

Use Solid Phase Extraction to Isolate Phenols from Aqueous Samples

ENVI-Chrom P is a highly crosslinked styrene-divinylbenzene resin that effectively extracts polar aromatic compounds from water samples. Compared with silica-based packings and traditional resin-based packings, ENVI-Chrom P packing yields superior recovery and selectivity, with less pretreatment.

Key Words:

- phenols
- polar compounds
- solid phase extraction
- ENVI-Chrom P tubes

Solid phase extraction (SPE) methods have been developed for many hydrophobic analytes, but extractions of hydrophilic phenols are difficult because these compounds have a strong tendency to hydrogen bond with water molecules. Extraction of phenols using silica-based SPE packings has yielded both poor selectivity and low recoveries for these polar compounds (1). Resin-based packings have required extensive pretreatment that is time-consuming and generates waste solvent (2). Because of these difficulties, Supelco environmental research scientists have evaluated a highly crosslinked, specially cleaned styrene-divinylbenzene resin, ENVI™-Chrom P, which was developed specifically for extraction of polar aromatic compounds from aqueous samples.

ENVI-Chrom P packing consists of small, nonionic, styrene-divinylbenzene copolymer beads (Table 1) that offer a much greater surface area than typical silica-based packings. The numerous active aromatic sites on the resin allow significant interaction with the aromatic phenols, improving recovery rates for many of these compounds. Background levels are negligible.

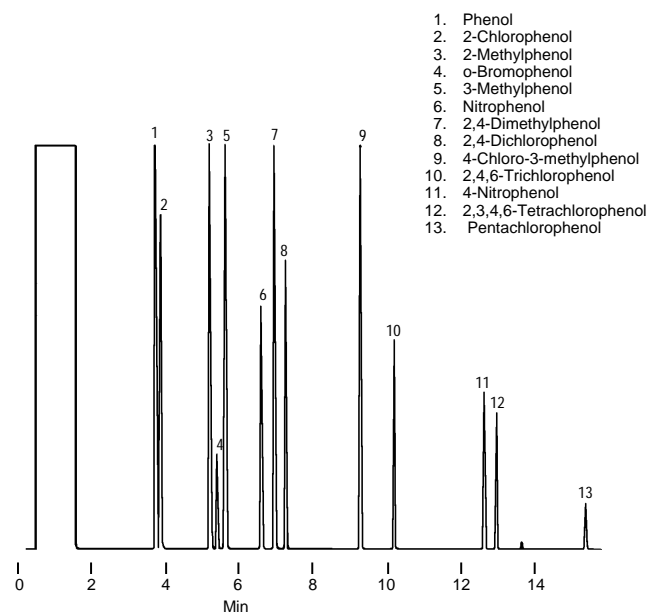
Table 1. Physical Characteristics of ENVI-Chrom P Styrene-Divinylbenzene Adsorbent

Resin:	nonionic, highly crosslinked styrene-divinylbenzene copolymer
Particle Size:	80-160µm
Surface Area:	800-950m ² /g
Mean Pore Size:	110-175Å

We extracted 12 US EPA-regulated phenols from aqueous samples, using ENVI-Chrom P SPE tubes and tubes containing a cyclohexyl bonded silica-based packing. The ENVI-Chrom P packing provided excellent recoveries. By following the method outlined in Table 2, mean recovery rates for these phenols were greater than 96% over a 1.0-2.5mg/liter concentration range (Table 3). Recovery of these analytes began to drop only at a sample concentration of 5.0mg/liter, which is twice the maximum concentration required by proposed EPA methods (1).

Figure A. Phenols Extracted from Aqueous Sample, Using ENVI-Chrom P SPE Tube

Column: PTE™ QTM, 15m x 0.53mm ID, 0.5µm film
Cat. No.: 25355
Oven: 65°C to 185°C at 10°C/min (hold 1 min) to 275°C at 20°C (hold 5 min)
Det.: FID, 330°C
Inj.: 1µL extract (see Table 2), 200°C, splitless injection, hold 45 sec



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Breakthrough capacities for both analyte and sample volume met these proposed requirements. ENVI-Chrom P tubes also introduced minimal additional background (Figure A).

ENVI-Chrom P tubes and the method in Table 2 eliminate the need for sample pH adjustments (and thus the need to handle strong acid solutions) and for "salting out." Elimination of salting out removes two potential sources of problems: the possibility of salt precipitation in the GC injection port and the possibility of contamination from salt stock solutions.

ENVI-Chrom P solid phase extraction tubes offer excellent extraction efficiency and reproducible recoveries of phenols from aqueous samples. These tubes are in stock, available for rapid delivery.

Table 1. Phenol Extraction from Aqueous Samples, Using ENVI-Chrom P SPE Tubes

SPE Tube:	ENVI-Chrom P, 6mL/250mg
Tube Conditioning:	6mL methyl t-butyl ether 6mL methanol 6mL deionized water Do not allow packing to dry between solvent washes or before adding sample.
Sample Addition:	Maximum of 100mL aqueous sample. Sample containing fine sediment may require prefiltration.
Drying:	Vacuum suction, 2 minutes maximum. (For completely dry samples, pass eluted sample through bed of sodium sulfate. Recoveries will be lower.)
Sample Elution:	Add 2mL methyl t-butyl ether, w/vacuum off. Allow solvent to wet packing and soak for 1 minute. • Add 2mL methyl t-butyl ether and allow elution to proceed at a dropwise rate. • Add methyl t-butyl ether until 5mL of eluant is collected in a volumetric flask. Sample volume may be carefully reduced, if necessary, but avoid volatilizing extracted phenols. Ethyl acetate may be substituted for methyl t-butyl ether.

Table 3. High Recovery Rates for Phenols, Using ENVI-Chrom P SPE Tubes

Analyte (Priority Pollutant Phenols)	Mean Recovery (% ± Coef. Variation)	
	ENVI-Chrom P Packing (n = 4 analyses)	Cyclohexyl-Bonded Silica (n = 5 analyses)
Phenol	101.8 ± 3.7	74.1 ± 8.5
2-Chlorophenol	104.7 ± 1.7	—
2-Methylphenol	107.0 ± 1.9	94.0 ± 2.4
o-Bromophenol	115.3 ± 1.4	90.9 ± 5.8
3-Methylphenol	104.9 ± 1.6	95.2 ± 2.2
2-Nitrophenol	96.3 ± 2.8	93.4 ± 2.0
2,4-Dimethylphenol	105.3 ± 2.2	95.0 ± 1.7
2,4-Dichlorophenol	106.3 ± 2.2	94.3 ± 2.2
4-Chloro-3-methylphenol	104.6 ± 2.0	96.4 ± 1.8
2,4,6-Trichlorophenol	103.9 ± 2.1	89.2 ± 3.9
2,4-Dinitrophenol	<30.0 [▲]	105.9 ± 1.7
4-Nitrophenol	99.9 ± 4.1	84.6 ± 19.8
2,3,4,6-Tetrachlorophenol	104.6 ± 1.5	80.6 ± 9.1
2-Methyl-4,6-dinitrophenol	<30.0 [▲]	98.0 ± 5.4
Pentachlorophenol	97.7 ± 4.8	72.9 ± 13.9

▲ Method not appropriate for extraction of this compound.

Tube: ENVI-Chrom P, 6mL/0.25g (3 lots of packing)
Cyclohexyl-bonded silica, 3mL/0.50g

Sample: 100mL water, pH 5.0 (ENVI-Chrom P tubes)
100mL water/20g NaCl, pH 2.3 (silica tubes)

Analyte Concentration: 2.5mg/L

Ordering Information:

ENVI-Chrom P SPE Tubes

3mL/0.25g, pk. of 54	57224
6mL/0.25g, pk. of 30	57225-U
6mL/0.5g, pk. of 30	57226

PTE-5 QTM Fused Silica Capillary Column

15m x 0.53mm ID, 0.5µm film	25355
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Contact our Technical Service Department

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References

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References not available from Supelco.

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