APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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MTBE and BTEX On-Site Rapid Screening of Contaminated Ground Water by Portable SPME-GC/MS Methodology

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[1-2]. 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 significant time and cost savings, and in the case of a spill, can lead to improved response times.

Sampling using the Custodion® SPME syringe, with separation and analysis using the Torion® T-9 portable GC/MS, allows rapid and on-site sample extraction and analysis, and provides actionable results within minutes. Using the portable Torion T-9 GC/MS, informed decisions can be made in critical situations, and can optimize follow-up activities by providing the user with the appropriate information to determine which, and if, samples should be sent to a laboratory for confirmatory analysis. In addition, on-site sample collection, extraction and analysis, using SPME and the Torion T-9 GC/MS, minimizes target analyte losses, compared to traditional techniques that risk analyte loss during complex storage and transport conditions, therefore ensuring the integrity of critical and valuable samples.

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Experimental

The Custodion SPME sampling device used in this application consists of a 1 cm length of silica fiber coated with polydimethylsiloxane (PDMS), divinylbenzene (DVB) and carboxen (CAR). The fiber is housed in a durable syringe that can be operated with a single hand, much like a retractable ballpoint 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.

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 deionized (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 Torion T-9 GC/MS, 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 41-500 Da.

Method Parameters

Table 1. Method Parameters.

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 df
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	41-500 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/MS separation of MTBE and BTEX in a ground water sample. All compounds were detected and positively identified by the Torion T-9 compound library. For co-eluting compounds, an onboard deconvolution algorithm was able to positively identify each component based on statistical treatment of the ion fragmentation data.

The use of the Torion T-9 Portable SPME-GC/MS method successfully collected, extracted, analyzed, and identified the compounds on interest, demonstrating that portable GC/MS is a powerful technique for infield analysis of complex samples and mixtures.

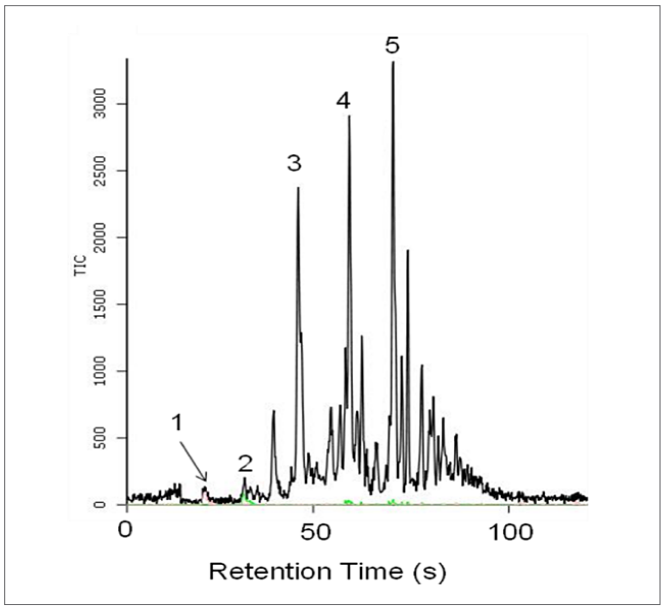

Figure 1. Chromatogram of MTBE and BTEX screening in petroleum contaminated ground water, 15 s immersion using a PDMS/DVB/CAR SPME fiber. Compound identification: (1) MTBE, (2) benzene, (3) toluene, (4) ethylbenzene, (5) xylenes.

Conclusion

MTBE and BTEX compounds were rapidly screened in petroleum contaminated ground water using solid phase microextraction (SPME) followed by on-site analysis with a Torion T-9 portable GC/MS. The SPME-GC/MS method was used to effectively extract, concentrate, separate and detect both MTBE and BTEX quickly and reliably on-site for rapid decision making and cost savings.

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.

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