

## MTBE and BTEX On-Site Rapid Screening of Contaminated Ground Water by SPME-GC-TMS

**MTBE and BTEX compounds were rapidly screened in petroleum contaminated ground water using solid phase microextraction (SPME) followed by on-site analysis with a TRIDION™-9 portable gas chromatograph-toroidal ion trap mass spectrometer (GC-TMS). The SPME-GC-TMS method can be used to effectively extract, concentrate, separate and detect both MTBE and BTEX quickly and reliably on-site for rapid decision making and cost savings.**

### Introduction

Methyl-tert-butyl ether (MTBE) and the combination of benzene, toluene, ethylbenzene, and xylenes (BTEX) are routinely monitored in soil and ground water to determine whether petroleum contamination has occurred due to industrial runoff or waste water effluent in order to protect local tributaries and surrounding property<sup>1-2</sup>. For industrial environmental monitoring programs, the ability to quickly analyze samples taken from nearby ground and water sources is key to environmental protection. The ability to analyze samples at the source without the need for fixed laboratory analysis can result in a significant time and cost savings, and in the case of a spill can lead to improved response times for clean up. A sampling and analysis methodology for MTBE and BTEX that includes on-site sampling and analysis with results produced in under 3 minutes without the need for sample storage at 4°C and transport back to a fixed base laboratory represents a significant time and cost savings.

The CUSTODION™ SPME syringe, together with the TRIDION™-9 gas chromatograph-toroidal ion trap mass spectrometer (GC-TMS) can be used for rapid on-site sample extraction and analysis for sample screening and decision making. Following on-site sample analysis, informed decisions can be made regarding which samples should be sent to a laboratory for confirmatory analysis. In addition, on-site sample collection and extraction using SPME minimizes target analyte losses compared to traditional techniques that risk analyte loss during storage and transport.

### Sampling Equipment

The CUSTODION SPME sampling syringe consists of a 1cm fiber coated with a liquid polymer film, solid sorbent or a combination of the two. The SPME fiber is housed in a durable syringe that can be operated with a single hand. Much like a retractable ball point pen, the push button trigger on top of the CUSTODION syringe allows the SPME fiber to be extended and retracted into and out of a protective syringe needle. The SPME fiber traps target analytes from air, headspace (solids or liquids), or direct sampling of liquids and dissolved solid samples.

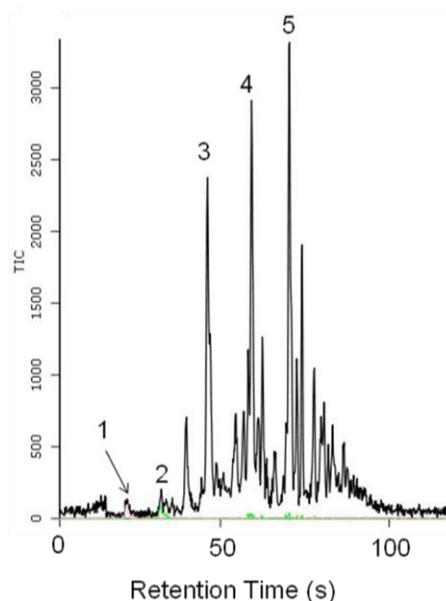


Figure 1. Chromatogram of BTEX screening in petroleum contaminated ground water, 15 s immersion using a PDMS/DVB/CAR SPME fiber. Compound identification: (1) MTBE, (2) benzene, (3) toluene, (4) ethylbenzene, (5) xylenes.

The CUSTODION PDMS/DVB/CAR SPME syringe used for this application consists of a 1cm length of silica fiber coated with polydimethylsiloxane (PDMS), divinylbenzene (DVB) and carboxen (CAR). Both MTBE and BTEX compounds were extracted directly from petroleum-contaminated water samples.

### Sample Preparation and Analysis

A water sample suspected of containing MTBE and BTEX in the presence of gasoline was provided by a major petroleum manufacturer. The sample was diluted 1:100 using DI water. The CUSTODION SPME fiber was directly immersed in the water sample for ~15 s at ambient temperature (~24°C).

After sampling, the SPME fiber was introduced into the injection port on the TRIDION-9 GC-TMS where the MTBE and BTEX were desorbed from the fiber into a low thermal mass capillary GC column. The GC temperature was then rapidly programmed from 50°C to 200°C at 2°C/s resulting in a total run time of 2.5 minutes. The low thermal mass GC was directly interfaced to a toroidal ion trap mass spectrometer (TMS) having a mass range from 50-500 Da. Overall, this method is rapid with a total SPME sampling time of 15 sec and GC-TMS analysis time of ~2.5 min.

### Experimental Conditions

Sampling:	Solid phase microextraction (SPME)
SPME Phase:	Polydimethylsiloxane/ divinylbenzene/carboxen (PDMS/DVB/CAR)
GC Inj. Temp:	270°C
GC Column:	MXT-5, 5 m x 0.1 mm, 0.4 µm d <sub>f</sub>
GC Carrier Gas:	Helium
GC Column Temp:	50-200°C at 2°C/s
Transfer Line:	270°C
Injection Split:	30:1 after 4 sec splitless
Mass Analyzer:	Toroidal ion trap (TMS)
TMS Mass Range:	50-500 Da
Ionization Mode:	In-trap electron impact (EI)
Detector:	Electron multiplier
Vacuum:	Roughing and turbo molecular pumps
MS Resolution:	Less than unit mass to 230 amu, nominal unit mass to 500 amu

### Results and Discussion

Figure 1 shows the GC-TMS separation of MTBE and BTEX in a ground water sample. All compounds were detected and positively identified by the TRIDION-9 compound library. For compounds which co-elute, an on-board deconvolution algorithm was able to positively identify each component based on statistical treatment of the ion fragmentation data.

SPME-GC-TMS is a useful method for on-site sample screening prior to transport for laboratory MTBE and BTEX analysis by GC-TMS. Water or solid samples can be screened using the TRIDION-9 GC-TMS for preliminary information on potential levels of contamination, if any. The CUSTODION SPME syringe and TRIDION-9 GC-TMS provide a fast, easy, accurate and reliable method for sample extraction and on-site analysis.

### References

1. Zhang, Z., Pawliszyn, J. *Analysis for organic compounds in environmental samples by headspace solid phase microextraction*. J. High Res. Chromatogr. (1993), 16(12), 689-92.
2. Almeida, C.M., Boas, L.V.J. *Analysis of BTEX and other substituted benzenes in water using headspace SPME-GC-FID: method validation*. Environ. Monit. (2004) Jan;6(1):80-8.

### Acknowledgements

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