

Application Note 28

Cleanup of Hazardous Waste Site Samples by Solid Phase Extraction

ENVI-Florisil solid phase extraction tubes meet the requirements for pesticide recovery from water, sediment, and soil samples taken from hazardous waste sites, as described in US EPA Contract Laboratory Program methodology. Mean recovery of nine pesticides was 82% or better, well within the specifications of the CLP method. A procedure for recovering the pesticides is described.

Key Words

• pesticides • solid phase extraction • CLP methods

The US Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) method for monitoring pesticides in water, sediment, and soil samples from hazardous waste sites specifies use of Florisil® solid phase extraction cartridges with stainless steel or Teflon® frits for sample cleanup (1). This mandatory cleanup procedure significantly reduces matrix interferences caused by polar compounds.

ENVI™-Florisil solid phase extraction tubes fulfill the pesticide recovery requirements of the EPA CLP pesticide method. These 6mL polypropylene tubes with stainless steel frits contain 0.5g or 1.0g of Florisil packing. The bed size used depends on the final extract volume required for injection. Manual injection requires a 1.0mL final extract volume and a 0.5g tube. If your autosampler operates reliably with 1.0mL of final extract, use a 0.5g tube, otherwise, prepare 2.0mL of sample extract using a 1g tube.

Table 1. Pesticide Recovery After Extraction on ENVI-Florisil SPE Tubes

Compound	Concentration (ng/mL)	Recovery (%)	
		1.0g Bed	0.5g Bed
α-BHC	25	81.8	90.0
γ-BHC	25	87.3	92.4
Heptachlor	25	90.6	89.2
Endosulfan I	25	91.8	101.6
Dieldrin	50	94.9	92.4
Endrin	50	108.2	93.7
4,4'-DDD	50	104.9	96.1
4,4'-DDT	50	95.6	102.0
Methoxychlor	250	110.0	91.7

To demonstrate the suitability of ENVI-Florisil tubes for the CLP pesticide method, we measured recoveries of the nine pesticides specified in the CLP method, for use as a cartridge performance check (Table 1). Samples of a standard containing the nine pesticides were prepared and concentrated as specified in the CLP method.

The ENVI-Florisil tubes were conditioned with 5mL of 90:10 (v/v) hexane:acetone, then 0.5mL of the pesticide mix was applied. The sample was eluted with 9.0mL of the hexane:acetone solution at a slow, dropwise rate, followed by 2mL of hexane. The eluant was concentrated to 1.0mL. GC analysis was performed on an SPB™-608 capillary column (30m x 0.53mm ID 1.0µm film).

Table 1 summarizes recovery data for tubes containing 0.5g and 1.0g beds of a typical lot of Florisil packing at mid-point analyte concentration levels. Mean recoveries were also determined for five lots of 0.5g tubes using the same procedure. Both studies confirmed that ENVI-Florisil tubes satisfy the CLP pesticide method requirement that target compounds be recovered at 80 to 120%.

ENVI-Florisil tubes are specifically designed and tested to ensure reduced matrix interferences and high analyte recovery for the EPA CLP pesticide method. We also offer a complete selection of products — including solid phase extraction tubes, columns, and standards — for other EPA methods. See our latest catalog for more information.

Ordering Information

Description	Cat. No.
ENVI-Florisil Tubes, 6mL, stainless steel frits	
1.0g pk. of 30	57053
0.5g pk. of 30	57046

Pesticide Individual Standard Mix A

5µg/mL each: α-BHC, γ-BHC, heptachlor, endosulfan I;
 10µg/mL each: dieldrin, endrin, 4,4'-DDD, 4,4'-DDT;
 50µg/mL methoxychlor. In 1mL hexane.

48796

SPB-608 Fused Silica Capillary Column

30m x 0.53mm ID, 1.0µm film

made-to-order

Reference

- US EPA Contract Laboratory Program Statement of Work for Organic Analysis, Multimedia Multiconcentration, Doc. No. OLM01.0.

Trademarks

ENVI, SPB — Sigma-Aldrich Co.

Florisil — U.S. Silica Co.

Teflon — E. I. du Pont de Nemours & Co., Inc.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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