APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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Qualifying Mustard Flavor by Headspace Trap GC/MS using the Clarus SQ 8

Mustard is a common condiment used across many cultures and culinary styles to enhance the dining experience. It is derived from the mustard seed and is used either as a dried spice, spread or paste when the dried spice is mixed with water, vinegar or other liquid. The characteristic sharp taste of mustard arises from the isothiocyanates (ITCs) present as result of enzymatic activity made possible when the ground seed is mixed with liquids. The focus of this application brief is the characterization of these ITCs by headspace trap gas chromatography/mass spectrometry (GC/MS) and a qualitative description of their relationship to sharpness in taste across various mustard products.

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Method

The experimental conditions for this analysis are given in Tables 1 to 4. The vials used are the standard 22-mL vials with aluminum crimped caps with PTFE lined silicon septa.

Table 1. **GC Conditions.**

Gas Chromatograph Clarus® 680	
Column	60 m x 0.25 mm x 1.0 µm Elite-5MS
Oven	35 °C for 5 min, then 6 °C/min to 245 °C
Injector	Programmable Split Splitless (PSS), 180 °C, Split OFF
Carrier Gas	Helium at 2.0 mL/min (28.6 psig initial pressure), HS Mode ON

Table 2. **HS Trap Conditions.**

Headspace System	**TurboMatrix™ 110 HS Trap**
Vial Equilibration	80 °C for 20 minutes
Needle	120 °C
Transfer Line	140 °C, long, 0.25 mm i.d. fused silica
Carrier Gas	Helium at 31 psig
Dry Purge	7 min
Trap	CarboPack C, 25 °C to 260 °C, hold for 7 min
Extraction Cycles	1 @ 40 PSI

Table 3. **MS Conditions.**

Mass Spectrometer	**Clarus® SQ 8S**
Scan Range	35 to 350 Daltons
Scan Time	0.1 s
Interscan Delay	0.06 s
Source Temp	180 °C
Inlet Line temp	200 °C
Multiplier	1700V

Table 4. **Sample Details.**

Sample	**Sample Weight (g)**
Mustard Seed (ground)	0.50
British Mustard Powder (dry)	0.50
British Mustard Powder (reconstituted)*	1.00
British Mustard	1.00
French Mustard	1.00

**reconstituted per manufacturer instructions*

Results

The total ion chromatogram obtained from French and British mustard samples are given in Figure 1. These intensity locked spectra demonstrated the higher level of ITCs present in British mustard as indicated by the larger peaks for allyl isothiocyanate (RT = 21.00 min) and 4-isothiocyanato-1-butene (RT = 24.28 min). This difference is indicative of the sharp verses smooth taste between British and French mustard.

The large presence of ITCs shown in Figure 1 is contrasted with the total ion chromatogram obtained from ground mustard seed given in Figure 2. In general ground mustard seed lacks volatile flavor compounds because the enzymatic activation with liquid has not been performed. Figure 3 demonstrates this activation with a comparison between dry British mustard powder and a reconstituted sample. In these labeled total ion chromatograms the intensity scales are locked between the spectrum and it is clear that activation with water has drastically increased ITC level in the reconstituted paste. The component identities were established by performing mass spectral library searches with the best match presented here. Peaks labeled with a single asterisk indicate detector overloading while peaks labeled with a double asterisk indicate inconclusive compound identification due to structural similarities.

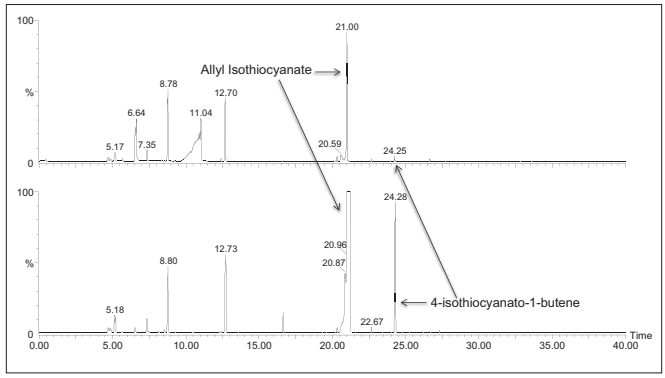

Figure 1. Full total ion chromatogram obtained from French (top) and British (bottom) mustards.

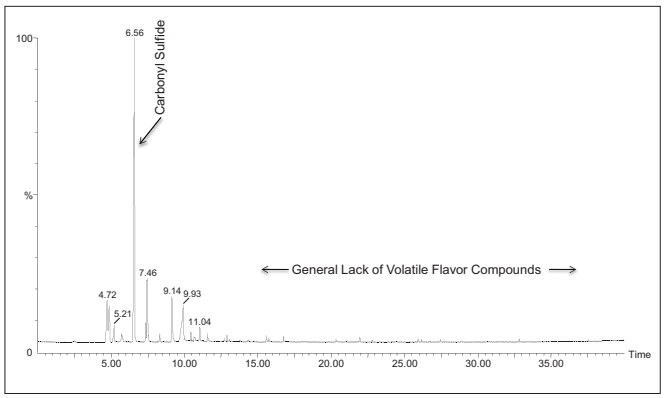

Figure 2. Full Total Ion Chromatogram obtained from mustard seed sample.

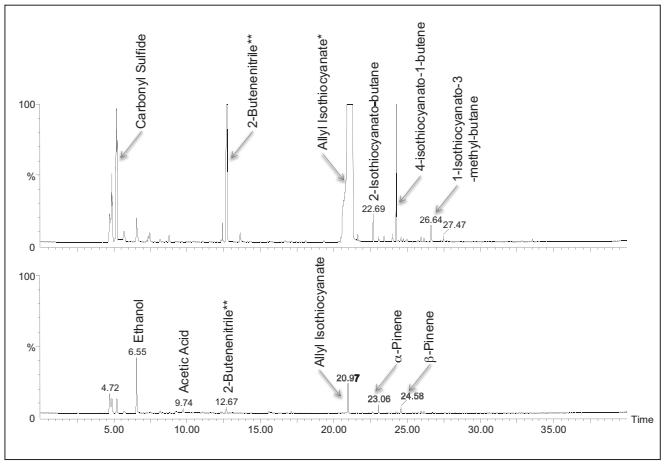

Figure 3. Full total ion chromatogram obtained from British mustard powder (bottom) and reconstituted British mustard powder (top).

Conclusions

This system provides a very simple and convenient way of characterizing the volatile flavor components of mustard based products. A rapid comparison between production samples may be made to monitor the enzymatic activation process and help producers arrive at the correct sharpness in taste. The combination of HS Trap with GC/MS allows for component detection at low-level concentration combined with the mass spectral compound identification.

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