Utilizing 624 GC Column Selectivity and Low Bleed Stationary Phase for the Analysis of Purgeable Volatile Organic Compounds by GC-MS

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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-624PLUS™ GC column. The highly selective ZB-624PLUS 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.

Materials and Methods

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-624PLUS
Dimensions:	30 meter x 0.25 mm x 1.40 µm
Part No.:	7HG-G040-27
Injection:	Split 50:1 @ 230 °C, 1 µL
Recommended Z-Guard™:	7CG-G000-00-GHK
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

Results

Figure 1.
Zebron ZB-624*PLUS* Advantages

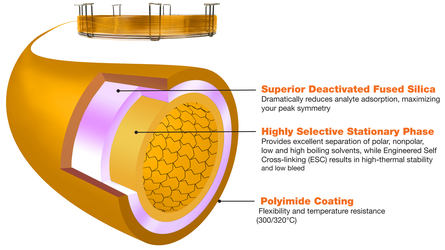
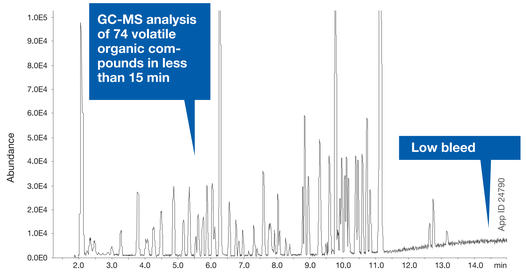


Table 1.
What makes the *PLUS* in ZB-624*PLUS*

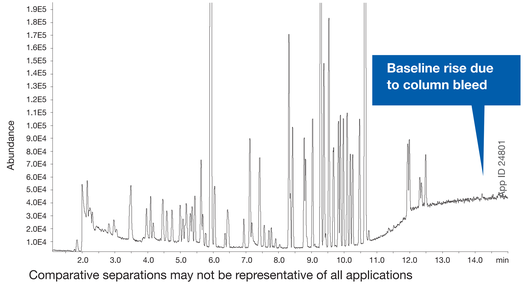
• **Low Bleed** Engineered Self Cross-linking™ (ESC™) provides high thermal stability with maximum column temperatures up to 300/320 °C.	• **Enhance Inertness** Proprietary superior deactivation gives great peak shape for troublesome compounds.
• **High Selectivity** A G43 phase that's highly selective for polar, non polar, low and high boiling solvents.	• **Column-to-Column Reproducibility** Excellent column-to-column reproducibility well suited for validated methods
• **Temperature Limits:** Push the temperature limits of traditional 624 and elute/bake high boiling analytes at 300/320 °C	• **MS Certified** Low bleed characteristics make it the ideal choice for GC-MS

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



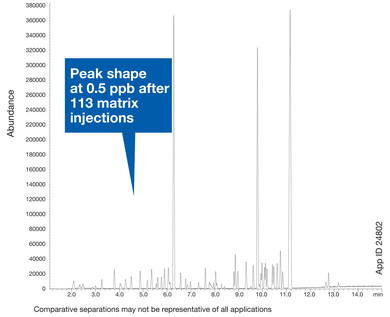
Comparative separations may not be representative of all applications

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



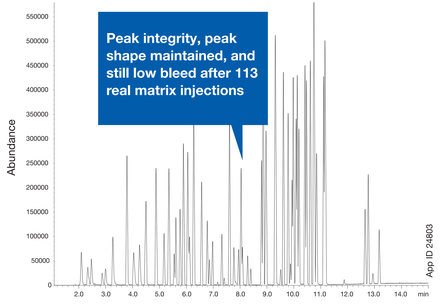
Comparative separations may not be representative of all applications

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



Comparative separations may not be representative of all applications

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



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. 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 analytes. For this study, a Zebron ZB-624PLUS GC column 30 m x 0.25 mm x 1.40 µm was considered for analysis. As shown in Figure 2, 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-linking (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 show 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, 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

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