

Extraction of TPH (Total Petroleum Hydrocarbon) from Soils by Pressurized Solvent Extraction (PSE)

Introduction

Pressurized solvent extraction is a new technique that reduces solvent consumption and sample preparation time. Solvent is pumped into an extraction vessel containing the sample and is heated and pressurized. The pressurized solvent at high temperature accelerates the extraction process by increasing the solubility of the analyte in the solvent and also increasing the kinetic rate of desorption of the analyte from the sample matrix.

Underground fuel tanks that develop leaks are a prime source of groundwater and soil contamination. The United States Environmental Protection Agency (EPA) estimates that there are presently 5 – 7 million underground storage tanks in the United States, and about 1.4 million of these are used to store petroleum fuel products. Approximately 1.6 million tanks are currently regulated by the EPA. Soxhlet and sonication techniques have been traditionally used to monitor the petroleum hydrocarbon contamination of soils and sediments caused by underground storage tank leakage. Unfortunately, these conventional extraction methods are labor intensive and require large volumes of solvent.

Pressurized solvent extraction can be used to replace Soxhlet and sonication techniques and is approved for use as EPA Method 3545A. This application describes a procedure for extracting petroleum hydrocarbon contaminants such as diesel and waste oil in soils with significant time-savings and solvent reduction. The method is applicable to the extraction of waste oil organics (WOO), diesel range organics (DRO), and total petroleum hydrocarbons from soils.

one PSE #217 APPLICATIONS






Equipment

- ✓ Applied Separations' Pressurized Solvent Extractor
 - *one* PSE or *fast* PSE
- ✓ 11mL Extraction Vessel-Cat. #10625
 - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ Gas chromatograph with flame ionization detector (GC-FID)
- ✓ Analytical Balance
- ✓ Sample concentrator

Solvents and Materials

- ✓ Acetone
- ✓ Methylene chloride
- ✓ *n*-Tricosane (C₂₃) as surrogate
- ✓ *Spe-ed*TM Matrix- Cat. #7950
- ✓ Collection Vials (60mL for extract collection)-Cat. #10650
- ✓ Cellulose Disk-Cat. #10711
- ✓ Ottawa Sand-Cat. #10584
- ✓ 10 x 250 mm chromatographic columns or disposable pipettes
- ✓ 100mL beaker
- ✓ Silica gel-100/200 mesh, Davisil grade 634 or equivalent

Summary of Method

 <p>1. Prepare Sample</p>	  <p>2. Load Sample</p>
 <p>3. Run Sample</p>	 <p>4. Collect Extract</p>

Procedure

Prepare Sample

Discard any foreign objects such as sticks, leaves, and rocks. Mix the sample thoroughly, especially composited samples. Inspect the dry sample. If the sample particle size is too large, grind or otherwise reduce the particle size of the soil so that it passes through a 1mm sieve. Soil samples that are wet should be weighed into a 100-mL beaker and mixed with *Speed*TM-Matrix.

*Note: Speed*TM-Matrix should be purified by heating at 400°C for four hours in a shallow tray or by extraction with methylene chloride. The silica gel should be activated by heating for at least 16 hours at 130°C in a shallow glass tray, loosely covered.

Place disposable cellulose filter into extraction vessel, on top of the vessel outlet frit. Place prepared sample into the vessel. Add 1-mL of *n*-tricosane to sample in extraction vessel.

Load Sample

Place the extraction vessel into the instrument as described in the *onePSE* operator's manual. Place a pre-cleaned collection vial in the instrument for each sample. Ensure that the collection vial is sufficiently large to hold the extract.

Run Sample

Select the appropriate stored extraction program and start.

Extraction Conditions

Program the following extraction parameters on the one PSE

Program A Mode – 1 mL vessel

Oven temperature:	175°C
Pressure:	130 BAR
Static time:	5 minutes
Cycles	1
Solvent*:	Methylene chloride/acetone (1:1, v/v)
Purge:	N=2
Flush program:	Solvent/gas/repeat flush:20 sec/2min/0

Optimize the conditions as needed. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the samples. Any pressure in the range of 100 BAR should suffice.

Once established, the same parameters should be used for all samples extracted for the same analysis type.

Collect Extract

Collect each extract in a clean 60mL vial. Allow the extract to cool after the extraction is complete.

Extract Cleanup

If it is necessary to remove coextracted interferants from the sample use EPA Method 3630. Weigh out 2-10 g of activated silica gel and place in a 10-mm ID glass chromatographic column. Top silica gel with 4 to 5 cm of sodium sulfate. Note that the amount of sodium sulfate will vary according to the moisture content of the sample. Transfer the sample extract onto the column. Rinse the collection vial two or three times and transfer each rinse to the column. Elute the column with sufficient hexane or methylene chloride to ensure recovery of the analytes (see EPA Method 8440 for guidance).

Analysis

After cleanup, the samples are ready for volume adjustment and further analysis. The samples can be analyzed by employing EPA Method 8015 (GC/FID).

References

US EPA Method 3545A – Pressurized Fluid Extraction
US EPA Method 8015- Nahalogenated Volatiles (GC/FID)

Safety

The use of organic solvents, elevated temperatures, and high pressures present potential safety concerns in the laboratory. Common sense laboratory practices can be employed to minimize these concerns. However, the following sections describe additional steps that should be taken.

Extraction cells in the oven are hot enough to burn unprotected skin. Allow the cells to cool before removing them from the oven or use appropriate protective equipment (e.g., insulated gloves or tongs), as recommended by the manufacturer.

During the gas purge step, some solvent vapors may exit through a vent port in the instrument. Connect this port to a fume hood or other means to prevent release of solvent vapors to the laboratory atmosphere.