APPLICATION BRIEF

Gas Chromatography/
Mass Spectrometry

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Qualifying Garlic Volatiles by Headspace GC/MS

Introduction

Garlic, Allium sativum L., has a strong history of culinary use due to its pungent aroma and flavour produced by sulphur containing organic compounds. There is increasing interest in these compounds as potential treatments for chronic diseases, such as cardiovascular disease and cancer. The food industry also has particular interest in garlic aroma compounds as the quality of garlic powder affects overall food quality[1].

Many of the aroma compounds are volatile thus suited for Headspace Gas Chromatography/Mass Spectrometry (HS-GC/MS) analysis. HS sampling heats up the sample and transfers the volatiles to the GC/MS without the need for steam distillation, or solid phase microextraction techniques, which can often be time-consuming[2].

This application brief demonstrates the identification of aroma compounds in garlic powder samples by HS-GC/MS, as an indicator of garlic quality. The analysis requires no sample preparation, making it a fast and simple method.

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Method

The analysis of garlic powders was carried out on a PerkinElmer Clarus® SQ 8 GC/MS system with a PerkinElmer Turbomatrix™ HS-40. Three Garlic powder samples were obtained from The Bart Ingredients Company (Bristol, UK). Standard 20 mL headspace vials (Part No. N9303348) and aluminium crimped caps with PTFE lined silicon septa were used (Part No. N9302975). The experimental conditions for this analysis are given in Tables 1 to 3.

Table 1. Headspace experimental conditions.

Headspace Sampler	Turbomatrix HS-40
Headspace Pressure	30 psi
Thermostat Vial Temperature	80 °C
Thermostat Time	15 min
Needle Temperature	90 °C
Transfer Line Temperature	110 °C
Vial Pressurise Time	1.00 min
Injection Time	0.02 min
Withdraw Time	0.20 min
Sample Weight	1.0 g

Table 2. GC experimental conditions.

Gas Chromatograph	PerkinElmer Clarus 680 GC
Column	Elite-5 MS capillary column (60 m x 0.25 mm x 0.25 µm)
Injection Port Type	Programmable split/splitless
Injector Temperature	220 °C
Injection Type	Split, 10:1
Carrier Gas	Helium, constant pressure (25 psi)
Oven Programme	35 °C for 2 min, 15 °C/min to 220 °C, 45 °C/min to 300 °C, hold for 2 min
Run Time	18 min
Withdraw Time	0.20 min
Sample Weight	1.0 g

Table 3. Clarus SQ 8 GC/MS experimental conditions.

Mass Spectrometer	PerkinElmer Clarus SQ 8 GC/MS
Ionisation Mode	EI
GC Inlet Line Temperature	200 °C
Ion Source Temperature	180 °C
Function Type	Full Scan
Scan Range	m/z 35-350
Scan Time	0.20 s
InterScan Delay Time	0.05 s

Results

Figure 1 shows the Total Ion Chromatograms (TICs) of the three garlic powder samples. The intensity scales of the chromatograms were normalized to each other to make comparisons between the garlic samples.

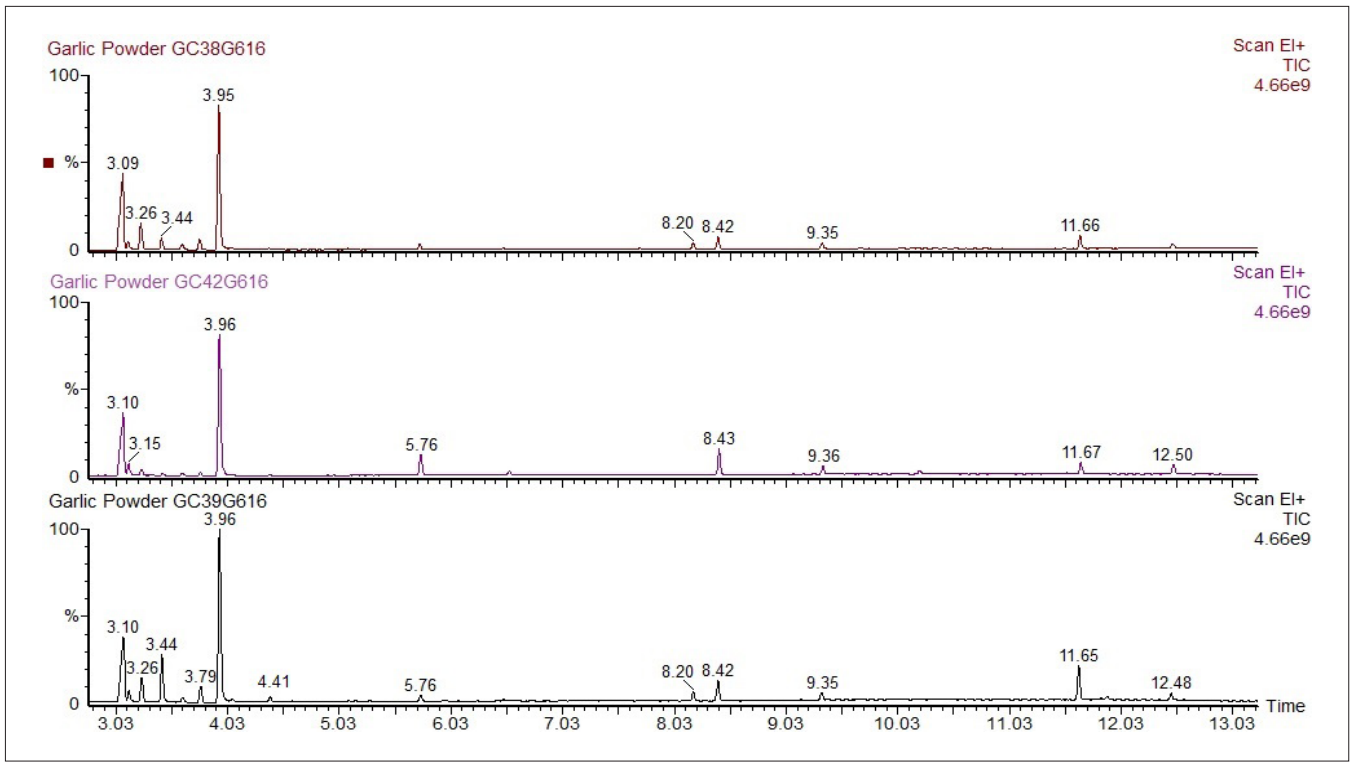

Figure 1. TICs of three garlic powder samples.

Component identification was achieved using the National Institute of Standards and Technology (NIST) Mass Spectral Database. Several sulphur-containing compounds were found in the garlic powder samples, with many being common to all samples. Table 4 shows the identified compounds in garlic powder GC-39G616. The garlic profiles showed some differences between samples. Samples GC-39G616 and GC-38G616 had an extra peak at 8.20 min which was not present in GC-42G616. NIST matching identified this peak as heptenal. Peak intensities varied largely with the garlic powder samples. The most dominant sulphur containing compounds for all samples were diallyl sulfide and diallyl disulfide. The unidentified peak at 3.09 minutes was common to all samples and blanks.

Table 4. Tentative identification of compounds based on NIST library matching for GC 39G616 garlic powder sample.

Time (min)	Compound
3.26	Acetaldehyde
3.44	Ethanol
3.79	Dimethylsulfide
3.95	2-propen-1-ol
4.41	Allyl mercaptan
5.76	1-propene, 1-methylthio-,(Z)-
8.20	Heptenal
8.42	Diallyl sulphide
9.35	Disulfide, methyl 2-propenyl
11.65	Diallyl disulphide
12.48	Trisulfide, methyl 2-propenyl

Conclusion

This application brief demonstrates the use of the PerkinElmer Clarus SQ 8 GC/MS with Headspace Turbomatrix Sampler for qualitative analysis of garlic powder aroma and flavour compounds as an indicator of garlic quality. It has been shown to be an effective and powerful tool for identification of unknown garlic aroma and flavour compounds, with no sample preparation to allow simple and fast characterisation.

References

1. E. Block, Garlic and Other Alliums: *The Lore and the Science,* RSC Publishing, 2010.
2. J.S. Pruthi, *Quality Assurance in Spices and Spice Product,* Allied Publishers Limited, 1998.

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