SCF 508

Extraction of Total Petroleum Hydrocarbons (TPH) from Soil Using Supercritical Fluids

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Introduction

The United States Environmental Protection Agency (EPA) estimates that there are presently 5 – 7 million underground storage tanks in the United States. Approximately 1.6 million tanks are currently regulated by the EPA, and 1.4 million are used to store petroleum fuel products. Those underground fuel tanks that have developed leaks are a prime source of groundwater contamination.

Supercritical Fluid Extraction of TPH from soil using carbon dioxide has been approved by the EPA as method 3560 and is currently used to monitor petroleum hydrocarbon contamination of soils, sediments, and fly ash.

Equipment

- ✓ Applied Separations' *Spe-ed*™SFE Supercritical Extraction System

Materials

- ✓ *Spe-ed* Matrix (Cat. #7950)
  - *Diatomaceous earth*
- ✓ Carbon dioxide – (SFC/SFE grade)
- ✓ Tetrachloroethylene (spectropholometric grade)
- ✓ C18/18% Collection cartridge (1g/6mL, Cat.#12007)
- ✓ Methylene chloride (pesticide grade)

Method

Weigh 3g of sample into a pre-cleaned drying dish. A drying agent (e.g. anhydrous magnesium sulfate or diatomaceous earth) may be added to samples that contain water in excess of 20% to increase porosity or to bind water. The amount of the drying agent will depend on the water content of the sample. Typically, a ratio of 1:1 works well for wet soils and sediment material.

Transfer the weighed sample to a clean extraction vessel and use two plugs of silanized glass wool to hold the sample in place. Fill the void volume with *Spe-ed* Matrix. Attach end fittings and install the extraction vessel in the oven.

Extract the sample according to the extraction conditions using either tetrachloroethylene as a collection solvent or C18 SPE cartridge as a collection sorbent. After the extraction, analyze the tetrachloroethylene solvent by EPA Infrared method 8440, or elute the C18 SPE cartridge with tetrachloroethylene and analyze by EPA Infrared method 8840. In addition, the analyst may elute the C18 SPE cartridge with methylene chloride and analyze by method 8015 non-halogenated volatile organics by gas chromatography.

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Extraction Conditions

Extraction vessel:	10 - 24mL
Sample:	3g
Pressure:	5000 psi
Temperature:	80ºC
CO_2 Flow Rate:	1L/min. (gas)
Collection:	C18 Cartridge or tetrachloroethylene (3mL)
Dynamic:	30 minutes

References

EPA Method 3560 Supercritical Fluid Extraction

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