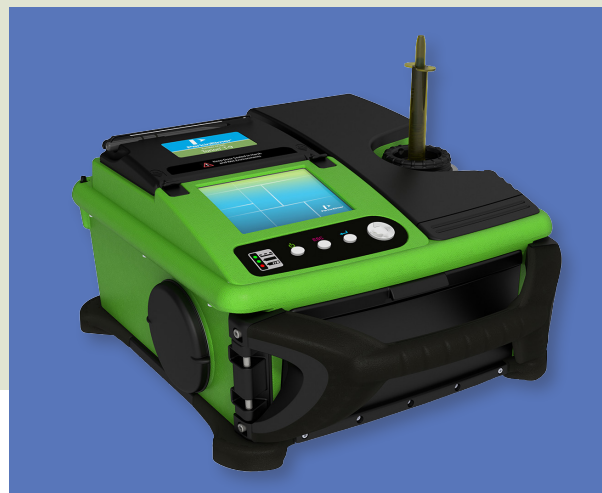


## Gas Chromatography/ Mass Spectrometry

# The Benefit of Field-Portable GC/MS as a Rapid Screening Tool for Semi Volatile Organic Compounds in Water



Torion T-9 Portable GC/MS

Over the years, many types of analytical instruments have been reduced to a portable or hand-held format to be used in the field, including XRF, LIBS, Raman, FT-IR and NIR analyzers.

However, shrinking a gas chromatograph/mass spectrometer (GC/MS) to a field-portable configuration, whilst maintaining laboratory analytical performance, is a much greater challenge. Most of the previous attempts have utilized “point-and-shoot” approaches, which have not required any type of sample preparation or sample introduction accessories. For that reason, the practical value of a field-portable instrument is reduced significantly if it necessitates complex sample preparation or delicate procedures are required to introduce the sample into the gas chromatograph.

In this technical note, we describe results from a novel, field-portable GC/MS system (Torion® T-9, PerkinElmer Inc., Shelton, CT) for use as a rapid screening tool for semi volatile organic compounds (SVOCs) and in particular phenolic compounds and phthalate esters in water samples with a typical analysis time of less than 10 minutes.

### Torion T-9 GC/MS Technology

The original system and its applicability for field-based analysis have been described previously in the open literature<sup>1,2</sup>. However, a number of recent improvements have been made by replacing the conventional capillary column with a low thermal mass (LTM) column bundle using direct-contact electrical resistive heating. This column provides identical heat distribution, but virtually eliminates cooler spots of traditional column technology, thus improving the chromatographic separation for SVOCs at the high temperature GC runs required for high boiling point compounds.

The mass spectrometer uses a toroidal ion trap configuration, which is well-suited for miniaturization compared to other designs. The novel configuration allows for large trapping volumes resulting in high ion counts, low noise levels and good spectral quality. The ion trap mass analyzer is heated to ~175-210 °C depending on the target analytes and operates under vacuum. This results in the electrodes staying clean for long periods of time, reducing the need for frequent maintenance. For a detailed description of the Torion T-9 GC/MS technology please refer to the following citation<sup>3</sup>.

### Sample Preparation Module

The capabilities of this GC-MS technology can be further enhanced by the use of a compact, battery-operated, rugged sampling accessory (SPS-3™, PerkinElmer Inc., Shelton, CT) for use in the field<sup>4</sup>. The choice of rapid sampling modules include solid-phase microextraction (SPME) and heated headspace (HS) for solids; needle traps (NT) for gaseous samples; together with purge and trap (P&T) and thermal desorption (TD) for liquids, as well as modules for the addition of internal standards (IS). Using this flexible sampling approach, the system can easily be configured for the many different and varied application requirements for sample preparation and analysis at a specific sampling location.

Let's now take a more detailed look at the methodology for using the instrument as a rapid screening tool for phenolic compounds and phthalate esters in water samples.

## Rapid Screening Tool for SVOCs

This portable GC/MS technology can also be used as a general screening tool for SVOCs in water using micro liquid extraction (MLE) and a coil wire filament (CWF). The experiments were carried out using tap water spiked with SVOCs at concentrations from low ppb-sub ppm levels. A small amount (0.2-0.5 mL) of suitable solvent, such as dichloromethane, hexane, pentane or acetone is used for extraction. Manual shaking and salting-out may be applied using NaCl at 0.5-3 % to speed up the extracting process. The extraction is performed for a few minutes, the solvent containing the analytes then applied on to the coil or if necessary, concentrated by letting the solvent evaporate after transferring to a small vial. Sample introduction using the coil is performed after solvent on the coil is evaporated. This particular screening test was carried out with a mixture of phenolic compounds, and phthalate esters in water. The mass spectrometer and chromatographic separation conditions are shown in Tables 1 and 2.

Figure 1 shows the total ion chromatogram of the separation of all the phenolic compounds in water, with phenol ( $C_6H_5OH$ ) identified with the bold red arrow. The extracted ion chromatogram of phenol is shown on the right with the reference mass spectrum from the NIST library below it. The full suite of phenols identified from left to right are (1) phenol (2) 4-methyl phenol, (3) 2-nitro phenol, (4) 3,5-dichloro phenol, (5) 4-chloro-3-methyl phenol, (6) 2,4,6-trichloro phenol, (7) 4-nitro phenol, (8) 2-methyl-4,6-dinitro phenol, and (9) pentachloro phenol.

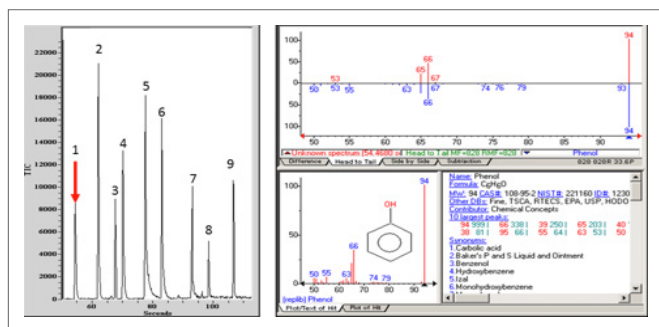


Figure 1. The total ion chromatogram of the separation of all the phenolic compounds in water with phenol ( $C_6H_5OH$ ) identified with the bold red arrow. The extracted ion chromatogram of Phenol is shown on the right, with the reference mass spectrum from the NIST library below it. The full suite of phenols going from left to right are (1) phenol (2) 4-methyl phenol, (3) 2-nitro phenol, (4) 3,5-dichloro phenol, (5) 4-chloro-3-methyl phenol, (6) 2,4,6-trichloro phenol, (7) 4-nitro phenol, (8) 2-methyl-4,6-dinitro phenol, and (9) pentachloro phenol.

The group of phthalate esters is shown in Figure 2, with dimethyl phthalate ( $C_{10}H_{10}O_4$ ) shown with the bold red arrow. The extracted ion chromatogram of dimethyl phthalate is shown on the right, with the reference mass spectrum from the NIST library below it. The full suite of phthalate esters identified from left to right are (1) dimethyl phthalate, (2) diethyl phthalate, (3) dibutyl phthalate, (4) benzyl butyl phthalate (5) di iso octyl phthalate and (6) di-n-octyl phthalate.

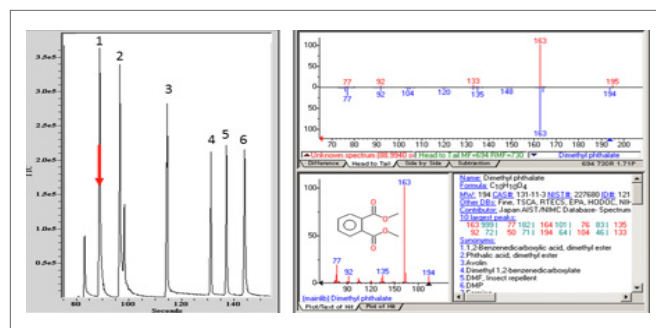


Figure 2. The total ion chromatogram of the separation of a group of phthalate esters with dimethyl phthalate ( $C_{10}H_{10}O_4$ ) seen with a bold red arrow. The extracted ion chromatogram of dimethyl phthalate is shown on the right with the reference mass spectrum from the NIST library below it. The full suite of phthalate esters going from left to right are (1) dimethyl phthalate, (2) diethyl phthalate, (3) dibutyl phthalate, (4) benzyl butyl phthalate (5) di iso octyl phthalate and (6) di-n-octyl phthalate.

The total running time for these screening tests for both phenols and phthalate esters, was less than five minutes. Ion molecule chemistry occurred to some degree on both types of samples, so absolute identification was confirmed using the NIST library search capability. Although the peak capacities are relatively low for these separations, the deconvolution algorithm helped to separate and identify the analytes with greater accuracy. Dynamic ranges and detection limits in real samples will be determined and presented in a future study.

Table 1. Mass spectrometer parameters for semi volatile organic compounds (SVOCs) in water.

Mass Spectrometer Operating Conditions	
Mass Spectrometer	Toroidal Ion Trap
Ionization Source	Electron Impact
MS Operating Temperature	200 °C
Mass Range	45-500 amu
Resolution	< 0.5 m/z at 300 amu
MS Scan Rate	10-15 scan/s
Detector	Electron Multiplier

Table 2. The chromatographic separation conditions for the screening of 9 phenolic compounds, 6 phthalate esters in water.

Gas Chromatographic Separation Conditions		
Sample Delivery	Coil Wire Filament Injection	Coil Wire Filament Injection
Injection Type	Split/splitless (10 s splitless)	Split/splitless (10 s splitless)
Injector Temperature	290 °C	300 °C
Transfer Line Temperature	270 °C	280 °C
Trap Temperature	200 °C	200 °C
Initial Temperature/ Hold Time	50 °C for 10 s	50 °C for 10 s
Temperature Ramp Rate	2 °C/s	2 °C/s
Final Temperature/ Hold Time	290 °C for 60 s	300 °C for 60 s

## Conclusion

There is a growing demand for the analysis of trace levels of volatile and semi-volatile organic compounds in air, water and solid matrix samples under harsh conditions in remote, field-based locations. This study has demonstrated that it is now possible to achieve laboratory-grade performance with a portable GC/MS combined with rapid sample preparation/introduction techniques. This combination enables a wide variety of environmental-based assays for both quantitative and qualitative screening purposes, which can provide fast, actionable data for non-technical and inexperienced operators in the field. It has been demonstrated in this technical note that the Torion T-9 GC/MS system has successfully identified nine phenolic compounds and six phthalate esters in water in less than five minutes.

## References

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