



## APPLICATION NOTE

### Gas Chromatography/ Mass Spectrometry

Authors:

Portable GC/MS Team

Shelton, CT

## Volatile Organic Compound Identification in Water by Portable SPME-GC/MS

### Introduction

Groundwater water sources are routinely tested for volatile organic compounds (VOCs) of environmental concern. The

current standard of practice is to collect water samples in specified containers and transport them under refrigeration to a fixed laboratory for analysis. These laboratories can be far from the sample site, and thus such practice may pose important challenges because volatile target analytes may be lost during sample storage and transport. Additionally, volatile compounds such as benzene, toluene and ethylbenzene are susceptible to biological degradation under environmental conditions where refrigeration alone may not be adequate to preserve these compounds in water samples for more than seven days<sup>1</sup>. Safeguarding sample integrity can be achieved by performing the sample collection, extraction and analysis on-site. The solid phase microextraction (SPME)-Torion® T-9 portable GC/MS technology provides the capability to perform on-site, near real-time chemical analysis for accurate and positive detection of analytes subject to degradation in complex samples.

## Experimental

For this application, a 65  $\mu\text{m}$  SPME fiber with Divinylbenzene/Polydimethylsiloxane (DVB/PDMS) phase was used to extract VOCs from a water sample spiked with 25 environmental contaminants. The SPME 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<sup>®</sup> syringe allows the SPME fiber to be extended and retracted into and out of a protective metal sheath.

A culinary water sample was spiked with a mixture of 25 target VOCs in methanol (Restek, Bellefonte, PA), with each compound at a concentration of  $\sim 0.04 \mu\text{g}/\mu\text{L}$  in the final sample solution (Figure 1). The Custodion SPME fiber was immersed in the water sample for 5 sec., which was adequate for VOC adsorption onto the SPME fiber. Immediately following sample collection, the Custodion SPME fiber was introduced into the heated injection port (270 °C) of the person-portable Torion T-9 portable GC/MS system, where the VOCs desorbed quickly from the SPME fiber into the GC inlet. The low thermal mass GC was temperature programmed at 2 °C/s from 40 ° to 270 °C to provide rapid separation of the VOC analytes. The system mass range is 41 to 500 Da, which is suitable for detection of most compounds of environmental concern. A VOC target compound library, based on retention time and key mass ions, was preloaded into the Torion T-9 portable GC/MS target compound library and used together with a proprietary deconvolution algorithm to positively identify all 25 volatiles in the sample. The sample analysis and compound identification required less than four minutes.

## Method Parameters

Table 1. Method Parameters.

Sampling	Solid phase microextraction (SPME)
SPME Phase	Divinylbenzene/Polydimethylsiloxane (DVB/PDMS, 65 $\mu\text{m}$ )
GC Inj. Temp	270 °C
GC Column	MXT-5, 5 m x 0.1 mm, 0.4 $\mu\text{m}$ df
GC Carrier Gas	Helium, 0.2 ml/min., constant pressure
GC Column Temp	40-270 °C, 2 °C/s
Transfer Line	270 °C
Injection Split Ratio	20:1
Mass Analyzer	Toroidal ion trap (TMS)
TMS Mass Range	41-500 Da
Ionization Mode	In-trap electron impact
Detector	Electron multiplier
Vacuum	Roughing and turbo molecular pumps
Resolution	Less than unit mass to 230 amu, nominal unit mass to 500 amu

## Results

Figure 1 shows the GC/MS separation of these 25 VOCs spiked into the culinary water matrix. Using a rapid GC temperature gradient, the separation was achieved in  $\sim 70$  s, with a total cycle time, including automated library identification of the VOCs, of less than four minutes. Using an embedded deconvolution algorithm, the co-eluting compounds Trichloroethylene and 1,2-Dichloropropane (Figure 1, compounds 10 and 11) were positively identified.

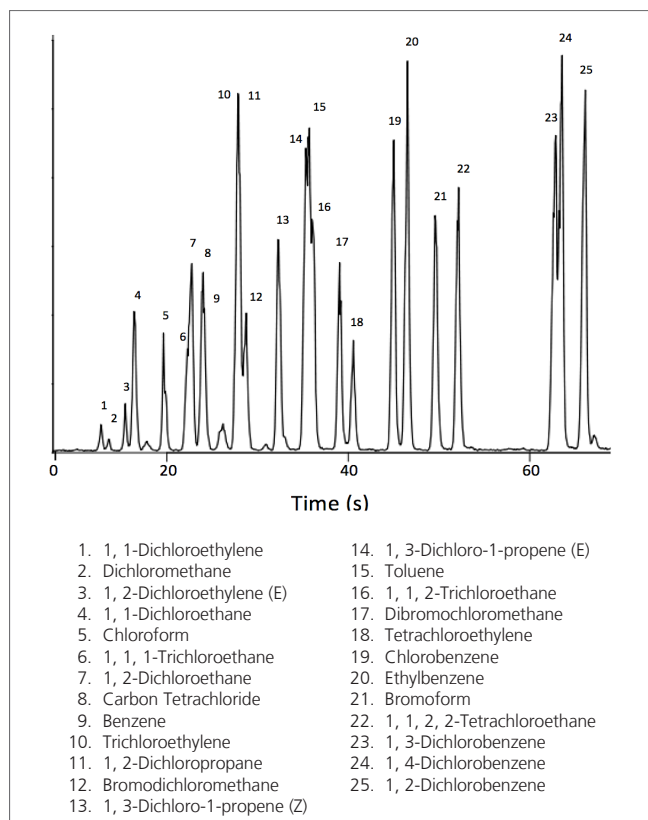


Figure 1. The chromatogram with positive identification of the 25 VOCs.

## Conclusion

Volatile organic compounds (VOCs) were rapidly sampled and concentrated from water in five seconds using a Custodion<sup>™</sup> SPME syringe. Compounds were analyzed using the Torion<sup>®</sup> T-9 portable GC/MS. The SPME-GC/MS technique can be used to effectively extract, concentrate, separate and identify complex mixtures of VOCs quickly and reliably in less than four minutes.

## References

- 40 CFR Chapter 1(7-1-07 edition), Appendix A to Part 136, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 624-Purgeables.