APPLICATION

Purgeable Volatile Organic Compounds by GC-MS using Zebron™ ZB-624*PLUS*™ GC Columns

Agustin Pierri[1], Ph.D., Allen Misa[2], and Ramkumar Dhandapani[2]
[1]Weck Laboratories, Industry, CA 91745 USA
[2]Phenomenex, Inc., 411 Madrid Ave., Torrance, CA 90501 USA

Ramkumar Dhandapani Ph.D.
Global Product Manager - GC

He has a PhD in Analytical Chemistry and a total of 14 years experience in chromatographic method development and troubleshooting. Ramkumar loves to write poems, read Shakespeare, and attend Shakespeare plays.

Introduction

Volatile organic compounds (VOCs), both synthetic and naturally-occurring are a global environmental concern and can be found in a variety of samples including waste water, drinking water, and ground water. The traditional approach to VOC analysis is GC-MS (Gas Chromatography coupled to a Mass Spectrometer) using purge and trap extraction or an alternative extraction technique based on regulatory adherence. These compounds can potentially cause harm to human health as well as the surrounding environment. As a result, regulatory agencies have set standards for monitoring VOCs in all samples regardless of water source.

Due to the varying compositions and range of volatilities of VOCs, a highly selective GC column is required for analysis to provide optimal separation of critical VOCs in a short run time. Additionally, the use of a Mass Spec certified GC column is necessary to reach top performance criteria. An added challenge is method capability to handle various sample matrices as dirtier samples such as wastewater and sludge can greatly decrease column lifetime.

This technical note adheres to EPA Method 8260 for Volatile Organic Compounds by GC-MS, a fast and robust GC-MS method using a Zebron ZB-624*PLUS* GC column. The highly selective ZB-624*PLUS* stationary phase allows for identification and quantification of 74 VOCs, while maintaining a rapid analysis time of 15 minutes. The results are excellent performance and peak shapes of critical VOCs after 113 injections of various matrices including groundwater, wastewater, and sludge.

Experimental Conditions

Purge and Trap Method Parameters

Sample Temp:	40 °C
Purge Time:	11 min
Dry Purge Time:	2 min
Cryo Focus Temp:	20 °C
Inject Time:	2 min
Cryo Inject temp:	180 °C
Desorb Preheat:	245 °C
Desorb Time:	4 min
Desorb Temp:	250 °C
Bake Time:	7 min
Bake Temp:	260 °C
BGB off Delay:	0.5 min
MCS Bake Temp:	200 °C
Line Temp:	110 °C
Valve Temp:	110 °C
Purge Temp:	37 °C
Sample Volume:	25 mL
Purge and Trap Model:	Tekmar® 3100
Trap:	Supelco® VOCARB 3000 Trap K (p/n 24920-U)

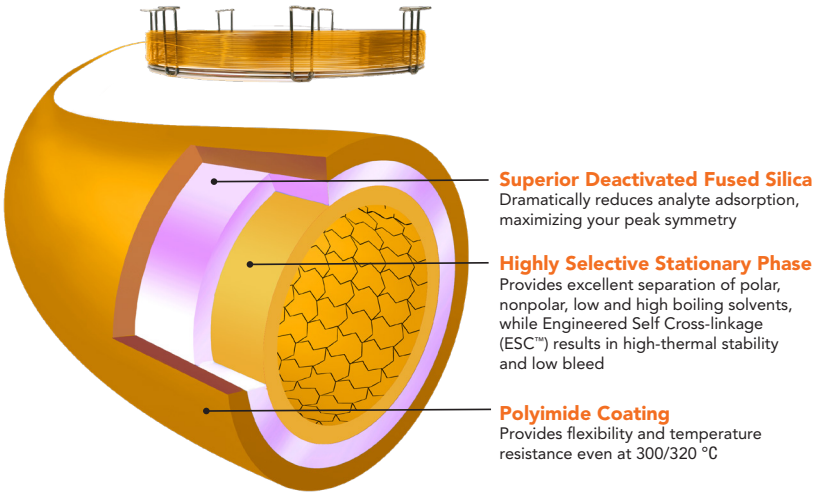
GC-MS Method Parameters

Column:	Zebron ZB-624*PLUS*
Dimensions:	30 meter x 0.25 mm x 1.40 µm
Part No.:	7HG-G040-27
Recommended Z-Guard™:	7CG-G000-00-GHK
Injection:	Split 50:1 @ 230 °C, 1 µL
Recommended Liner:	Zebron PLUS Straight Z-Liner™
Liner Part No.:	AG2-0A03-05 (for Agilent® & Thermo Scientific® systems)
Carrier Gas:	Helium @ 0.7 mL/min (constant flow)
Oven Program:	40 °C for 2 min, to 210 °C @ 17 °C/min for 3 min
Detector:	MSD @ 250 °C
Sample:	74 Volatile Organic Compounds spiked in 25 mL water See **Table 1**. for complete analyte list and retention times)

MS Conditions

Transfer Line Temperature:	250 °C
MS Source:	230 °C
Quad Temp:	150 °C
Emission Energy:	70eV
Scan Range:	50–550 m/z
Solvent Delay:	1.0 min
Software:	MassHunter® Software (Agilent Technologies)

Figure 1.
ZB-624*PLUS* Advantages



APPLICATION

Figure 2.
5 ppb Organic Volatiles by GC-MS on a Zebron™ ZB-624*PLUS*™ GC Column

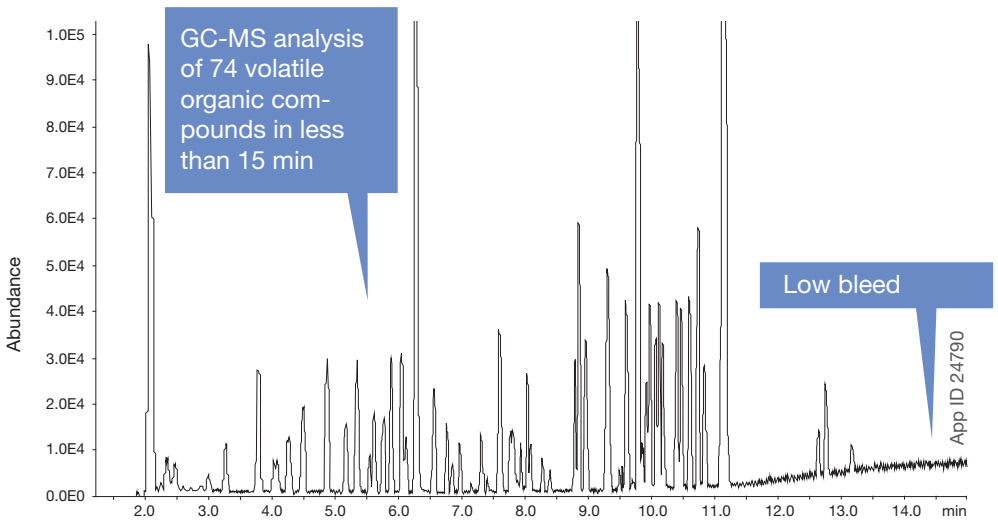
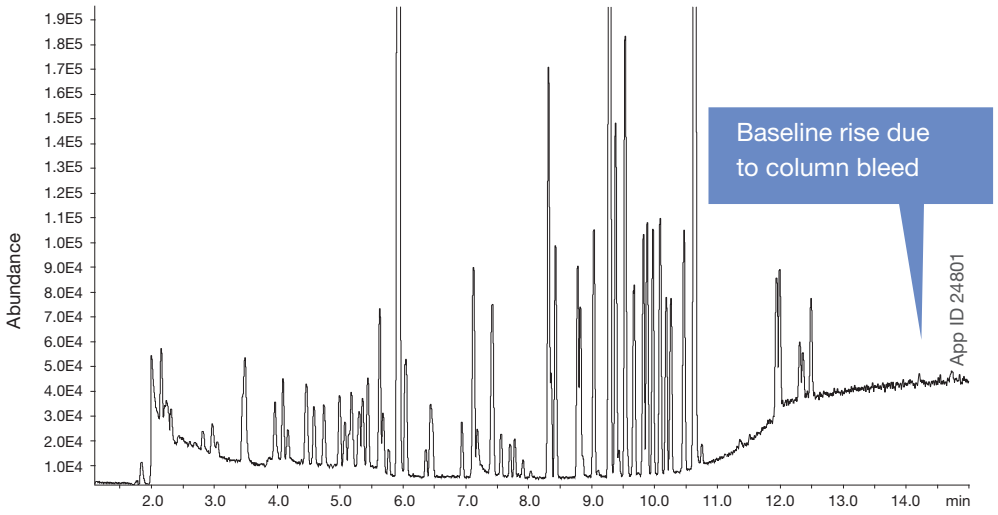


Figure 3.
5 ppb Organic Volatiles by GC-MS on an Agilent® DB®-624 GC Column



GC-MS Conditions

Conditions for both Figure 2 and Figure 3:

Column: Zebron ZB-624*PLUS*
Agilent DB-624
Dimensions: 30 meter x 0.25 mm x 1.40 µm
Part No.: 7HG-G040-27 (Zebron ZB-624*PLUS*)
122-1334 (Agilent DB-624)
Injection: Split 50:1 @ 230 °C, 1µL
Recommended Liner: Zebron PLUS Straight Z-Liner™
Liner Part No.: AG2-0A03-05 (for Agilent® & Thermo Scientific® systems)
Carrier Gas: Helium @ 0.7 mL/min (constant flow)
Oven Program: 40 °C for 2 min, 210 °C @ 17 °C/min for 3 min
Detector: Mass Spec transfer line @ 250 °C

Comparative separations may not be representative of all applications.

Table 1.
Analyte List

	Analytes	RT (min)
1.	Dichlorodifluoromethane	2.097
2.	Fluorobenzene (Istd)	6.281
3.	Chloromethane	2.329
4.	Vinyl chloride	2.456
5.	Bromomethane	2.865
6.	Chloroethane	2.990
7.	Trichlorofluoromethane	3.266
8.	1,1-Dichloroethene	3.793
9.	Carbon disulfide	4.039
10.	Trichlorotrifluoroethane	3.786
11.	Methylene chloride	4.261
12.	Acetone	3.823
13.	trans-1,2-Dichloroethene	4.503
14.	Methyl-t-butyl ether	4.484
15.	Tert-butyl alcohol(TBA)	4.319
16.	Diisopropyl ether	4.875
17.	1,1,-Dichloroethane	4.880
18.	Ethyl-t-butyl ether	5.177
19.	cis-1,2-Dichloroethene	5.351
20.	2,2-Dichloropropane	5.359
21.	Bromochloromethane	5.553
22.	Chloroform	5.615
23.	Carbon tetrachloride	5.901
24.	1,1,1-Trichloroethane	5.769
25.	2-butanone (MEK)	5.322
26.	1,1-Dichloropropene	5.890
27.	Benzene	6.056
28.	t-Amyl methyl ether	6.126
29.	1,2-Dichloroethane	6.091
30.	Trichloroethene	6.570
31.	Dibromomethane	6.862
32.	1,2-Dichloropropane	6.782
33.	Bromodichloromethane	6.982
34.	cis-1,3-Dichloropropene	7.323
35.	2-chloroethyl vinyl ethane	7.164
36.	MiBK	7.410
37.	trans-1,3-Dichloropropene	7.773
38.	1,1,2-Trichloroethane	7.948
39.	Toluene	7.607
40.	1,3-Dichloropropane	8.089
41.	Dibromochloromethane	8.284
42.	1,2-Dibromoethane	8.402
43.	Tetrachloroethene (PCE)	8.041
44.	2-hexanone	8.089
45.	1,1,1,2-Tetrachloroethane	8.864
46.	Chlorobenzene	8.801
47.	Ethylbenzene	8.859
48.	m,p-Xylene	8.966
49.	Bromoform	9.512
50.	Styrene	9.320
51.	o-Xylene	9.305
52.	1,1,2,2-Tetrachloroethane	9.876
53.	1,2,3 Trichlopropane	9.943
54.	Isopropylbenzene	9.611
55.	4-Bromofluorobenzene S1	9.789
56.	Bromobenzene	9.934
57.	n-Propylbenzene	9.978
58.	2-Chlorotoluene	10.084
59.	4-Chlorotoluene	10.185
60.	1,3,5-Trimethylbenzene	10.127
61.	tert-Butylbenzene	10.416
62.	1,2,4-Trimethylbenzene	10.468
63.	sec-Butylbenzene	10.613
64.	1,3-Dichlorobenzene	10.755
65.	4-Isopropyltoluene	10.740
66.	1,4-Dichlorobenzene	10.845
67.	1,2-Dichlorobenzene	11.171
68.	1,2-Dichlorobenzene-d4 S2	11.153
69.	n-Butylbenzene	11.110
70.	1,2-Dibromo-3-chloropropane	11.877
71.	1,2,4-Trichlorobenzene	12.650
72.	Naphthalene	12.939
73.	Hexachlorobutadiene	12.768
74.	1,2,3-Trichlorobenzene	13.176

Figure 4.
0.5 ppb Organic Volatiles by GC-MS on a Zebron ZB-624*PLUS* After 113 Sample Injections

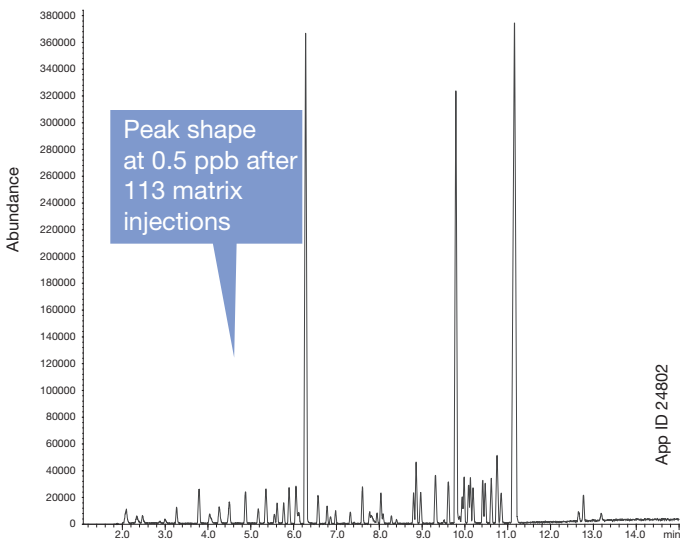
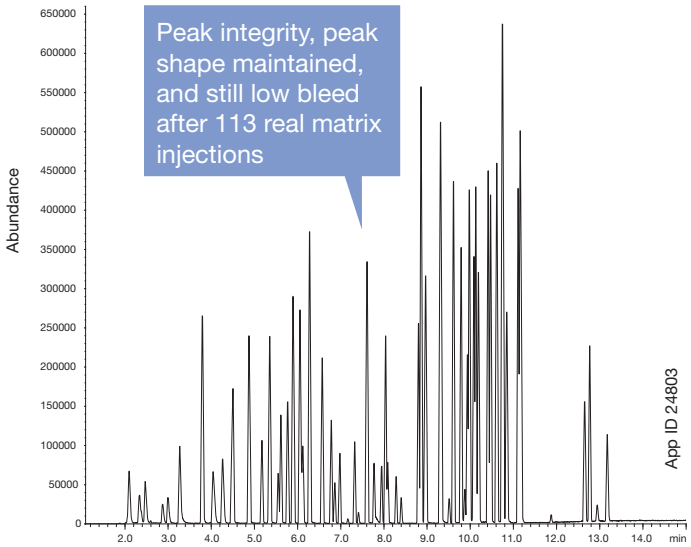


Figure 5.
5 ppb Organic Volatiles by GC-MS on a Zebron ZB-624*PLUS* After 113 Sample Injections



Results and Discussion

Organic volatile compounds in water is a very critical analysis. It requires a high efficiency GC column dimension for optimal separation of 74 analytes (**Table 1**.). Since this method is run on a GC-MS, the transfer line which serves as the interface between GC and MS is kept at higher temperature, in order to prevent condensation of analytes. The traditional 624 phase has 260 °C as column maximum temperature and will start to bleed if transfer line is maintained at 250 °C. Zebron ZB-624*PLUS* GC column is a low bleed MS certified column that has a maximum temperature limit of 300 °C/ 320 °C GC. This reduces baseline disturbance at high temperature and helps with accurate detections of higher boiling analyte. For this study, a Zebron ZB-624*PLUS* GC column 30 m x 0.25 mm x 1.40 µm was considered for analysis. As shown in **Figure 1**, ZB-624*PLUS* provides optimal separation of 74 VOCs with high efficiency and resolution. Compared to traditional 624 phase GC columns with a maximum temperature of 260 °C, the ZB-624*PLUS,* due to its extensive Engineered Self Cross Linkage (ESC™), Mass Spec compatibility and maximum temperature of 300/320 °C, gives the flexibility to elute out high boiling solvents without causing column bleed (**Figure 1**.).

Illustrated in **Figure 3**, an Agilent DB®-624 GC column was run under the same method parameters using the same 74 VOCs. Due to the temperature limit of the traditional 624 column at 260 °C, the chromatogram shows the baseline rising indicating stationary phase bleed. This not only affects the quantitation of higher boiling analytes, but also results in mass spec source contamination. **Figures 4** and **5** shows the separation of 74 VOCs at 0.5 ppb and 5 ppb levels after 113 injections on ZB-624*PLUS*. These injections included samples from wastewater, sludge, and groundwater. The retention, peak symmetry, and efficiency of all the analytes were maintained even after 113 injections of real sample matrix at both at 5 ppb and 0.5 ppb levels. Additionally, with the Zebron ZB-624*PLUS* upper temperature limit of 300/320 °C, it gave the flexibility to bake out analytes and regenerate the GC column when needed. The low bleed column provided steady baseline even at higher temperatures and helped in repeatable quantitation of the 74 compounds. The highly selective ZB-624*PLUS* stationary phase allowed for identification and quantification of 74 VOCs, while maintaining a rapid analysis time of 15 minutes, and resulted in excellent performance and peak shapes of 74 VOCs after 113 injections of various matrices including groundwater, wastewater, and sludge.

Conclusion

Volatile organic compound analysis via GC-MS and purge and trap is routine analysis for environmental testing and requires a high efficiency, low bleed, Mass Spec certified GC Column for optimal separation of critical VOCs. The Zebron ZB-624*PLUS* GC column is a low bleed and high efficiency column which has produced a shorter run time of 15 minutes for 74 VOCs with an added flexibility to bake out contaminants at 300/320 °C. The Zebron ZB-624*PLUS* is suitable for VOC analysis by EPA methods 524, 624, and 8260 and provides reproducible quantitation of volatile analytes.

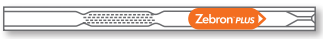
Acknowledgement

We would like to provide special thanks to Weck Laboratories for contributing this application.

WECK LABORATORIES, INC.

APPLICATION

Ordering Information
Zebron™ PLUS GC Inlet Liners

Description	Application	Inlet Style	Dimensions ID x L (mm)	Deactivation	Part No.	Unit
For 5890, 6890 and 7890 Models						
Straight Z-Liner™ 	Dirty samples, Volatiles, High initial oven temperatures	S/SL	4 x 78.5	PLUS Inert	AG2-0A03-01 AG2-0A03-05 AG2-0A03-25	ea 5/pk 25/pk

Australia
t: +61 (0)2-9428-6444
f: +61 (0)2-9428-6445
auinfo@phenomenex.com

Austria
t: +43 (0)1-319-1301
f: +43 (0)1-319-1300
anfrage@phenomenex.com

Belgium
t: +32 (0)2 503 4015 (French)
t: +32 (0)2 511 8666 (Dutch)
f: +31 (0)30-2383749
beinfo@phenomenex.com

Canada
t: +1 (800) 543-3681
f: +1 (310) 328-7768
info@phenomenex.com

China
t: +86 400-606-8099
f: +86 (0)22 2532-1033
cninfo@phenomenex.com

Denmark
t: +45 4824 8048
f: +45 4810 6265
nordicinfo@phenomenex.com

Finland
t: +358 (0)9 4789 0063
f: +45 4810 6265
nordicinfo@phenomenex.com

France
t: +33 (0)1 30 09 21 10
f: +33 (0)1 30 09 21 11
franceinfo@phenomenex.com

Germany
t: +49 (0)6021-58830-0
f: +49 (0)6021-58830-11
anfrage@phenomenex.com

India
t: +91 (0)40-3012 2400
f: +91 (0)40-3012 2411
indiainfo@phenomenex.com

Ireland
t: +353 (0)1 247 5405
f: +44 1625-501796
eireinfo@phenomenex.com

Italy
t: +39 051 6327511
f: +39 051 6327555
italiainfo@phenomenex.com

Luxembourg
t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

Mexico
t: 01-800-844-5226
f: 001-310-328-7768
tecnicomx@phenomenex.com

The Netherlands
t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

New Zealand
t: +64 (0)9-4780951
f: +64 (0)9-4780952
nzinfo@phenomenex.com

Norway
t: +47 810 02 005
f: +45 4810 6265
nordicinfo@phenomenex.com

Portugal
t: +351 221 450 488
f: +34 91-413-2290
ptinfo@phenomenex.com

Spain
t: +34 91-413-8613
f: +34 91-413-2290
espinfo@phenomenex.com

Sweden
t: +46 (0)8 611 6950
f: +45 4810 6265
nordicinfo@phenomenex.com

Switzerland
t: +41 61 692 20 20
f: +41 61 692 20 22
swissinfo@phenomenex.com

United Kingdom
t: +44 (0)1625-501367
f: +44 (0)1625-501796
ukinfo@phenomenex.com

USA
t: +1 (310) 212-0555
f: +1 (310) 328-7768
info@phenomenex.com

All other countries
Corporate Office USA
t: +1 (310) 212-0555
f: +1 (310) 328-7768
info@phenomenex.com

Ordering Information

Zebron ZB-624PLUS GC Columns

ID(mm)	df(µm)	Temp. Limits °C	Part No.
20-Meter			
0.18	1.00	-20 to 300/320	7FD-G040-22
30-Meter			
0.25	1.40	-20 to 300/320	7HG-G040-27
0.32	1.80	-20 to 300/320	7HM-G040-31
0.53	3.00	-20 to 300/320	7HK-G040-36
60-Meter			
0.25	1.40	-20 to 300/320	7KG-G040-27
0.32	1.80	-20 to 300/320	7KM-G040-31
0.53	3.00	-20 to 300/320	7KK-G040-36

Note: If you need a 5 in. cage, simply add a (-B) after the part number, e.g., 7HG-G040-27-B. Some exceptions may apply. Agilent 6850 and some SRI and process GC systems use only 5 in. cages.

guarantee

If Phenomenex products in this technical note do not provide at least an equivalent separation as compared to other products of the same phase and dimensions, return the product with comparative data within 45 days for a FULL REFUND.

www.phenomenex.com
Phenomenex products are available worldwide. For the distributor in your country, contact Phenomenex USA, International Department at international@phenomenex.com

TN60320418_W