

## **Extraction of Organochlorine Pesticides from Soils by Pressurized Solvent Extraction (PSE)**

### **Introduction**

Organochlorines include such compounds as chlordane, toxaphene, dieldrin, polychlorinated biphenyls, DDT, hexachlorobenzene, and hexachlorocyclohexanes. These toxic compounds are still present throughout the environment. The presence of organochlorines in soil is a pressing environmental concern that calls for efficient methods of regulation. Unfortunately, traditional methods of extraction are time consuming, labor intensive and require the use of large volumes of organic solvent.

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. Pressurized solvent extraction can be used to replace Soxhlet and sonication techniques and is approved for use as EPA Method 3545A.

This application describes the rapid extraction of organochlorine pesticides from soils, clays, sediments, sludges, and waste solids using pressurized solvent extraction. The *fast* PSE is an automated system that processes six samples simultaneously. The parallel processing technology of the *fast* PSE dramatically increases sample throughput compared to Soxhlet or pressurized solvent extraction systems that employ serial processing. In addition to rapid extraction times, significant reduction in solvent consumption is achieved.

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## Equipment

- ✓ Applied Separations' *fast* PSE Pressurized Solvent Extractor
- ✓ 11 mL Extraction Vessels-Cat.#10625
  - Note: the *fast* PSE can run 6 samples simultaneously
- ✓ GC or GC/MS
- ✓ Oven – for drying
- ✓ Desiccator
- ✓ Crucibles
- ✓ Grinding Apparatus
- ✓ Analytical Balance




## Solvents and Materials

- ✓ Acetone (pesticide grade)
- ✓ Hexane (pesticide grade)
- ✓ N<sub>2</sub> – high purity grade
- ✓ S/S Frits (10 micron)- Cat. #10710
- ✓ Collection Vials (60mL for extract collection)-Cat.#10650
- ✓ Cellulose Filter Disk-Cat. #10711
- ✓ *Spe-ed*<sup>TM</sup> Matrix-Cat.#7950
- ✓ Ottawa Sand–Cat. #10548

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## Summary of Method

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

## Procedure

### *Sample Preparation*

Sediment/soil samples: Decant and discard any water layer on a sediment sample. Mix the sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks. Air-dry the sample at room temperature for 48 hours in a glass tray or on hexane-rinsed aluminum foil. Alternatively, mix the sample with an equal volume of *Spe-ed* Matrix (pelletized diatomaceous earth) until a free-flowing powder is obtained.

**Note:** Dry, finely ground soil/sediment allows the best extraction efficiency for nonvolatile, nonpolar organics. Air-drying may not be appropriate for the analysis of the more volatile organics because of losses during the drying process. The use of sodium sulfate as a drying agent can lead to clogging of the frits in the vessel with recrystallized sodium sulfate.

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Dry sediment and soil samples amenable to grinding. Grind or otherwise reduce the particle size of the sample so that it either passes through a 1-mm sieve or can be extruded through 1-mm hole. Disassemble grinder between samples, according to manufacturer's instructions, and decontaminate with soap and water, followed by acetone and hexane rinses.

Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The analyst may add anhydrous sodium sulfate, *Spe-ed* Matrix (pelletized diatomaceous earth), Ottawa sand, or other clean, dry reagents to the sample to make it more amenable to grinding.

Grind a sufficient weight of the dried sample to yield the sample weight needed for the determination method (usually 10-30g). Grind the sample until it passes through a 10-mesh sieve.

### ***Determination of Percent Dry Weight***

When sample results are to be calculated on a dry weight basis, a second portion of sample should be weighed at the same time as the portion used for analytical determination.

***Warning:*** *The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated sample.*

Immediately after weighing the sample for extraction, weigh 5-10g of the sample into a tared crucible. Dry this aliquot overnight at 105°C. Allow to dry in a desiccator before weighing. Calculate the % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100\%$$

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## ***Load Sample***

Prepare the extraction vessels for analysis by placing a cellulose filter disk in the bottom opening followed by a 10µm s/s frit, and secure them in place with a retaining nut. Transfer the ground sample to an extraction vessel of the appropriate size for the aliquot. Generally, an 11mL vessel will hold about 10g of material, a 22mL vessel will hold about 20g of material, and a 33mL vessel will hold about 30g of material. The weight of a specific sample that a vessel will contain depends upon the bulk density of the sample and the amount of drying agent that must be added to the sample in order to make it suitable for extraction. Analysts should ensure that the sample aliquot extracted is large enough to provide the necessary sensitivity and choose the extraction vessel size accordingly. Matrix spikes and surrogate spikes may be added to the appropriate extraction vessels as required. Add clean Ottawa sand to within 1 cm of the top of the vessel's interior flange as directed by the User's Manual.

Once all extraction vessels are packed, place the extraction vessels into the instrument as described in the *fast* PSE operator's manual. Place a pre-cleaned collection vial in the instrument for each sample. Ensure that the pump is primed and that the extraction solvent is acetone/hexane (1:1, v/v). Program the instrument using the following parameters:

## ***Extraction Conditions***

*Program the following extraction parameters on the fast PSE*

Program A Mode – 11mL vessels

Solvent:	Acetone/Hexane (1:1, v/v)
Temperature:	100 ° C
Pressure:	100 Bar
Static:	5 minutes
Solvent Module:	1*
Cycles:	1
Pause:	N=0
Flushing Program:	Solvent/gas/repeat flush: 1min/ 2 min/ 0



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**\*Note:** *If automatic solvent selection module is used, enter the appropriate position number (i.e. 2,3, or 4).*

Optimize the conditions as needed. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction vessel 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.

The extract is now ready for concentration, cleanup, or analysis, depending on the extent of interferants and the determinative method to be employed. Refer to EPA Method 3600 for guidance on selecting appropriate cleanup methods. Excess water present in extracts may be removed by filtering the extract through a bed of anhydrous sodium sulfate. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

### **References**

US EPA Method 3545 – Pressurized Fluid Extraction  
US EPA Method 3600 – Clean up

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## 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 vessels in the *fast* PSE oven are hot enough to burn unprotected skin. Allow the vessels 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. This precaution also applies to the removal of post extraction solvent from the collected extract.