

## Appendix C Soil Cleaning Procedures

### C.1. Procedures.

Soils contain a variety of metals and organics that may react with target analytes. Therefore, soil cleaning is usually required. Use mild treatments to preserve the character of the soil. Otherwise, the soil may become sandy and unable to mimic the complexing and absorption nature of real-world samples. The soil would then be an unsuitable matrix for PE samples. Suitable treatment methods described below are acid wash for inorganic contaminants and solvent extraction for organic contaminants.

**C.1.1. Acid Wash.** Acid wash is usually used to reduce inorganic contaminants such as metals from soils. Follow these steps to acid wash soil:

- (1) Weigh 10 grams of soil and place into a 4-liter plastic container along with a Teflon<sup>®</sup>-coated magnetic stirring bar.
- (2) Add 4 liters of pH 2 nitric acid solution and start stirring.
- (3) Set the stirrer speed to suspend most of the soil into the acid solution, but not so fast that the stirring bar cannot track the magnet.
- (4) Check the pH and add 50% v/v (1+1) nitric acid to bring the pH to less than one if not already at that level.
- (5) Check the pH hourly to ensure it does not rise above two. If the pH rises above two, adjust to below pH 2 with 50% v/v (1+1) nitric acid.
- (6) Remove the container from magnetic stirrer after stirring for four hours and allow to stand until all particulate matter has settled.
- (7) Decant carefully and dispose of the acid solution according to regulations.
- (8) Add four liters of deionized water and start the stirring again.
- (9) Stop the stirring after one hour and allow the soil to settle to the bottom again.
- (10) Decant the wash water and repeat the water washing two or more times.
- (11) Check the pH of the final wash water. It should be between pH 4 and pH 6. If the pH is less than 4, additional water washes are required until the pH is within the required range.
- (12) Do not add base to adjust the pH under any circumstances.
- (13) Filter the washed soil through a 0.45- $\mu$ m membrane filter and air dry.
- (14) Store the treated soil in a nitric acid-washed plastic jar with a Teflon<sup>®</sup>-lined cap at 4°C in the dark. This treated soil will be used to prepare PE samples for inorganic analyses.

If a large quantity of acid-washed soils is needed, follow these steps:

- (1) Treat multiple portions of soils.
- (2) Combine all treated portions into one container after the second wash.
- (3) Wash the combined soil with deionized water until the pH falls in the required range.
- (4) Filter through a 0.45- $\mu$ m membrane filter and allow to air dry.
- (5) Store the treated soil in an acid-washed plastic jar with a Teflon<sup>®</sup>-lined cap at 4°C in the dark.

Note: The acid wash procedures may be scaled up; however, the proportions of nitric acid solution to soil must remain the same in all cases.

**C.1.2. Solvent Extraction.** Solvent extraction is usually used to reduce organic contaminants such as semivolatile organic compounds from soils. Because soils do not always contain high concentrations of organic contaminants, it is preferred to collect clean soils from uncontaminated sites rather than to clean dirty soils from contaminated sites. Follow these steps for solvent extraction:

- (1) Place 20 grams of dry soils in an extraction thimble or between two plugs of glass wool.
- (2) Extract the soils with 300 mL of appropriate solvent (e.g., 1:1 [v/v] acetone/hexane, 10:1 [v/v] toluene/methanol, or methylene chloride) in a Soxhlet extractor for 16 - 24 hours.
- (3) Air dry the extracted soils and then thermally treat them at 105 - 125°C overnight to remove the remaining extraction solvents.
- (4) Store the treated soil in a clean glass jar with a Teflon<sup>®</sup>-lined cap at 4°C in the dark. This treated soil will be used to prepare PE samples for organic analyses.
- (5) To prepare a large quantity of solvent-extracted soils, individually treated portions may be combined into one container and mixed by tumbling and shaking to provide a uniform blend of treated soils.

Note: The above-mentioned procedures may be scaled up; however, the proportions of extraction solvent to soil should remain the same in all cases.