Application Note 29

Purge Trap for GC Analyses of Volatile Aromatic (BTEX) Compounds in Soils and Sediments

The BTEXTRAP purge trap was designed specifically for monitoring benzene, toluene, ethylbenzene, and the xylene isomers (BTEX compounds). The trap allows large amounts of methanol (used in the US EPA Method 5030 procedure for preparing soil and sediment samples) to pass through the adsorbent beds while retaining the analytes of interest. In evaluations of trap performance, relative analyte recovery was consistent for samples containing a low level and a high level of added methanol. (ChromFax No.: 394029)

Key Words:

- BTEX compounds • volatile organic compounds
- purge and trap • soil analyses • sediment analyses

Analysts who monitor hazardous waste sites for volatile organic compounds (VOCs) under the requirements of the Resource Conservation and Recovery Act Method SW-846 must concentrate their samples prior to chromatographic analysis. Purge and trap is a widely used preparative procedure for the extraction and concentration of VOCs, including benzene, toluene, ethylbenzene and xylene (BTEX), in soil, sediment, and water matrices. For solid wastes, such as soils and sediments, the purge and trap method (US EPA Method 5030) requires that a portion of the sample be dispersed in methanol to dissolve the volatile organic constituents (1). The methanolic solution is diluted with water in a specially designed purging chamber, to obtain estimated analyte concentrations within 10 to 200ppb. It is then analyzed by purge-and-trap GC following the normal method for water — volatile compounds are purged from the water sample, trapped on an adsorbent trap, and delivered to the gas chromatographic column by thermal desorption.

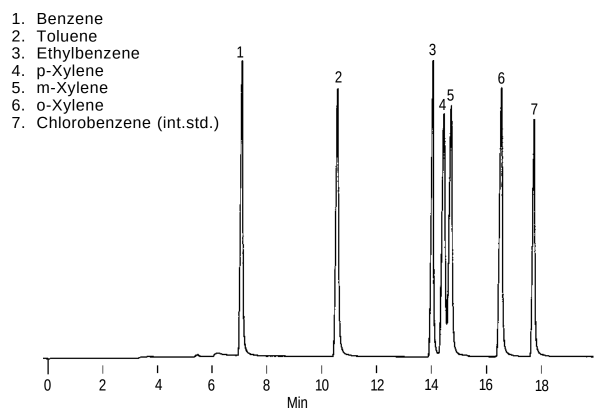
This preparative method results in a large methanol concentration which is then purged with the analytes. If retained by the purge trap, the methanol will create a huge solvent peak that will interfere with the chromatographic analysis of benzene. This is especially evident when using a capillary column designed to separate the para- and meta-xylene isomers, and an FID detector. However, large volumes of methanol can also quench the signal of a PID detector.

The BTEXTRAP™ purge trap was developed specifically for monitoring benzene, toluene, ethylbenzene, and the xylene isomers. This unique purge trap allows large volumes of methanol to pass through the adsorbent beds during the purging process, while retaining the analytes of interest, making it ideal for monitoring these compounds in soil and sediment matrices.

Figure A. Volatile Aromatic Compounds Collected on a BTEXTRAP Purge Trap

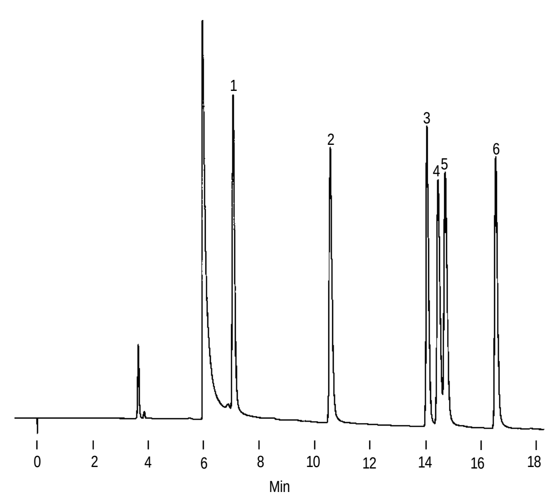
Sample:	A1 - 5mL, 10ng/mL each analyte A2 - 25µL methanol added
Trap:	**BTEXTRAP**
Purge:	11 min
Dry Purge:	5 min
Purge Flow Rate:	40mL/min
Desorb.:	260°C/6 min
Bake:	270°C/4 min
Column:	**SUPELCOWAX 10, 60m x 0.75mm ID, 1.0µm film**
Cat. No.:	**23723**
Oven:	50°C (8 min) to 100°C at 4°C/min
Carrier:	helium, 10mL/min
Det.:	FID

Low Methanol Concentration (<1µL/mL)



713-0001

High Methanol Concentration (5µL/mL)



712-0724

The BTEXTRAP bed structure was designed for optimum performance under the standard operating parameters specified in many VOC purge and trap methods. These methods typically specify a sample volume of 5mL, a purge time of 11 minutes, and purge flow rates of 25 to 40mL/min. We limited drying time to 5 minutes to reduce analysis time. This drying time provides added removal of methanol after the purge is complete. Because the Carbopack™ adsorbents used are hydrophobic, water retention is not a concern.

A 30mg bed of Carbopack B is sufficient to fully retain benzene in the 11 minute purge and 5 minute dry times, yet does not retain significant amounts of methanol. A 300mg bed of Carbopack C is required to fully retain the other, larger compounds without allowing them to break through to the Carbopack B bed. The Carbopack C bed also prevents high molecular weight contaminants from being adsorbed on the Carbopack B bed. (For monitoring compounds more volatile than benzene, we recommend our VOCARB 3000 and VOCARB 4000 traps, which include an additional adsorbent, Carboxen™-1000). The thermal stability of these advanced materials allows the use of higher temperatures in the desorption and baking cycles, for better focusing of analytes and elimination of analyte carryover to subsequent analyses.

Performance of the BTEXTRAP purge trap was evaluated using a mixture of volatile aromatic compounds containing methanol in low (<1µL/mL water) and high (5µL/mL water) concentrations (Figure A). We obtained excellent resolution of benzene, toluene, ethylbenzene and the xylene isomers with minimal solvent interference. Three analyses were made at five analyte concentrations (Tables 1 and 2). The elevated methanol level had minimal effect on relative analyte recovery (RR) and relative standard deviation (RSD). Correlation coefficients, calculated from the average (three analyses) of the raw area counts at each level for the two methanol levels were, in all cases, greater than 0.9992, indicating that the higher methanol concentration did not appreciably affect linearity.

The BTEXTRAP purge trap is very effective in purge and trap analyses of BTEX compounds. It offers reproducible results and excellent linearity over a wide calibration range. Because its performance is not affected by high methanol concentrations, the BTEXTRAP is ideal for monitoring volatile aromatic compounds in soil in sediment matrices where methanol extraction is required.

Table 1. Reproducibility (%RSD) for Low Methanol Concentration (<1µL/mL)

	Analyte Concentration				
Compound	1 ng/mL	5 ng/mL	10 ng/mL	50 ng/mL	100 ng/mL
Benzene	2.39	1.65	0.86	0.27	0.66
Toluene	0.33	2.36	0.78	1.08	0.85
Ethylbenzene	0.74	1.47	0.92	0.36	1.75
p-Xylene	0.47	1.69	1.24	0.28	1.29
m-Xylene	0.60	1.64	1.15	0.25	1.19
o-Xylene	0.51	0.96	1.79	0.20	0.73

Mean recovery normalized to chlorobenzene internal standard at same concentration (n=3 runs).

Table 2. Reproducibility (%RSD) for High Methanol Concentration (5µL/mL)

	Analyte Concentration				
Compound	1 ng/mL	5 ng/mL	10 ng/mL	50 ng/mL	100 ng/mL
Benzene	4.29	0.27	1.84	0.49	0.45
Toluene	2.77	1.06	1.37	0.33	0.14
Ethylbenzene	2.23	1.11	0.90	0.22	0.07
p-Xylene	2.58	0.87	1.44	0.29	0.12
m-Xylene	1.34	0.51	1.16	0.27	0.08
o-Xylene	1.30	0.70	2.06	0.30	0.07

Mean recovery normalized to chlorobenzene internal standard at same concentration (n=3 runs).

Ordering Information:

BTEXTRAP Purge Traps

Instrument	Cat. No.
CDS Peakmaster	**21158**
Dynatech® "Dyna" models	**21084**
O.I. Analytical 4460	**21145**
O.I. Analytical 4560	**24939**
Tekmar® LSC-1, LSC-2, LSC-2000, 4000	**21064**
Tekmar 3000	**24919**

All traps constructed of 1/8" OD stainless steel and accurately produced to instrument manufacturer's specifications. Thermocouple included on Dynatech and O.I. Analytical traps.

SUPELCOWAX™ 10 Borosilicate Glass Capillary Column

60m x 0.75mm ID, 1.0µm film **23723**

BTEX Mix 1mL **48026**

200µg/mL each component in methanol.

Benzene	Toluene	o-Xylene
Ethylbenzene	m-Xylene	p-Xylene

Reference

1. Solid Waste Methods SW 846 — Purge and Trap (5030A). Available from National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 USA, 703-487-4780.

Contact our Technical Service Department (phone 800-359-3041 or 814-359-3041, FAX 814-359-5468) for expert answers to your questions.