Determination of Volatile Organic Compounds in Environmental Samples as per HJ639 using Zebron™ ZB-624*PLUS*™ GC Column

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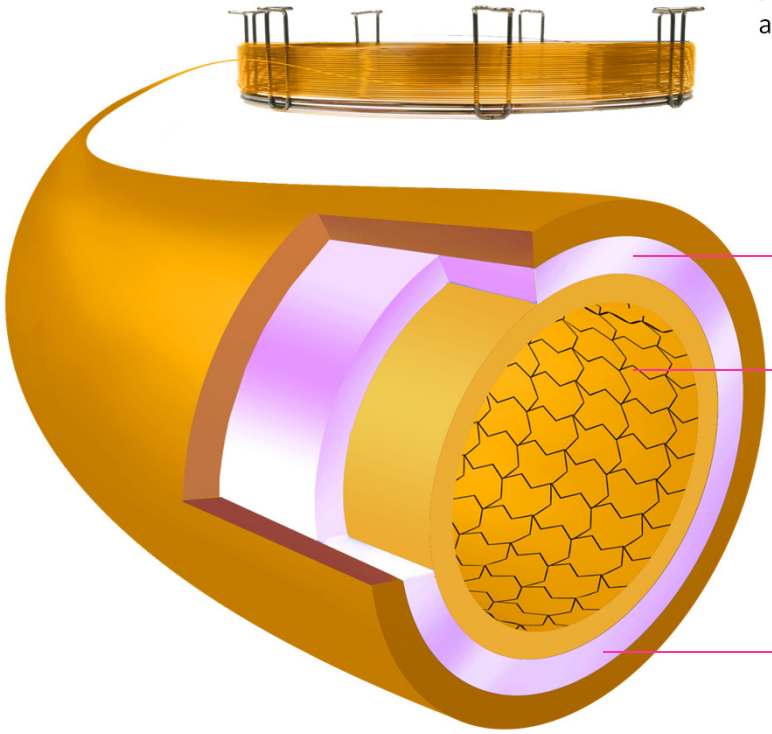
Overview

A common technique for the sampling of Volatile Organic Compounds (VOCs) in water is purge and trap technique. This technique is technically referred to as dynamic headspace extraction as it is an exhaustive extraction and preconcentration technique that allows low level detection of volatile compounds. Purge and Trap in conjunction with a GC-MS is a very powerful analytical technique for volatile organic compounds (VOCs) and BTEX compounds in environmental samples. Due to the expansive list of analytes, the method requires a GC-MS for accurate identification and quantification of analytes that may have chromatographic coelutions.

A 624 selectivity is commonly used for the GC separation. However, traditional GC columns with 624 selectivity are not mass spectrometer compatible and can cause excessive bleed that can contaminate the mass spec source. Presented in this application note are the separation of VOCs using Zebron ZB-624*PLUS*, which is a GC-MS compatible low bleed stationary phase (**Figure 1**). This methodology is adopted from regulation HJ639-2012 and retention data is presented in **Table 1.** The method demonstrated in this application note is precise with less than 4% RSD for peak area, linearity, and accurate with % recovery ranging from 93-110% at concentration levels 5 µg/L and 50 µg/L (**Table 2**). As represented in **Figure 2**, ZB-624*PLUS* provides optimal separation of over 60 VOCs within 27 minutes analysis time. Due to low bleed and MS compatibility of ZB-624*PLUS* stationary phase through Engineered Self Cross-linking™ (ESC™), the column provides steady baseline that facilitates detection of VOC as low as 1 ppb (**Figure 3)**.

Figure 1. Benefits of Zebron™ ZB-624*PLUS* for VOC Analysis

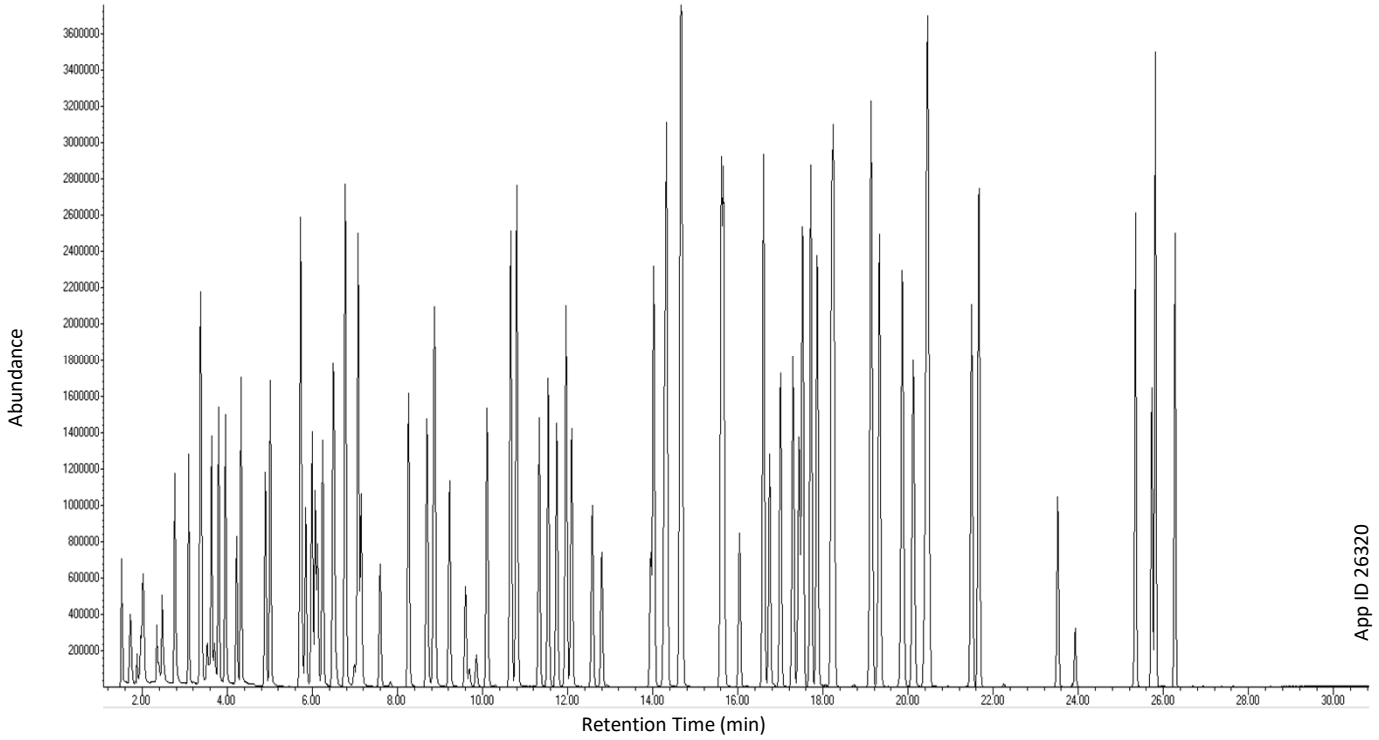


Superior Deactivated Fused Silica
Provides symmetric peak shape for active analytes

Highly Selective Stationary Phase
Retains 624 selectivity for low, high boiling, polar and nonpolar solvents
Extensive ESC™ strengthens the polymer resulting in low bleed and 300/320 °C temperature resistance

Flexible Polyimide Coating
Remains flexible even at 300/320 °C temperature

Figure 1. Volatile Organic Compounds by Purge and Trap GC-MS on Zebron™ ZB-624*PLUS*™ GC Column at 100 ppb



Purge and Trap Conditions

Purge and Trap Concentrator	EST Analytical Evolution 2
Trap Type	K
Valve Oven Temp.	140 °C
Transfer Line Temp.	140 °C
Trap Temp.	35 °C
Moisture Reduction Trap (MoRT) Temp.	39 °C
Purge Time	11 min
Purge Flow	40 mL/min
Dry Purge Temp.	Off
Dry Purge Flow	40 mL/min
Dry Purge Time	1.0 min
Desorb Pressure Control	On
Desorb Pressure	5 psi
Desorb Time	2.0 min
Desorb Preheat Delay	10 sec
Desorb Temp.	250 °C
Moisture Reduction Trap (MoRT) Bake Temp.	250 °C
Bake Temp	210 °C
Sparge Vessel Bake Temp.	110 °C
Bake Time	6 min
Bake Flow	40 mL/min
Purge and Trap Auto-Sampler	**EST Analytical Centurion WS**
Sample Type	Water
Internal Standard Vol.	5 µL
Sample Volume	5 mL

GC-MS Conditions

Column: Zebron ZB-624*PLUS*™
Dimension: 30 meter x 0.25 mm x 1.4 µm
Part No.: 7HG-G040-27
Injection: Split 30:1 @ 220 °C
Carrier Gas: Helium @ 1.0 mL/min (constant flow)
Oven Program: 40 °C for 2 min to 120 °C @ 5 °C/min
for 2 min, to 220 °C @ 10 °C/min for 2 min
Detector: GC-MS
Transfer Line Temperature: 180 °C
Mode Scan (35-300 m/z)
Source Temperature: 230 °C
Quad Temperature: 150 °C
Solvent delay: 0.7 min

Analyte details and retention time of analytes on page 4

Data Courtesy : EST Analytical Inc. Phenomenex is not associated with EST Analytical Inc.

Figure 3. Volatile Organic Compounds by Purge and Trap GC-MS on Zebron™ ZB-624*PLUS*™ GC Column at 1 ppb

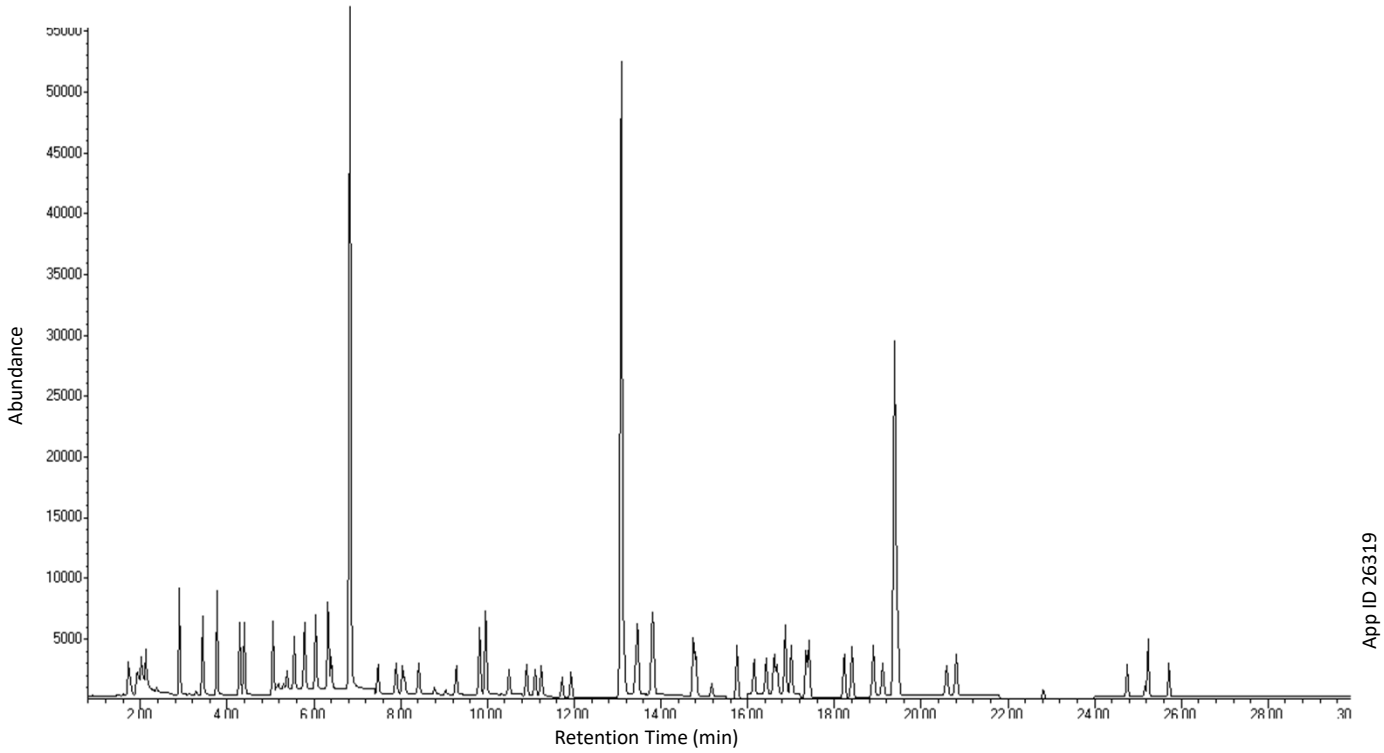


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Table 1. Retention time peak identification of Volatile Organic Compounds

Compound	Average Retention Time (n=7)	Compound	Average Retention Time (n=7)
Vinyl Chloride	2.02	Chlorobenzene-d5 (IS)	13.95
1,1-Dichloroethene	3.36	Chlorobenzene	14.02
Methylene Chloride	3.96	1,1,1,2-Tetrachloroethane	14.28
trans-1,2-Dichloroethene	4.32	Ethylbenzene	14.32
1,1-Dichloroethane	4.9	Xylene (m+p)	14.67
2-chloro-1,3-butadiene	5.01	Xylene (o)	15.61
cis-1,2-Dichoroethene	5.72	Styrene	15.66
2,2-Dichloropropane	5.73	Bromoform	16.04
Bromochloromethane	6.07	Isopropylbenzene	16.6
Chloroform	6.24	BFB SUR	17
Dibromofluoromethane SUR	6.48	Bromobenzene	17.29
1,1,1-Trichloroethane	6.51	1,1,2,2-Tetrachloroethane	17.44
1,1-Dichloropropene	6.77	1,2,3-Trichloropropane	17.52
Carbon Tetrachloride	6.78	n-Propylbenzene	17.71
Benzene	7.08	2-Chlorotoluene	17.87
1,2-Dichloroethane	7.15	4-Chlorotoluene	18.2
Fluorobenzene (IS)	7.59	1,3,5-Trimethylbenzene	18.25
Trichloroethene	8.26	tert-Butylbenzene	19.13
1,2-Dichloropropane	8.69	1,2,4-Trimethylbenzene	19.33
Dibromomethane	8.85	sec-Butylbenzene	19.87
Bromodichloromethane	9.22	1,3-Dichlorobenzene	20.12
1-chloro-2,3-epoxypropane	9.86	1,4-Dichlorobenzene-d4 (IS)	20.4
cis-1,3-Dichloropropene	10.11	Isopropyltoluene	20.45
Toluene-d8 SUR	10.66	1,4-Dichlorobenzene	20.47
Toluene	10.8	1,2,-Dichlorobenzene	21.49
trans-1,3-Dichloropropene	11.33	n-Butylbenzene	21.66
1,1,2-Trichloroethane	11.74	1,2-Dibromo-3-chloropropane	23.52
Tetrachloroethene	11.96	1,2,4-Trichlorobenzene	25.35
1,3-Dichloropropane	12.09	Hexachlorobutadiene	25.73
Dibromochloromethane	12.58	Naphthalene	25.81
1,2-Dibromoethane	12.8	1,2,3-Trichlorobenzene	26.27

Data Courtesy : EST Analytical Inc. Phenomenex is not associated with EST Analytical Inc.

Table 2. Precision, Accuracy and Linearity of Volatile Organic Compounds on Zebron™ ZB-624*PLUS*™

Compound	Calibration Curve RF	Calibration Curve Linearity (%RSD)	Calibration Curve Linearity (R^2)	Precision %RSD (5µg/L)	% Recovery (5µg/L)	Precision %RSD (50µg/L)	% Recovery (50µg/L)
Vinyl Chloride	0.469	6.10	0.999	4.30	93.34	3.49	97.97
1,1-Dichloroethene	0.348	7.69	0.999	3.20	99.11	4.46	105.19
Methylene Chloride	0.433	4.86	1.000	2.30	103.23	3.31	106.19
1,1-dichloroethane	0.772	7.32	1.000	3.60	100.49	3.52	107.51
2-chloro-1,3-butadiene	0.715	7.85	0.999	3.74	98.17	4.24	107.62
trans-1,2-Dichloroethene	0.402	6.13	0.999	3.14	101.40	3.64	107.02
cis-1,2-Dichloroethene	0.468	7.80	1.000	2.50	98.71	3.54	108.30
2,2-Dichloropropane	0.689	5.55	0.999	6.60	98.00	5.44	108.68
Bromochloromethane	0.273	6.19	1.000	2.81	100.57	2.91	104.87
Chloroform	0.779	6.94	1.000	2.27	102.49	3.49	108.26
Dibromofluoromethane SUR	0.409	8.16	1.000	2.44	100.17	3.38	108.55
1,1,1-Trichloroethane	0.683	7.01	1.000	4.26	98.69	4.21	107.11
Carbon Tetrachloride	0.572	7.88	0.999	5.49	94.34	4.55	106.46
1,1-Dichloropropene	0.601	6.66	0.999	4.62	98.40	4.59	106.80
Benzene	1.717	6.48	1.000	2.96	100.31	3.48	107.84
1,2-Dichloroethane	0.657	7.24	1.000	3.65	102.83	3.41	109.36
Trichloroethene	0.444	8.04	1.000	2.61	98.06	3.62	107.34
1-chloro-2,3-epoxypropane	0.143	6.10	1.000	5.98	89.00	3.06	97.02
1,2-Dichloropropane	0.459	7.47	1.000	2.59	100.14	3.54	109.03
Dibromomethane	0.302	8.12	1.000	2.56	98.46	3.00	106.53
Bromodichloromethane	0.625	8.49	1.000	2.36	97.74	3.40	109.88
cis-1,3-Dichloropropene	0.778	8.65	1.000	2.38	97.34	3.65	109.78
Toluene-d8 SUR	1.494	7.67	1.000	2.75	99.20	3.63	108.95
Toluene	1.095	7.56	1.000	2.50	99.23	3.63	108.98
trans-1,3-Dichloropropene	0.732	9.10	1.000	2.31	97.69	3.69	109.99
1,1,2-Trichloroethane	0.418	8.50	1.000	2.52	100.94	3.19	108.20
Tetrachloroethene	0.373	9.98	0.999	3.60	95.77	3.28	105.99
1,3-Dichloropropane	0.758	7.70	1.000	2.47	102.00	3.29	108.67
Dibromochloromethane	0.481	10.72	1.000	2.66	93.31	3.30	109.50
1,2-Dibromoethane	0.466	8.89	1.000	1.86	99.57	3.02	108.86
Chlorobenzene	1.451	7.26	1.000	2.43	100.91	3.21	107.36
1,1,1,2-Tetrachloroethane	0.542	7.74	1.000	2.54	97.69	3.06	107.12

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Ethylbenzene	2.545	6.56	0.999	3.15	100.34	3.44	107.37
Xylene (m+p)	3.919	6.53	0.999	3.00	101.00	3.35	107.10
Xylene (o)	2.044	7.07	1.000	2.48	100.34	3.12	107.75
Styrene	1.651	7.85	1.000	2.72	99.06	3.26	108.43
Bromoform	0.458	11.04	1.000	3.81	89.63	2.63	107.35
Isopropylbenzene	2.471	7.04	0.999	2.96	98.63	3.65	107.25
BFB SUR	0.726	7.27	1.000	2.66	100.91	3.25	107.43
Bromobenzene	1.026	6.87	1.000	2.13	102.34	3.29	107.78
1,2,3-trichloropropane	1.202	7.16	1.000	3.87	99.14	2.34	106.11
1,1,2,2-Tetrachloroethane	0.894	6.95	1.000	3.46	101.54	2.53	106.02
n-Propylbenzene	2.918	6.42	0.999	3.05	99.43	3.84	107.30
2-Chlorotoluene	0.569	6.94	1.000	2.72	100.57	3.16	107.27
4-Chlorotoluene	0.593	7.90	1.000	2.65	99.31	3.24	107.87
1,3,5-Trimethylbenzene	2.037	6.52	0.999	2.82	99.74	3.34	106.62
tert-Butylbenzene	2.018	6.85	0.999	3.43	98.20	4.01	107.02
sec-Butylbenzene	0.517	8.13	0.999	3.17	97.20	3.93	107.49
1,2,4-Trimethylbenzene	2.074	7.24	0.999	2.42	99.54	3.18	107.00
1,3-Dichlorobenzene	1.138	7.22	1.000	2.50	100.17	3.01	106.68
Isopropyltoluene	2.189	7.47	0.999	3.23	97.97	3.91	107.14
1,4,-Dichlorobenzene	2.202	7.55	1.000	2.85	99.57	3.24	106.05
1,2,-Dichlorobenzene	2.123	7.45	1.000	3.31	98.97	3.04	105.53
n-Butylbenzene	3.912	7.70	0.999	3.32	96.14	4.31	107.41
1,2-Dibromo-3-chloropropane	0.552	8.49	1.000	5.69	93.69	2.67	103.26
1,2,4-Trichlorobenzene	1.463	9.11	1.000	3.22	95.57	3.14	106.07
Naphthalene	5.006	7.20	1.000	3.15	97.97	2.80	104.39
Hexachlorobutadiene	0.604	8.45	0.999	2.99	96.14	4.58	104.33
1,2,3-Trichlorobenzene	1.389	8.64	1.000	3.46	95.89	2.94	104.75
Average *(page 6 & 7)*	**1.166**	**7.55**	**1.000**	**3.16**	**98.58**	**3.46**	**106.94**

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