

INTERNAL USE ONLY

Preparing Soil Samples for Solid Phase Extraction

Cleanup and trace enrichment of soil samples are important goals of solid phase extraction (SPE) in environmental analysis. SPE is commonly used to extract organic pollutants from environmental samples such as soils, groundwater, or tap water. Since soils are complex samples (that is, soils are “dirty” samples – just a little SPE humor for you), proper pretreatment steps (also known as matrix manipulations) are required to have a sample with a sufficiently low viscosity and particulate content that it is able to flow through the SPE tube. This TECH TIP offers strategies for preparing soil samples for SPE.

Matrix manipulations

The analysis of soils by SPE involves first the extraction of analytes by using an organic solvent (or acid or base), followed by cleanup and purification by SPE. (CAUTION: The presence of naturally occurring humic substances often leads to reduced recoveries. Their impact is readily evident during method development when spiked surrogate samples are compared to spiked sample blanks. For complete details refer to the tech tip called “Strategies for Minimizing Humic Acid Interferences in Environmental Samples”.)

General protocol of pretreatment includes a sequence of steps:

1. *Remove the rocks from sample so that you are left with a fine powder.*
2. *Sonicate (or shake/stir) the sample in the proper solvent – that is, select the appropriate organic solvent and turn on the sonicator (or other device available for stirring sample).*

Obviously, this is a very critical step in extracting the target analytes from the soil into the solvent for further processing by SPE. It is common to use a water-miscible organic solvent such as methanol, acetone or acetonitrile. The solvent must have sufficient contact time with the soil so that it can extract the organic analytes. Sonicating or stirring the sample helps ensure proper mixing of the sample.

3. *Filter sample.*
4. *Collect the supernatant.*
5. *Dilute with water (RP methods) to reduce the organic content of the sample to less than 10%.*

Since these polar organic solvents can evaporate slowly, it is preferred to dilute with water to lower the organic solvent content and apply to a reversed phase SPE tube. However, this can greatly increase the sample size. A rule of thumb for suggesting a proper bed mass for processing these larger samples: polymer resin = 500mg sorbent for every 100g of soil; silica-based (C18) = 1g sorbent for every 100g of soil.

6. *Apply to the SPE tube. For SPE methods see the specific application notes listed below or contact the Technical Department.*

IMPORTANT! Modifications to this general protocol might be necessary to adapt it to your specific target analyte(s).

Here are some examples from methods found on the Internet illustrating the above steps in action to prepare a soil sample for SPE:

1. **small sample (5g)**

Add 5g moist soil with known water content to 5mL distilled water. Shake by sonication for 15 minutes; add 5mL of acetone and shake by sonication for 15 minutes. Centrifuge the mixture at 4500 rpm for 10 minutes. Decant the supernatant into a reservoir containing 500mL distilled water (to lower the organic content of the sample prior to loading onto a SPE tube). Proceed to SPE cleanup.

2. **large sample (50g)** note the amount of contact time during the shaking process.

Weigh 50g of wet soil into an Erlenmeyer flask and add 200ng of surrogate standard to the sample. Add 120mL of the extraction solvent acetone-water-acetic acid (80+19+1, v/v/v), cover the top of the Erlenmeyer flask with aluminum foil and shake overnight. Centrifuge the sample at 3000 rpm for 5 minutes and filter the supernatant into a round-bottom flask. Add 50mL of the extraction solvent, re-extract the sample by ultrasound, centrifuge and filtrate supernatant into the round-bottom flask once more. Evaporate the combined extracts to approximately 20mL by means of a rotary evaporator and add approximately 180mL of HPLC-grade water to this extract. Acidify the sample to pH2 with concentrated hydrochloric acid and add one per cent of methanol (2mL). Proceed to SPE cleanup.

For additional info on SPE methods for pesticides extracted from an aqueous sample refer to:

TN-021 PAHs extracted from water using strata-X

TN-022 Urea pesticides extracted from water using strata-X

ER-007 PAHs extracted from water using Strata C18-E

ER-008 PCBs extracted from water using Strata C18-E