APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

Author

Lee Marotta

PerkinElmer, Inc.
Shelton, CT 06484 USA

The Determination of Low Level Benzene, Toluene, Ethyl Benzene, and Xylenes (BTEX) in Drinking Water by Headspace Trap GC/MS

Introduction

BTEX is a grouping of structurally similar volatile organic compounds including benzene, toluene, ethyl benzene and the three xylene isomers. These compounds are known pollutants and are typically found near petroleum production and storage sites. BTEX are regulated toxic compounds while benzene is also an EPA target carcinogen. The investigation of these compounds, especially in drinking water at low levels, is critical to protect public health. This application note focuses on exceeding the current EPA detection limit requirement for BTEX while meeting and/or exceeding all other criteria in EPA method 524.2 for these analytes.

Instrumentation

A PerkinElmer® TurboMatrix™ Headspace (HS) sample handling system was used to volatilize and concentrate BTEX in water samples. To enhance detection limits, an inline trap was employed, which focused these analytes prior to injection onto the analytical column. A PerkinElmer Clarus® SQ 8S Gas Chromatograph Mass Spectrometer (GC/MS) configured with the standard capacity turbo molecular pump was the analytical system used.

PerkinElmer®
For the Better

The GC provided rapid cool down to shorten the period between injections (more samples analyzed in a "clock"). Using the temperature programmable low volume inlet improved peak efficiency by reducing "dead" volume and resulted in enhanced resolution, faster chromatography and improved detection limits. The PerkinElmer SQ 8S MS operating in full scan mode was used for this analysis, providing up to 20 times improved detection limits for this application.

Experiment and Results

The experimental conditions are presented in Tables 1 – 3. An Elite 624 column (20 m x 0.18 mm x 1.0 µm) was used in this application, which is also the column of choice for several laboratories analyzing volatile organic compounds by HS Trap. The narrow bore, shorter, efficient column aided in enhancing peak efficiency for shorter analysis time and signal-to-noise performance. Analyte equilibrium was empirically determined to be eight minutes.

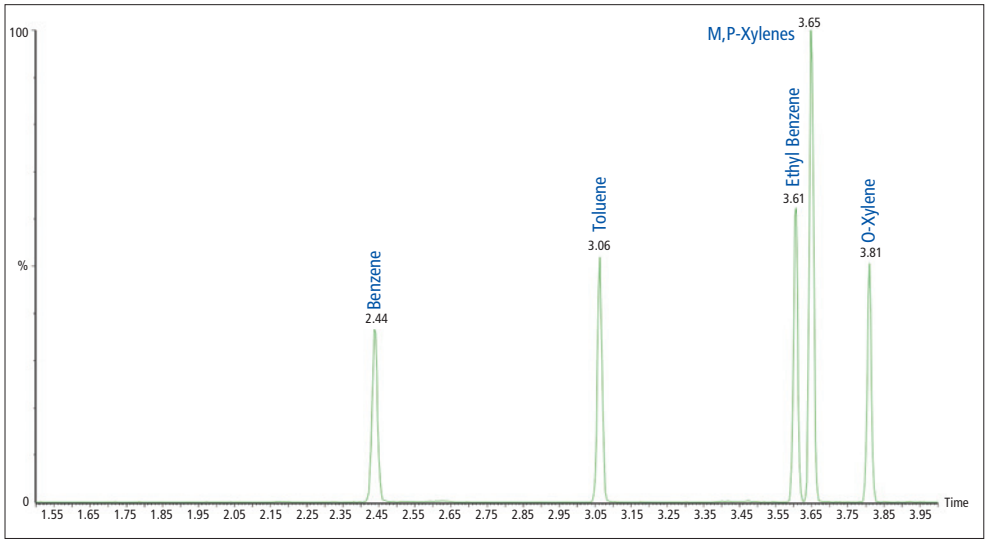

Figure 1. An example mass at 4.0 ppb acquiring in full scan.

Figure 1 presents a sample chromatogram recorded at 4.0 parts per billion (ppb) acquiring in full scan. Analytical results are displayed in Table 4 and include the 12-point calibration curve results, signal-to-noise recorded for the 0.02 ppb standard and precision measurements performed using 1.0 ppb standards.

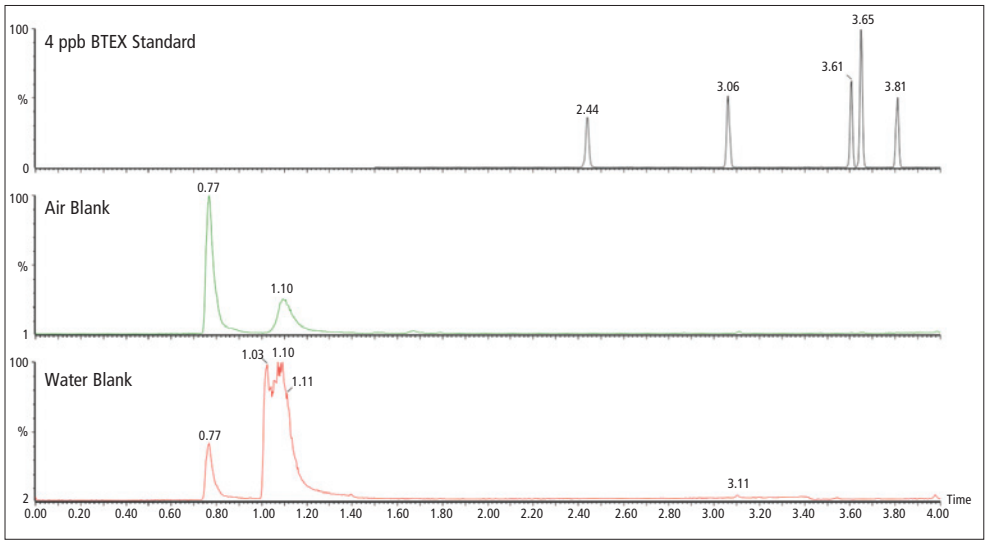

Figure 2. Water blank is the bottom chromatogram; air blank is the middle chromatogram; 4.0 ppb standard demonstrating separation from water is the top chromatogram.

Figure 2 illustrates the excellent water management ability of the HS Trap system. In these experiments, a two minute dry purge completely isolated the water from the target analytes. A three minute dry purge reduced water levels to baseline intensity, however a quicker analytical method was the goal of this application so a longer dry purge was avoided.

***Table 1.* Headspace Trap Conditions.**

Headspace System	TurboMatrix HS Trap
Sample Temperature	80 ˚C
Needle Temperature	110 ˚C
Transfer Line Temperature	120 ˚C
Trap Low/Trap High	35 ˚C to 260 ˚C
Equilibration Time	8 min
Dry Purge	2.0 min
Trap Hold	2.5 min
Trap Material	Carbopack B & X
Outlet Split	n/a
All HS Pressures	23.3 psi

***Table 2.* Gas Chromatograph Conditions.**

GC/MS	Clarus SQ 8S
Column	Elite 624-20 m x 0.18 mm x 1.0 µm
Oven	40 ˚C for 0.5 min, then 35 ˚C/min to 185 ˚C
Injector (PSS)	Temp Programmable Split/Splitless at 180 ˚C
Inlet Configuration	HS Mode turned ON
Carrier Program (He)	1 mL/min for 0.4 min, then 0.7 mL/min
Split Flow from GC	n/a

***Table 3.* Mass Spectrometer Conditions.**

Ionization Mode	Electron Impact
Acquisition	Full Scan
Mass Range	35 to 350 amu
Filament Delay	1.5 min
Scan Speed	0.15 sec
Interscan Delay	0.04 sec
Run Time	4 min
Ion Source Temperature	200 ˚C
Transfer Line Temperature	200 ˚C

***Table 4.* Analytical Results.**

	s/n at 0.02 ppb	Linearity, r^2 0.02 to 60 ppb	Precision at 1 ppb (n=7)
Benzene	370 to 1	0.9996	2.85%
Toluene	550 to 1	0.9994	2.76%
Ethyl Benzene	578 to 1	0.9993	2.53%
m,p-Xylenes*	670 to 1	0.9997	1.07%
o-Xylene	240 to 1	0.9994	3.86%

**The amounts are double for meta and para xylenes since they co-elute.*

Conclusion

In this application note the analysis of BTEX in water samples by HS Trap GC/MS using the Clarus SQ 8S Mass Spectrometer was performed. An analytical technique with a short cycle time and excellent performance is described. The analysis of BTEX using a mass spectrometer such as the Clarus SQ 8S not only allows the benefit of added sensitivity (therefore lower detection limits) but also additional analyte confirmation, which provides molecular level identification thus limiting false positives. Water management in the system was exceptional, and water from the matrix could be fully eliminated with an additional minute of dry purge time. With the sample preparation and mechanical advantages HS trap delivers versus purge and trap systems, greater uptime using this approach can be expected as well. This solution provides contract laboratories with compelling reasons and benefits for investing in this system, such as longer intervals between system maintenance, operator ease of use, fast cycle times (72 samples analyzed within a 12 hour clock), instrument and method robustness and optimal analytical performance.

PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

PerkinElmer®

010004_01