

Adsorbent Trap for GC Analyses of Volatile Organic Compounds in Wastewater

Adsorbents used in VOCARB purge traps offer superior adsorption/desorption characteristics, thermal stability, and hydrophobicity for monitoring volatile organic compounds (VOCs) in water. Response factors for compounds listed in US EPA Method 624 show that VOCARB 3000 traps meet the performance requirements for Methods 624/1624.

Key Words:

- volatile organic compounds • purge and trap
- water analyses

Analysts monitoring wastewater samples for volatile organic compounds (VOCs) according to US EPA Methods 624/1624 must concentrate their samples, using a purge and trap technique, prior to chromatographic analysis. Volatile compounds are purged from the water sample, collected on an adsorbent material, and delivered to the gas chromatography (GC) column by thermal desorption. This type of concentration and sample transfer can present several difficulties. Water vapor can be introduced from the trap to the GC column, causing interference with early-eluting gaseous volatiles. Sample components can be

introduced onto the column as a wide band, instead of a focused plug, making resolution difficult. Careful selection of adsorbent materials can minimize these problems.

US EPA provisions for *best available technology* allow the use of new traps (and analytical columns) not listed in EPA methods, if they meet the QA/QC criteria stated in the method. (Consult your regional EPA office before using a non-specified trap). Advanced materials, such as the Carbotrap™ and Carboxen™ adsorbents used in our VOCARB™ traps, can offer superior adsorption/desorption characteristics, thermal stability, and hydrophobicity. These adsorbents can also be used in combination with traditional materials for traps that are tailor-made for a specific application.

A VOCARB 3000 trap efficiently traps and releases the range of compounds listed in EPA Methods 624 and 1624. Good overall responses for the Method 624/1624 analytes were obtained using a VOCARB 3000 trap. Response factors, calculated with respect to bromochloromethane or difluorobenzene, show that the VOCARB 3000 trap meets performance requirements for Methods 624/1624 (Table 1). The results indicate that VOCs can be analyzed at concentrations of 20 to 200ppb in 5mL of water. In all cases, the relative standard deviation (%RSD) is less than 15% and most results show deviations less than 7%.

Table 1. Response Factors for EPA Method 624 Compounds, Using a VOCARB 3000 Trap (20, 50, 100, 150, 200ppb in 5mL)

Compound	Mean	Std. Dev.	%RSD	Compound	Mean	Std. Dev.	%RSD
Chloromethane	1.014	0.139	13.7	Bromodichloromethane	0.084	0.003	3.4
Vinyl chloride	1.308	0.185	14.1	2-Chloroethylvinyl ether	0.043	0.003	6.2
Bromomethane	1.144	0.108	9.5	cis-1,3-Dichloropropene	1.009	0.070	6.9
Chloroethane	0.519	0.056	10.9	Toluene	1.149	0.151	13.2
Trichlorofluoromethane	1.354	0.213	15.8	trans-1,3-Dichloropropene	0.296	0.011	3.7
1,1-Dichloroethene	1.145	0.139	12.1	1,1,2-Trichloroethane	0.520	0.039	7.5
Methylene chloride	1.270	0.159	12.5	Tetrachloroethene	0.480	0.063	13.2
1,2-Dichloroethene	1.008	0.062	6.1	Dibromochloromethane	0.767	0.039	5.1
1,1-Dichloroethane	2.106	0.066	3.1	Chlorobenzene	1.159	0.048	4.2
Chloroform	2.373	0.104	4.4	Ethyl benzene	0.518	0.030	5.8
Tetrachloroethane	1.356	0.105	7.7	Bromoform	0.961	0.070	7.3
Carbon tetrachloride	1.284	0.124	9.6	1,1,2,2-Tetrachloroethane	0.073	0.006	8.4
Benzene	0.960	0.039	4.0	1,3-Dichlorobenzene	1.304	0.085	6.5
1,2-Dichloroethane	0.047	0.002	4.2	1,4-Dichlorobenzene	1.374	0.084	6.1
Trichloroethene	0.487	0.042	8.6	1,2-Dichlorobenzene	1.281	0.064	5.0
1,2-Dichloropropane	0.533	0.012	2.2				

Table 2. Percent Recovery of Selected VOCs, Using Various Purge Traps (20ppb each compound in 5mL water)

Compound	Trap / Percent Recovery			
	VOCARB 3000 ^a	EPA Method 624 ^b	VOCARB 4000 ^c	EPA Method 524.2 ^d
Gases				
Bromomethane	92	110	72	92
Chloroethane	103	90	95	98
Chloromethane	102	3	102	55
Dichlorodifluoromethane	113	NA	86	54
Vinyl chloride	117	16	100	60
Less-Volatile VOCs				
Bromoform	83	92	77	92
2-Chloroethylvinyl ether	43	56	0	NA
1,3-Dichlorobenzene	97	99	98	98
2,2-Dichloropropane	90	95	78	41
Naphthalene	53	NA	68	78
1,2,4-Trichlorobenzene	84	NA	100	98
1,2,4-Trimethylbenzene	77	NA	92	95

^aCarbopack™ B (10cm) / Carboxen 1000 (6cm) / Carboxen 1001 (1cm)

^bTenax® TA (15cm) / silica gel (8cm)

^cCarbopack C (8.5cm) / Carbopack B (10cm) / Carboxen 1000 (6cm) / Carboxen 1001 (1cm)

^dTenax TA (7.7cm) / silica gel (7.7cm) / charcoal (7.7cm)

The thermal stability of the adsorbents used in the VOCARB 3000 trap allows a higher desorption temperature (250°C) than does the EPA-listed trap (180°C), so that the analytes are released more rapidly, in a focused plug rather than in a broad band, for improved peak shape and resolution. Rapid thermal desorption also may allow use of a low carrier gas flow rate (2mL/min).

A higher desorption temperature can induce thermal and catalytic breakdown of some compounds, however. Although our VOCARB 4000 trap provides slightly better recovery of heavier volatiles, such as naphthalene and the tri-substituted benzenes (listed in US EPA Method 524.2) compared to the VOCARB 3000 trap, the VOCARB 4000 trap catalytically breaks down 2-chloroethylvinyl ether and, to some degree, 2,2-dichloropropane (Table 2). Bromoform and bromomethane show a degree of thermal breakdown. The VOCARB 3000 trap does not promote catalytic breakdown, and causes less thermal breakdown than the VOCARB 4000 trap does.

Choosing an adsorbent trap for your particular application often involves tradeoffs. Compared to other available traps, the VOCARB 3000 provides good overall recovery of volatiles without catalytic breakdown.

Ordering Information:

VOCARB Purge Traps	VOCARB 3000	VOCARB 4000
Instrument	Cat. No.	Cat. No.
CDS Peakmaster	21159	21156
Dynatech® "Dyna" models	21085-U	21083
O.I. Analytical 4460	21131-U	21143
O.I. Analytical 4560	24940-U	24938
Tekmar® LSC-1, LSC-2, LSC-2000, 4000	21066-U	20308
Tekmar 3000	24920-U	24918

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.

VOCOL™ Fused Silica Capillary Column

60m x 0.53mm ID, 3.0µm film 25381

Trademarks

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Dynatech — Dynatech Precision Sampling Corp.

Tekmar — Tekmar Co.

Tenax — Enka Research Institute Arnhem

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

Contact our Technical Service Department
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