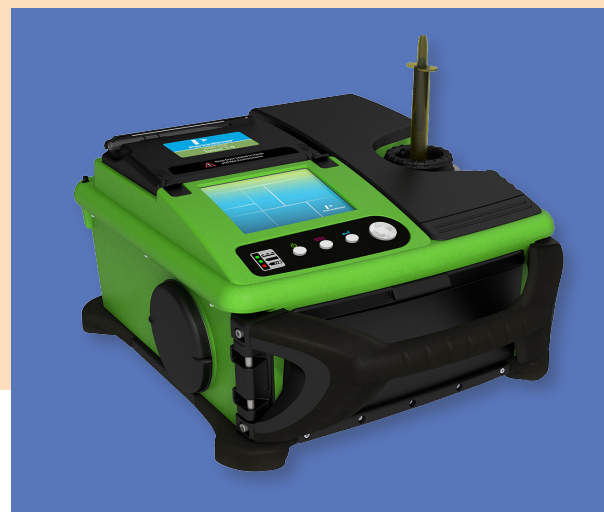## Gas Chromatography/ Mass Spectrometry

The Benefit of Field-Portable GC/MS for the Rapid Sampling and Measurement of Terpenes

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 identifying and quantifying a mixture of terpenes 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 analysis of a suite of terpenes with a wide range of boing points.

PerkinElmer®
For the Better

The Analysis of Terpenes

Terpenes are a large class of organic compounds, produced by a variety of plants, including conifers, hops, and cannabis with a typical boiling point range of 150-200 °C. They are the primary constituents of the essential oils of many types of plants and flowers widely used as fragrances in perfumery, as well as for medicinal purposes. Synthetic variations and derivatives of natural terpenes are also used for a variety of aromas and flavors used as food additives. Therefore, to exemplify the capability of this technology, four terpene compounds were spiked into 200 mL of 0.6% NaCl in water. The analytes were then extracted using half/half solid phase micro extraction (SPME) polydimethylsiloxane/divinylbenzene (PDMS/DVB) 65 μm fibers at room temperature (22 °C) for 15 minutes without shaking or vibrating. With this sampling approach, the fiber is placed half in the head space and half immersed into the liquid phase of the sample, as shown in Figure 1.

Instrumental Conditions

This sample was then injected into the GC/MS system using mass spectrometer and chromatographic separating conditions shown in Table 1 and 2 respectively.

The total ion chromatogram (TIC) of the four terpenes ((+)-alpha-pinene, Myrcene, (+)-alpha-limonene, and Isolongifolene), is shown in Figure 2.
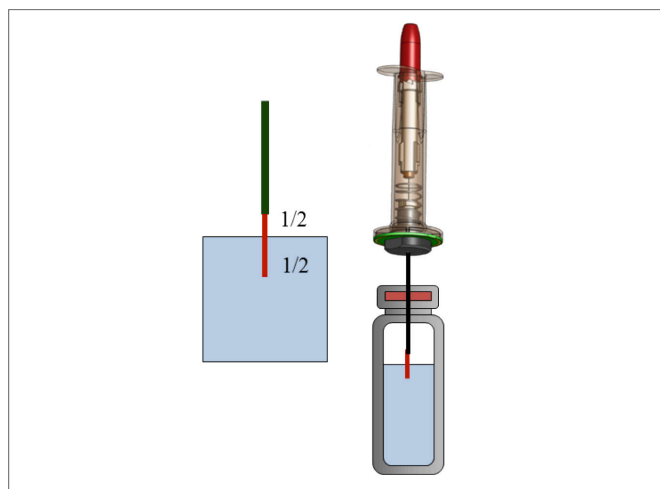

Figure 1. The four terpene analytes were extracted by half/half SPME (PDMS/DVB 65 μm fibers) at room temperature (22 °C) for 15 minutes, before being injected into the GC/MS.

Table 1. Mass Spectrometer Parameters.

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 a suite of terpenes.

| Gas Chromatographic Separation Conditions	
Sample Delivery	Coiled Wire Filament Injection
Injection Type	Split/Splitless
Injector Temperature	300 °C
Transfer Line Temperature	280 °C
Trap Temperature	200 °C
Column Technology (Restek®, State College, PA)	MXT® -5: low-polarity phase diphenyl dimethyl polysiloxane; 5 m x 0.1 mm x 4 μm
Initial Temperature/ Hold Time	50 °C for 10 s
Temperature Ramp Rate	2 °C/s
Final Temperature/Hold Time	280 °C for 50 s
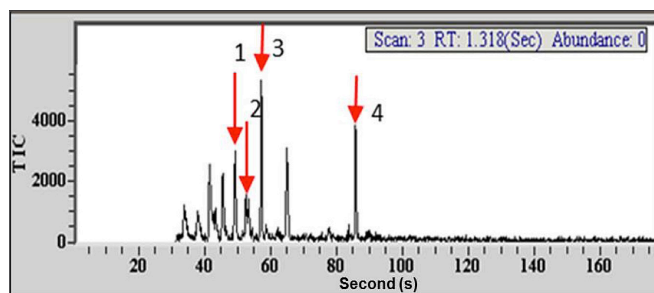
Figure 2. Total ion chromatogram of (1) (+)-alpha-pinene, (2) Myrcene, (3) (+)-alpha-limonene, and (4) Isolongifolene.

A four point calibration graph was then generated for the four terpene compounds. The concentrations of the standards and the respective calibration plots with correlation coefficients (R^2) are shown in Figure 3. It should be noted that the estimated detection limit for the four compounds was 20 ppt, which was based on the statistical analysis of multiple replicates of the lowest standard (Sample 1 in Fig. 3).
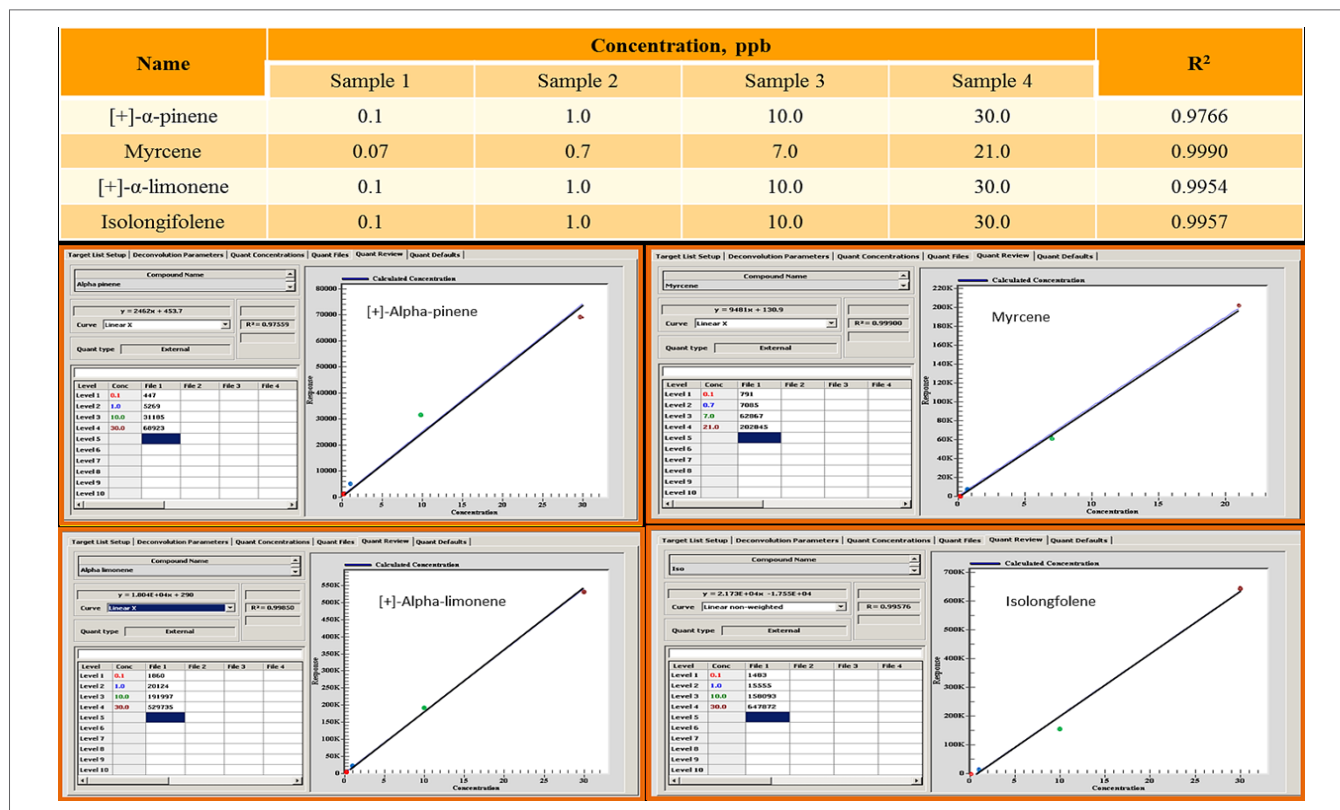
Name	Concentration, ppb				R^2
	Sample 1	Sample 2	Sample 3	Sample 4	
[+]-α-pinene	0.1	1.0	10.0	30.0	0.9766
Myrcene	0.07	0.7	7.0	21.0	0.9990
[+]-α-limonene	0.1	1.0	10.0	30.0	0.9954
Isolongifolene	0.1	1.0	10.0	30.0	0.9957

Figure 3. Calibration plots of the four terpene compounds.

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 Application Note that the Torion T-9 GC/MS system has detected and measured a suite of terpenes at low ppt concentrations in under 10 minutes total analysis time.

References

1. Hand-Portable Gas Chromatograph-Toroidal Ion Trap Mass Spectrometer (GC-TMS) for Detection of Hazardous Compounds; J. A. Contreras et.al., Journal of American Society of Mass Spectrometry, Vol 19, Issue 10, p 1425–14, (2008).

2. Trace Analysis in the Field Using Gas Chromatography-Mass Spectrometry; T. V. Truong et.al., Scientia Chromatographica, 6(1):13-26, (2014).

3. Torion T-9 Portable GC/MS Product Note, https://www.perkinelmer.com/lab-solutions/resources/docs/PRD_Torion-T-9-GCMS_012311B_01.pdf.

4. SPS-3 Sample Preparation Module for Torion T-9, http://www.perkinelmer.com/lab-solutions/resources/docs/PRD_Sample_Prep_Station(013095_01).pdf.

For a complete listing of our global offices, visit www.perkinelmer.com/ContactUs