Rapid Characterization of Garlic Volatiles No Sample Prep Required!

Using Headspace GC/MS and an Rxi®-5ms Capillary Column

By Julie Kowalski, Innovations Chemist; Michelle Long, Innovations Chemist;
Jason Thomas, Innovations Chemist; and William Goodman*, GC/MS Applications Specialist

- No sample preparation! Eliminate complicated steps required by other methods.
- Rapid screening of garlic-specific flavor and odor compounds.
- Speedy determination of volatiles profile.

Garlic, *Allium sativum* (L.), has a rich history in cooking and medicinal use. Recently, garlic supplements have gained popularity for boosting immune and cardiovascular health. Chromatographic methods for garlic are used by the dietary supplements industry to detect volatiles, such as sulfide degradents, that may affect the acceptability of supplements to the consumer. The headspace gas chromatography mass spectrometry (HS GC/MS) method for garlic and garlic powder shown here requires no sample preparation—making the bench work simple and fast. Other methods involve steam distillation, solid phase trapping solvent exchange, headspace solid phase microextraction, and simultaneous distillation and solvent extraction, which can be difficult and time-consuming.

This HS GC/MS analysis was done using a 30m x 0.25mm ID x 1.0µm Rxi®-5ms column and a PerkinElmer TurboMatrix 40 Trap Headspace Sampler. Conditions used are shown in the figure and were set to optimize the comparison. Several sulfur components were identified including allyl methylsulfide, 3,3'-thiobis-1-propene, allyl mercaptan and diallyl disulfide. Diallyl disulfide appeared to be the dominant component for both garlic preparations. The fingerprint, or relative ratios, of the other components were distinct for fresh garlic and powdered garlic (Figure 1).

Headspace GC/MS is an effective technique for rapid characterization of garlic and garlic powder samples. The experimental set-up shown here is ideal for both screening and low-level trace analysis. This method provides a fast assessment of garlic quality and is applicable to the determination of low-level sulfur containing compounds from odorless supplements.

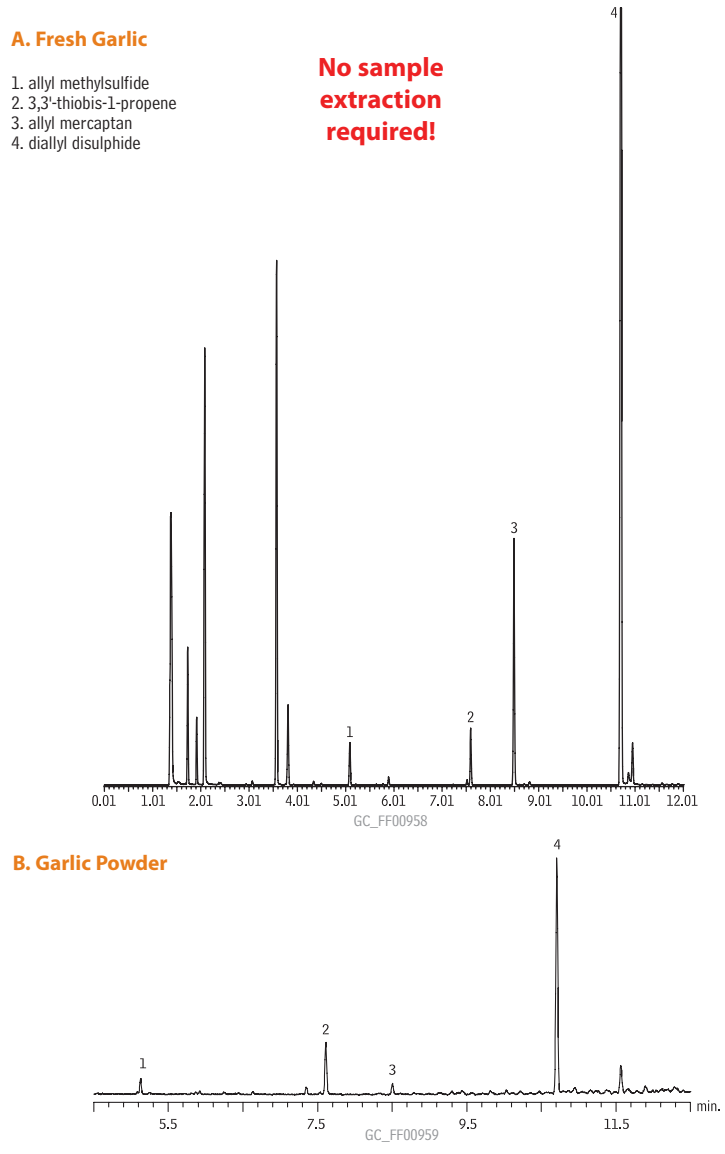
* PerkinElmer

Rxi®-5ms Column (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.25mm	1.00	-60 to 330/350°C	30-Meter	13453

Figure 1 Rapid screening of garlic volatiles—analyze samples in less than 11 minutes! (Total ion chromatogram)



Column: Rxi®-5ms, 30m, 0.25mm ID, 1.0µm (cat.# 13453) with a 5m, 0.32mm ID IP deactivated guard column (cat.# 10044); Sample: A. fresh garlic B. garlic powder
Inj.: split (10:1); Inj. temp.: 220°C; Flow rate: 1.5mL/min.; Oven temp.: 35°C (hold 1 min.) to 220°C @ 15°C/min. to 300°C @ 45°C/min.; Det: MS; Scan range: 35-350amu; Ionization: EI; Mode: scan
Headspace Conditions
Instrument: PerkinElmer TurboMatrix 40 Trap Headspace Sampler; Column pressure:15psi (103kPa); Inj. pressure: 30psi (207kPa); Thermostat time: 15 min.; Vial pressurize time: 1 min.; Withdraw time: 0.2 min.; Injection time: 0.02 min.; Oven temp.: 80°C; Needle temp.: 90°C; Transfer temp.: 110°C