Standard Operating Procedure:
Removing carbonates from soil for measurement of organic carbon and/or ^{13}C signature

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1.0 PURPOSE

The purpose of this standard is to provide a procedure for the removal of carbonates from soil for measurement of organic.

2.0 DEFINITIONS

A corrosive liquid is one that will destroy and damage other substances with which it comes in contact. Of most concern is living tissue such as eyes, skin and respiratory tract.

The centrifuge is a commonly used tool in laboratory research. It uses centrifugal force to separate substances in liquid or solid media according to particle size and density differences.

3.0 POTENTIAL HAZARDS

Hydrochloric acid is corrosive and causes eye and skin burns, if contacted. May cause severe respiratory tract irritation with possible burns. May cause severe digestive tract irritation with possible burns.

Grinding of soil may release sediment dust.

Rotors on high-speed centrifuge and ultracentrifuge units are subject to mechanical stress that can result in rotor failure. Improper loading and balancing of rotors can also cause failure. Aerosolization of biological, chemical, or radioactive materials can cause unintended exposure.

4.0 PERSONAL PROTECTIVE EQUIPMENT

Use chemical splash goggles for eye protection for operations that present splash hazards. Hand protection requires gloves or other gloves developed that withstand corrosive liquids for at least the amount of time given to perform the procedure, if available. Check glove manufacturer for recommendations on a suitable glove. Wear a lab coat and closed-toe shoes with non-slip soles.

5.0 ENGINEERING AND VENTILATION CONTROLS

Adequate ventilation is essential when working with corrosive liquids because of the irritating effects of corrosives on the respiratory system. Prepare and process samples in a fume hood.

6.0 SPECIAL HANDLING PROCEDURES

1. Never allow any unprotected part of the body to touch corrosive liquids;
2. Do not breathe corrosive fumes
3. If sediment dust is generated while grinding soil, perform grinding in the fume hood.

7.0 FIRST AID

Hydrochloric acid first aid:
Eyes: Get medical aid immediately. Do NOT allow victim to rub eyes or keep eyes closed. Extensive irrigation with water is required (at least 30 minutes).
Skin: Get medical aid immediately. Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Destroy contaminated shoes.
Ingestion: Do not induce vomiting. If victim is conscious and alert, give 2-4 cup-fuls of milk or water. Never give anything by mouth to an unconscious person. Get medical aid immediately.
Inhalation: Get medical aid immediately. Remove from exposure and move to fresh air immediately. If breathing is difficult, give oxygen. Do NOT use mouth-to-mouth resuscitation. If breathing has ceased apply artificial respiration using oxygen and a suitable mechanical device such as a bag and a mask.

If sediment dust is contacted in eyes, flush eyes with plenty of water for 15 minutes. Seek medical aid, if eye irritation continues.

8.0 SPILL AND ACCIDENT PROCEDURES

In the event of a small spill of a corrosive liquid (< 1 liter) such as 0.1- 6 M hydrochloric acid;

1. Cover the liquid entirely with sodium bicarbonate.
2. Sweep up bicarbonate/acid paste and prepare for hazardous waste disposal.

In the event of a large chemical spill, follow these guidelines:

1. Notify everyone in the immediate area and the supervisor.
2. Evacuate personnel from the spill area.
3. Deny entry.
4. Notify lab supervisor and/or Risk Management for further clean-up.

9.0 SEQUENCE OF OPERATION for removing carbonates from soil measurement of organic carbon and/or ^{13}C signature.

1. Grind soil.
2. Put a small amount (2000 ug) of soil into a small test or microfuge tube. Use smaller amounts if soils are rich in carbonates. Include checks to confirm a successful procedure if desired. These can be for example, soils of known organic carbon and/or 13C content, artificially enriched with different amounts of carbonate.
3. Add 0.1 M HCl, leaving enough space in the tubes for bubbles to form. More concentrated acid may used if carbonate is expected to be high, up to approx. 1 M HCl.
4. Make sure acid and soil are well mixed, and wait for bubbles to stop.
5. Test the pH of at least some of the samples with pH paper or indicator.
6. If the pH is close to 7, all the acid has been consumed and there is still carbonates in the sample. Add a small aliquot of concentrated, i.e. 6 M HCl to the tube to renew the acid to o.1M or whatever was used before. Mix and test pH again.
7. Once the pH remains low, less than approx. 2, all carbonate has been consumed.
8. Centrifuge the samples, pour off the liquid into _____ and dry the samples. Alternatively, simply dry the samples, this will take longer but no dissolved carbon will be lost. Make sure samples are completely dry so that no HCl remains.
9. Weigh an appropriate amount into tin capsules for analysis.

10. WASTE DISPOSAL

Anything less than a pH of 4 cannot go down the sink. Pour all waste into a properly labeled hazardous waste jar.