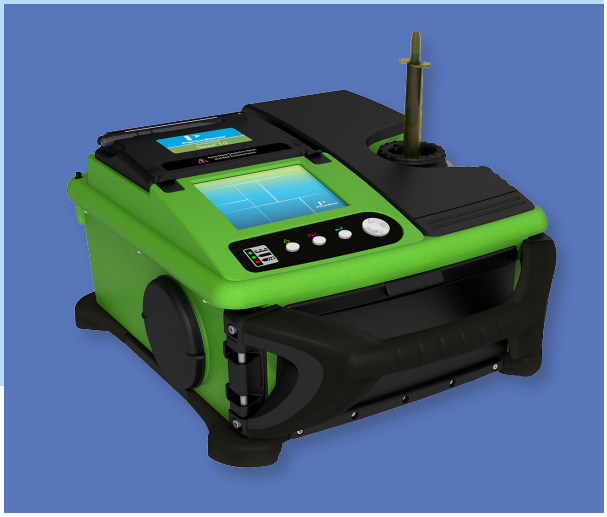
TECHNICAL NOTE

The Benefit of Field-Portable GC/MS for the Rapid Characterization of a Suite of Organochlorine Pesticides in Black Tea Samples

Gas Chromatography/ Mass Spectrometry



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 characterization of a suite of organochlorine pesticides in black tea 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 and characterization of a suite of organochlorine pesticides in black tea.

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Characterization of a Suite of Organochlorine Pesticides in Black Tea

Capillary gas chromatography is the technique most widely used for the analysis of organochlorine pesticides in food, water or soil samples. EPA RCRA Method 8081B is typically used to determine the concentrations of various organochlorine pesticides in extracts from environmental solid and liquid matrices, using fused-silica capillary columns with an electron capture detector (ECD) detector[5]. However, the majority of this class of pesticides cannot be determined in a single analysis, because the chemical and chromatographic behaviors of many of these compounds can result in coelution of some target analytes in complex sample matrices. For that reason GC/MS is often the preferred way of separating and quantifying a suite of organochlorine pesticides.

For this investigation 20 g dry black tea was steeped in 500 mL of hot water and left for four days at 22 °C. Fifteen mL sample was then spiked at three concentration levels (A, B, C) of seven different organochlorine insecticides shown in Table 1, with boiling points ranging between 275 °C and 425 °C.

The sample preparation was performed using SPME fibers conditioned at 220 °C for 60 min, while the immersion extraction process was carried out at 22 °C for 10 min by stirring with a bar mixer at 300 rpm. The fibers were then rinsed after the extraction with deionized water for 10 s without vibration. The sample was injected at 270 °c for 40 s (15 s splitless). After injection the fibers were washed with DI water for 30 s and conditioned at 270 °C for 30 s (in the GC injector). The mass spectrometer and GC chromatographic separation conditions are shown in Table 2 and 3.

Table 1. Concentration ranges of the organochlorine pesticides used in this study.

No.	Name	Sample A Concentration (ppb)	Sample B Concentration (ppb)	Sample C Concentration (ppb)
1	Benfluralin	4	40	200
2	Lindan	2	20	100
3	Heptachlor	2	20	100
4	Chlorthal dimethyl	4	40	200
5	Heptachlor epoxide	2	20	100
6	Endrin	2	20	100
7	Methoxychlor	2	20	100

Table 2. Mass spectrometer parameters for a suite of organochlorine pesticides in tea.

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 3. The GC chromatographic separation conditions for a suite of organochlorine pesticides in tea.

Gas Chromatographic Separation Conditions	
Injection Type	Split/Splitless
Injector Temperature	290 °C, 20 s splitless with pre-run split closed
Transfer Line Temperature	270 °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	2 °C/s
Final Temperature/Hold Time	300 °C for 150 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 identified and characterized a suite of high boiling point organochlorine pesticides in tea samples in less than 10 minutes.

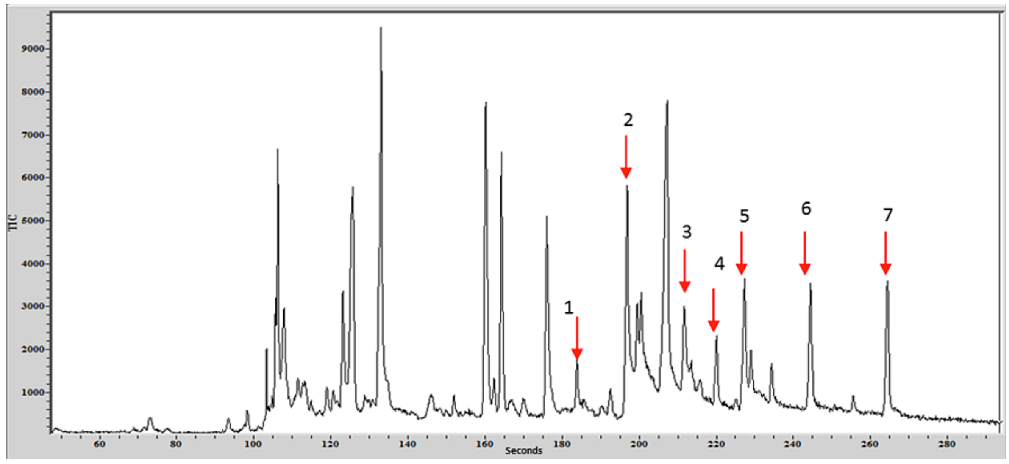

Figure 1. The TIC of the seven organochlorine pesticides in sample B (identity and concentration of pesticide shown in Table 1).

References

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5. EPA Test Method 8081B: The Analysis of Organochlorine Pesticide Compounds by GC Capillary Column Technology, United States Environmental Protection Agency, Revision 2, 2007: https://www.epa.gov/sites/production/files/2015-12/documents/8081b.pdf.

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