

## Volatile Organic Compounds Screening in Soil using SPME-GC-TMS

**Volatile organic compounds (VOCs) were extracted and concentrated from soil using water, a salting-out technique, and a CUSTODION™ solid phase microextraction (SPME) syringe. The TRIDION™-9 portable gas chromatograph-toroidal ion trap mass spectrometer (GC-TMS) was used to separate and identify 37 VOCs in less than 2 min. The SPME-GC-TMS technique is fast, reliable, and eliminates the need for special laboratory equipment or solvents for VOC screening from complex matrices in the field.**

### Introduction

The ability to screen soil samples in the field by identifying VOCs is a valuable tool where sampling decisions are required in near real-time. Results of the screening procedure may be used to guide sample collection activities and can help identify which sampling and/or extraction methods are most effective. U.S. EPA Screening Method 3815 provides limited VOC concentration information by only reporting an estimate of the total VOC concentration in a sample. This method uses a photoionization detector (PID) without any separation technique, thus individual VOCs and their relative concentrations are not identified and reported.<sup>1</sup> The CUSTODION SPME syringe in combination with the TRIDION-9 portable GC-TMS was used to positively identify 37 VOCs and their relative concentrations in the field in under 2 minutes.

### Sampling Equipment

The CUSTODION SPME syringe consists of a 1 cm SPME fiber coated with 50-100 µm of liquid polymer film, solid sorbent or a combination. The SPME polymer phase traps target analytes from air, headspace, liquids, or dissolved solid samples.<sup>2</sup> The SPME fiber is housed inside the CUSTODION syringe in a manner similar to a retractable ball point pen. A push button trigger on top allows the SPME fiber to be extended and retracted into and out of a protective sheath using a single hand. In this application, a CUSTODION SPME syringe with a 65 µm Polydimethylsiloxane/Divinylbenzene (PDMS/DVB) phase was used to extract volatile compounds from a soil sample.

Only water was required as the extraction solvent, which eliminates organic solvent interferences during GC analysis and reduces overall sampling and analysis costs.

1	Trichlorofluoromethane	20	Chlorobenzene
2	1,1-Dichloroethylene	21	1,1,1,2-Tetrachloroethane
3	Dichloromethane	22	Ethylbenzene
4	MTBE	23	Xylene
5	1,2-Dichloroethylene (Z)	24	Bromoform
6	Chloroform	25	Styrene
7	1,1,1-Trichloroethane	26	1,1,2,2-Tetrachloroethane
8	1,2-Dichloroethane	27	Isopropylbenzene
9	Carbon Tetrachloride	28	Bromobenzene
10	Benzene	29	1,2,3-Trichloropropane
11	2-Butanone	30	1,3,5-Trimethylbenzene
12	Trichloroethylene	31	1,2,4-Trimethylbenzene
13	Bromodichloromethane	32	1,3-Dichlorobenzene
14	1,3-Dichloro-1-propene (Z)	33	1,4-Dichlorobenzene
15	1,3-Dichloro-1-propene (E)	34	1,2-Dichlorobenzene
16	Toluene	35	1,2-Dibromo-3-chloropropane
17	1,1,2-Trichloroethane	36	1,2,4-Trichlorobenzene
18	4-Methyl-2-pentanone	37	Naphthalene
19	Tetrachloroethylene		

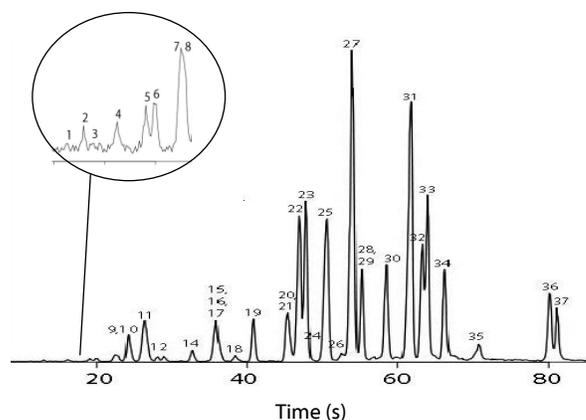


Figure 1. Chromatogram of 37 volatiles extracted from soil.

### Sample Preparation and Analysis

A Standard Reference soil sample prepared by the supplier with a VOC mixture of 42 compounds at concentrations ranging from ~15,000 µg/kg (15 ppm, w/w) to ~2400 µg/kg (2.4 ppm, w/w) was obtained and analyzed (NSI Solutions, Raleigh, NC). 5 mL of water with 25% NaCl (w/v) was added to 5 grams of the spiked soil sample. The sample vial was shaken vigorously by hand for 10 sec, after which the CUSTODION SPME fiber was exposed to the sample headspace for 50 seconds. The shaking/exposure technique was

repeated 5 times for a total sampling time of ~5 minutes. Following extraction the CUSTODION SPME syringe was inserted into the TRIDION-9 GC-TMS injection port (250°C) where the VOCs were desorbed and transferred to a low thermal mass capillary GC column (MXT-5, 5 m x 0.1 mm, 0.4 µm d<sub>i</sub>). Ultra-high purity helium was used as the GC carrier gas under constant flow conditions. The GC temperature program conditions were 40° to 280°C at 2°C/s for total run times under 2.5 min. The GC is directly interfaced to the TMS detector, which has a mass range of 45-500 Da and an average scan rate of ~10Hz. A user defined deconvolution target compound library positively identified the VOCs.

### Experimental Conditions

Sampling:	Solid phase microextraction (SPME)
SPME Phase:	Divinylbenzene/Polydimethylsiloxane (DVB/PDMS, 65 µm)
GC Inj. Temp:	250°C
GC Column:	MXT-5, 5 m x 0.1 mm, 0.4 µm d <sub>i</sub>
GC Carrier Gas:	Helium, 0.2ml/min, constant pressure
GC Column Temp:	40-280°C at 2°C/s
Transfer Line:	250°C
Injector Split Ratio:	20:1
Mass Analyzer:	Toroidal ion trap (TMS)
TMS Mass Range:	45-500 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 and Discussion

Figure 1 shows the GC-TMS separation of VOCs extracted from a spiked soil sample. 37 of the 42 VOCs were identified in less than two minutes. In some cases, analytes co-eluted (e.g., 1,3-Dichloro-1-propene (E), Toluene, and 1,1,2-Trichloroethane compounds 15-17) but were positively identified by the target compound library using the automated on-board deconvolution algorithm. The 5 analytes not detected included 2-Hexanone, Acetone, Bromomethane, Chloroethane, and Chloromethane. These compounds are all very volatile and may have either been lost from the sample during extraction, lost prior to analysis through handling, or these particular compounds may have suffered selective discrimination during the SPME sampling process.

The CUSTODION SPME and TRIDION-9 GC-TMS are uniquely suited for near real-time field analysis screening of VOCs and other organic compounds to support rapid decision making in the field. If additional sample analyses are required, results can be obtained in minutes. This SPME-GC-TMS sample screening method allows the user to determine the presence of individual VOCs and their relative concentrations. Following initial screening, highly concentrated samples can be diluted before causing cross-over contamination during analysis on laboratory-based instruments. This reduces sample re-analysis costs and instrument down time from carryover and contamination that may occur following analysis of highly concentrated samples on GC-MS systems.

### References

1. EPA SW-846 Method 3815 *Screening Solid Samples for Volatile Organics*, Revision 0, February 2007, <http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/3815.pdf>
2. Zhang, Zhouyao; Pawliszyn, Janusz. *Analysis for organic compounds in environmental samples by headspace solid phase microextraction*. Journal of High Resolution Chromatography (1993), 16(12), 689-92.

### Acknowledgements

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