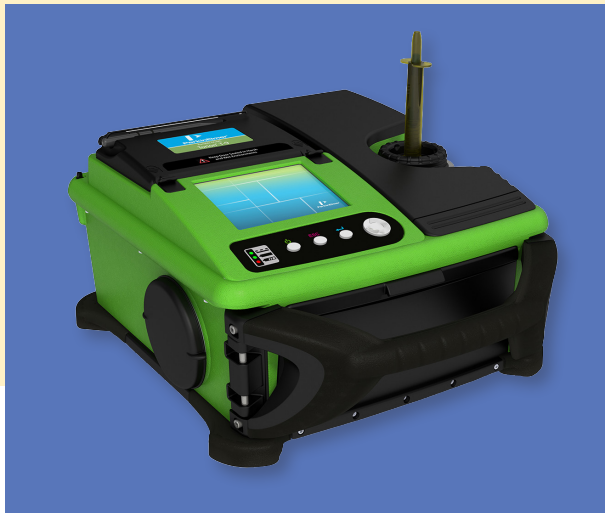
TECHNICAL NOTE

Gas Chromatography/
Mass Spectrometry

The Benefit of Field-Portable GC/MS for the Rapid Sampling and Measurement of Geosmin in Drinking 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 the rapid identification and measurement of geosmin in drinking 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[1, 2]. 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[3].

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[4]. 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 the determination of geosmin in drinking water.

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Analysis of Geosmin in Drinking Water

Geosmin is an organic compound produced by a variety of microorganisms and bacteria. It has a distinct earthy flavor and aroma, and is responsible for the earthy taste of beets and the strong scent that occurs in the air when rain falls after a dry spell of weather. Geosmin is produced by several classes of microbes, including cyanobacteria and actinobacteria, and is released when these microbes die. Communities whose water supplies depend on surface water can periodically experience episodes of unpleasant-tasting water when a sharp drop in the population of these bacteria releases geosmin into the local water supply[5]. Chemically, it is a bicyclic alcohol with a formula of $C_{12}H_{22}O$, and a derivative of decahydro naphthalene, commonly known as decalin. Its boiling point is ~270 °C[6].

20 ppt of Geosmin was spiked into 500 mL of a water sample. Without any pretreatment step, it was then trapped on Polydimethylsiloxane (PDMS) particles (125 -180 µm size) packed in a deactivated stainless steel solid phase extraction (SPE) desorption tube at ambient temperature using a flow rate of 25-35 mL/min delivered by a vacuum pump. The target analyte was then transferred into a PDMS needle trap using the instrument's thermal desorber system. The desorption step was carried out at 200 °C at 6 mL/min for 10 min, using He carrier gas. Sample introduction into the GC-TMS using the needle trap was conducted at 270 °C for 60 s. A schematic of the sample delivery approach is shown in Figure 1.

The mass spectrometer and chromatographic separation conditions are shown in Table 1 and 2 respectively. The total ion chromatogram (TIC) of the separation is shown in Figure 2, together with extracted ion chromatogram (RIC), showing the parent molecular ion and the associated fragments of geosmin, which is confirmed by the NIST reference mass spectrum underneath it. Figure 3 shows the deconvoluted chromatogram and mass spectrum, demonstrating that the 20 ppt geosmin is well-separated using the instrument's deconvolution algorithm. Based on the statistical analysis of the geosmin calibration, it was estimated that the detection limit was in the order of single digit ppt levels.

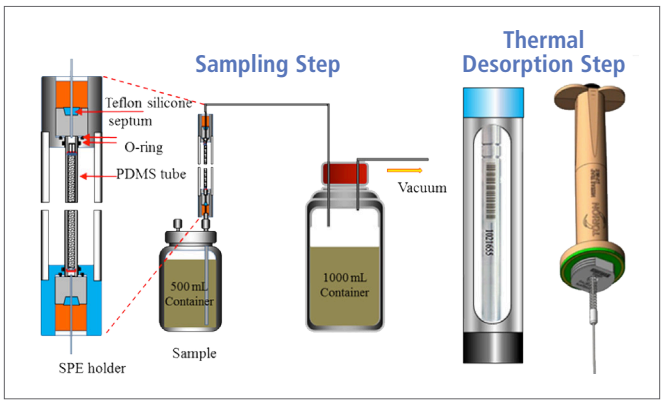

Figure 1. The sampling procedure and thermal desorption step for the analysis of geosmin by GC/MS.

Table 1. Mass spectrometer parameters for geosmin in water.

Mass Spectrometer Operating Conditions	
Mass Spectrometer	Toroidal Ion Trap
Ionization Source	Electron Impact
MS Operating Temperature	190 °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 geosmin in water.

Gas Chromatographic Separation Conditions	
Sample Delivery	Needle Trap Injection
Injection Type	Split/Splitless
Split Injection Times	
10:1 Split on	20 s
10:1 Split off	40 s
50:1 Split on	40 s
50:1 Split off	80 s
Injector Temperature	270 °C
Transfer Line Temperature	250 °C
Trap Temperature	190 °C
Column Technology (Restek®, State College, PA)	MXT® -5: low-polarity phase diphenyl dimethyl polysiloxane; 5 m x 0.1 mm x 0.4 µm
Initial Temperature/ Hold Time	50 °C for 10 s
Temperature Ramp Rate	1 °C/s
Final Temperature/Hold Time	300 °C for 10 s
Carrier Gas Inlet Pressure	26 psi

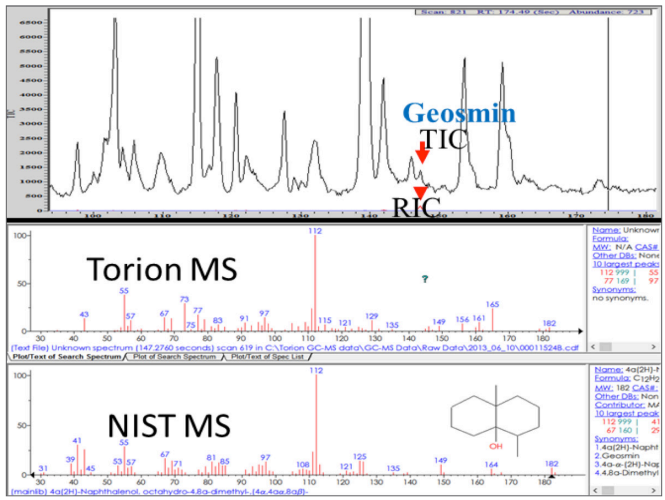

Figure 2. Total ion (TIC) and extracted ion chromatograms (RIC) of geosmin and its MS fragments in a water sample, identified and confirmed by the mass spectrum from the NIST reference library.

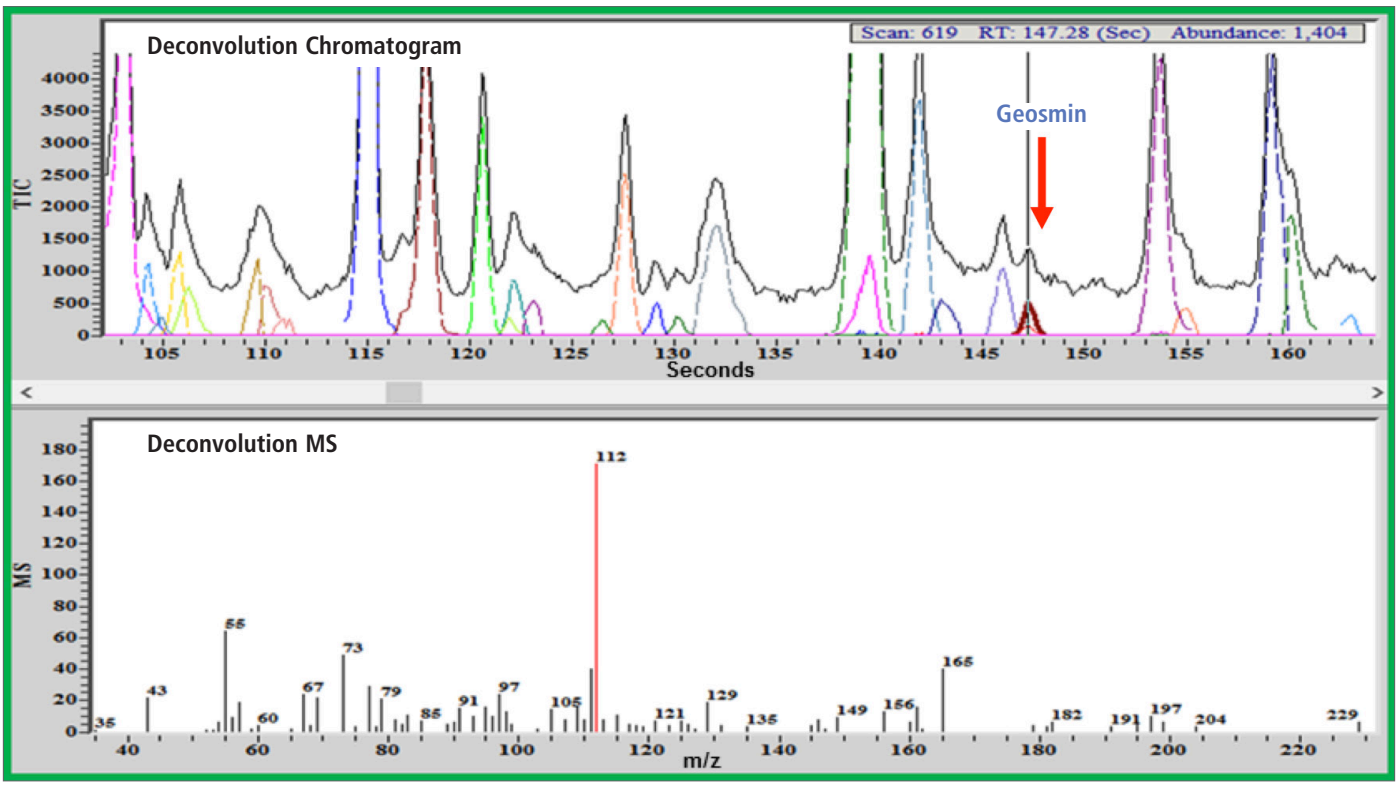

Figure 3. The deconvoluted chromatogram and mass spectrum demonstrating that the geosmin is well-separated using the instrument's deconvolution algorithm.

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 detected and confirmed the presence of single digit ppt levels of geosmin in drinking water in under 10 minutes.

References

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