4.2. Reagents

4.1 Reagent grade 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.

4.2 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.

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

- Nitric acid

5. Estimated Time

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

6. Procedure

6.1 Part II: Determination of Lead levels using Atomic absorption spectroscopy
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.

6.1. Preparation of the soil samples

6.2 From the Mason jar with mesh on top, sieve approximately 10.0 g soil on to the plastic weighing dish.

6.3 Transfer all the weighed soil to a 250-mL ehrlemeyer (conicle-y shaped flask). Label the flask with your initials on label tape.

6.2. Extracting the Lead:

6.4 In the fume hood - Add 20 mL of 1M nitric acid CAREFULLY (slowly) to the soil.

6.5 Heat the mixture near boiling on the hot plate (medium setting) until the fumes are almost colorless. Heat for 5 -10 additional min. Do not let the mixture go dry - add a few mLs of water if needed. At end, with tongs, remove flask from plate, and cool 5-10 min. Then take back to bench.

6.6 CAREFULLY add 75 mL of deionized water to the flask and swirl GENTLY. Let stand 3-5 min so heavier particles can settle out.

6.7 Take a funnel and put a small wad of glass wool in the funnel and place the funnel in a 100-mL beaker. Label this beaker with your initials on tape.

6.8 Carefully transfer the acid/soil solution from the flask to the beaker. You will not remove all the soil particles, but most of the larger ones will be filtered out.

6.9 Using a syringe, remove

6.10 Fill the volumetric flask to the circular mark on the neck with deionized water. Mix well by putting your thumb over the stopper and inverting the flask several times. Let stand several minutes to allow soil to settle down. Slowly pour 15 mL of the water at the top of the volumetric flask into the centrifuge tube to take over the KSD main building.

- SO get centrifuge tubes and then move them to Keck? How are we going to store the tubes if necessary? How are we moving the tubes to Keck? Could it be better to do the whole thing at Keck?

6.3. Calibrating Solutions:

6.11 Blank Solutions: Blank Solutions Explanation

6.12 Standard Solutions: Standard Solutions Explanation

6.13 Spiked Solutions: Spiked Solutions Explanation

6.4. D. Determining the lead content:

6.14 Centrifuge a 15-mL sample until solution is clear. Usually 3 to 4 min. Use a syringe and Millipore filter to push (gently) about 10 mL of solution through the filter into the small labeled sample vial.

6.15 Measure the concentration of the solution on the Atomic Adsorption Spectrometer. Multiply the reading for your sample by 10 to calculate the lead concentration in

your soil sample. The final result is in âĂIJppmâĂİ, which means milligrams (0.0010 g) of lead per kilogram of soil.

6.5. Dispose of sample and solutions

6.16 Clean up your bench space.

7. References