Application Note 65

ENVI-18 SPE Tube Ensures Low Background for Monitoring Organic Compounds in Drinking Water by EPA Method 525

ENVI-18 solid phase extraction tubes help ensure rapid, economical, reliable sample preparation and provide excellent analyte recovery and clean chromatograms in the screening or monitoring of drinking water samples.

Key Words:
- ENVI-18 SPE tubes • organic contaminants
- drinking water • EPA Method 525

Based on an ongoing review of sample preparation techniques, the US Environmental Protection Agency (EPA) has designated solid phase extraction cartridges for the cleanup and concentration of organic contaminants from drinking water samples (1,2).

ENVI™-18 SPE tubes are compatible with the large sampled volume (1 liter) and low pH (pH 2) specified in EPA Method 525. They can consistently accept large samples and low pH without loss of phase from the silica support. EPA precautions warn that, if phase is stripped from the extraction tube packing at pH 2, the chromatographic analysis can be complicated by high background (Figure A). High background can obscure compounds of interest. In contrast, the specially prepared packing in ENVI-18 tubes meets the background specifications set by the EPA for extraction analysis by GC-MS (Figure B). ENVI-18 tubes also ensure consistent, high compound recovery .

Supelco has also developed procedures for extracting several classes of environmental contaminants from water samples — phthalate esters, organochlorine pesticides and triazine herbicides, and polynuclear aromatic hydrocarbons — using drinking water samples spiked with contaminants (Tables 1 and 2).

To significantly reduce preparation time, sample volumes were kept at 250mL. These smaller samples can also reduce the costs and inconveniences of sampling, transporting, and storing larger

Figure A. GC-FID Chromatograms: ENVI-18 Phase Is Not Stripped from the Support at pH 2

Sample: 1 liter water
Extraction Tube: Supelclean ENVI-18, 6mL, 0.5g packing
Cat. No.: **57064**
Column: **PTE-5 QTM fused silica, 15m x 0.53mm ID, 0.50µm film**
Cat. No.: **25355**
Inj.: 1µL

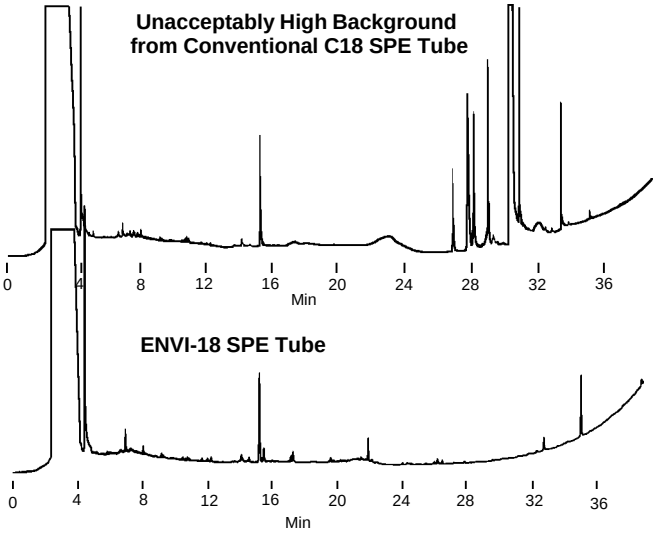

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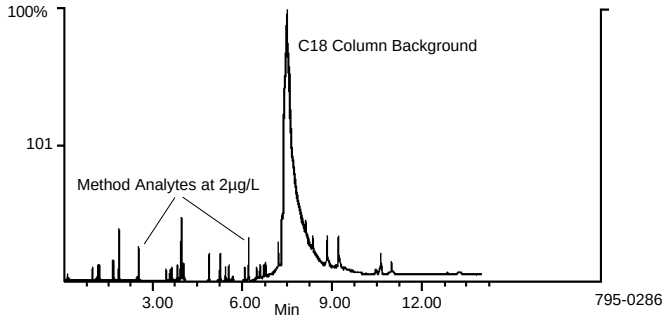
Figure B. Total Ion Chromatograms from Extracted Blank SPE Tubes

Conditions for Figure B2
Sample: 1 liter water
Extraction Tube: Supelclean ENVI-18, 6mL, 0.5g packing
Cat. No.: **57064**
Column: **PTE-5 fused silica, 30m x 0.25mm ID, 0.25µm film**
Cat. No.: **24135-U**
Col. Temp.: 40°C (1 min) rapidly to 160°C (3 min) to 320°C at 6°C/min
Carrier: helium, 33cm/sec
Inj.: see Figure A

B1 — Conventional C18 Tube — Unacceptable Background*



B2 — ENVI-18 Tube

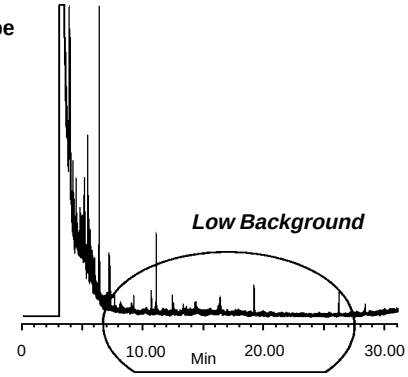


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*Figure B1 adapted from Reference 1.

samples. Optimized methods for these smaller sample volumes ensure that elution volumes are reduced – yet compounds of interest can be easily analyzed and quantified without excessive solvent consumption or prolonged eluate concentration times.

Table 1. Procedures for Extracting Organic Compounds from Drinking Water, Using ENVI-18 SPE Tubes (500mg packing bed)

Phthalates	
Conditioning:	2 x 6mL methylene chloride:methanol (1:1) 6mL methanol 6mL deionized water
Sample Addition:	250mL water, unadjusted pH passed slowly through tube (10mL/min)
Drying:	5-10 min
Elution:	2 x 1.5mL methylene chloride:methanol (1:1)* concentrate extract to 1mL
Total Time:	30-40 min
Pesticides/Herbicides	
Conditioning:	2 x 6mL hexane:ethyl ether (1:1) 6mL methanol 6mL deionized water
Sample Addition:	250mL water, unadjusted pH passed slowly through tube (10mL/min)
Drying:	5-10 min (or use sulfate tube)
Elution:	2 x 1.5mL hexane:ethyl ether (1:1)* concentrate extract to 2mL
Total Time:	30-40 min
Polynuclear Aromatic Hydrocarbons	
Conditioning:	2 x 6mL toluene:methanol (10:1) 6mL methanol 6mL deionized water
Sample Addition:	250mL water, unadjusted pH passed slowly through tube (10mL/min)
Drying:	5-10 min
Elution:	2 x 1mL toluene:methanol (10:1)
Total Time:	30-40 min

* Extract can be passed through a bed of sodium sulfate, to remove traces of water, before concentration.

With our extraction procedures, recovery exceeded 90% for almost every compound spiked into the water samples (Table 2). We noted significant recovery variation in only one case. In analyses of PAHs, recovery of acenaphthylene appeared to depend on the drying step before elution. If the packing bed was slightly moist, recovery was 90% or greater. If the bed was allowed to dry completely, recovery was reduced.

Excellent analyte recovery and very clean chromatograms can be obtained by using ENVI-18 SPE tubes. If you are screening drinking water samples according to EPA Method 525 – or monitoring phthalates, organochlorine pesticides, triazine herbicides, or PAHs in drinking water – Supelco's ENVI-18 SPE tubes will help ensure rapid, economical, reliable sample preparation.

Reference 1 available from: NTIS, 5285 Port Royal, Springfield, VA USA 22161 (order number PB 89-220-461).

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

Table 2. Recovery of Organic Compounds

Compound	Recovery (mean % ± CV)**
Phthalates (20µg/L)	
Dimethyl phthalate	93 ± 5.9
Diethyl phthalate	97 ± 4.6
Di-n-butyl phthalate	97 ± 6.8
Benzyl butyl phthalate	100 ± 5.4
Bis(2-ethylhexyl)adipate	99 ±11
Bis(2-ethylhexyl)phthalate	111 ± 8.3
Pesticides (8µg/L)	
Hexachlorobenzene	87 ±11
Lindane	99 ±13
Heptachlor	96 ±12
Aldrin	94 ±13
Heptachlor epoxide	98 ±13
Endrin	93 ±11
Methoxychlor	110 ±13
Simazine	89 ± 5.1
Atrazine	100 ± 5.1
Polynuclear Aromatic Hydrocarbons (20µg/L)	
Acenaphthylene	66 ±28
Fluorene	95 ± 7.5
Phenanthrene	95 ± 8.0
Anthracene	95 ±11
Pyrene	97 ± 7.1
Benzo(a)anthracene	94 ± 8.3
Chrysene	101 ± 8.8
Benzo(a)fluoranthene	103 ±10
Benzo(k)fluoranthene	103 ± 9.4
Benzo(a)pyrene	94 ±11
Indeno(1,2,3-cd)pyrene	102 ±10
Dibenzo(a,h)anthracene	104 ± 9.7
Benzo(ghi)perylene	101 ±11

**Mean for 5 extractions.

Ordering Information:

Description	Cat. No.
ENVI-18 Solid Phase Extraction Tubes	
100mg (1mL), box of 100	**57062**
500mg (3mL), box of 50	**57063**
500mg (6mL), box of 30	**57064**
1000mg (6mL), box of 30	**57065-U**
Capillary GC Columns	
PTE™-5 column 30m x 0.25mm ID x 0.25µm film	**24135-U**
PTE-5 QTM column 15m x 0.53mm ID x 0.50µm film	**25355**
Chemical Standards	
Phthalate Esters Mix 525***	**48223**
Polynuclear Aromatic Hydrocarbons Mix 525***	**48953-U**

***Components are listed in Table 2.

References

1. Method 525. Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry (Revision 2.1), Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH USA 45268.
2. National Primary Drinking Water Regulations; Analytical Techniques 40 CFR Parts 141 and 143 (Final Rule), Federal Register 53 (No. 33), 5142-5147 (Feb. 19, 1988).

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