APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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Analysis of Consumer Products by Headspace Trap GC/MS using the Clarus SQ 8

Fragrant soaps and detergents are a ubiquitous part of our modern society and add a certain romance to what would otherwise be mundane household chores. Great care and expense is spent in formulating the exact mixture of fragrant organic compounds to differentiate "apple blossom" or "crisp apple" from simple "apple" scent. At the other end of the spectrum are "fragrance-free" products – products that contain no fragrance producing organics, natural or otherwise. Unfortunately the term "fragrance-free" is unregulated and the actual composition of these products is left to the manufacturer's discretion. For both situations a comprehensive analytical technique is necessary to measure both composition and quality of any volatile organic compounds present. In this application brief we describe a quick and simple analytical technique using headspace trap gas chromatography/mass spectrometry (GC/MS) to determine the volatile fragrance compounds contained in various consumer 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. Liquid samples were placed directly into the sample vials. The two solid bar soap samples were thinly sliced into the sample vials to produce a maxima of surface area.

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 min
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**
Liquid fabric softener	0.50 g
Liquid laundry detergent	0.50 g
Fruit scented dishwashing liquid	0.50 g
Fruit scented liquid hand soap	0.50 g
Fruit scented bar hand soap	0.50 g
"Fragrance-free" facial cleanser bar	0.50 g

Results

The total ion chromatogram obtained from the dishwashing liquid, fabric softener and laundry detergent samples are given in Figure 1. Compound identification is performed using the installed spectral search functionality in the TurboMass™ software in conjunction with the NIST® library and represents the highest probability result. Where possible the common (non-IUPAC) name is used. Compounds labeled with an asterisk (*) indicate a saturated signal and (fn) indicates compounds that likely serve a non-scent related function in the product. Both the fabric softener and laundry detergent, though not marketed directly as fragranced, contain a number of fragrant organic compounds that produce their characteristic sweet scent. The dishwashing liquid contains, in addition to the large limonene and ethyl methylbutyrate peaks, a number of additional trace fragrant compounds as illustrated in Figure 2. This complicated combination gives rise to its "apple blossom" scent. The total ion chromatogram obtained from the liquid hand soap sample, given in Figure 3, also presents a very refined combination of fragrant organic compounds. The combination of fruit scent combined with the smooth butter gives rise to its "white citrus" scent.

The total ion chromatograms from the two bar soap samples are given in Figure 4. The fruit scented bar soap clearly contains higher concentrations of fragrant organic compounds but the "fragrance-free" facial cleanser bar also contains these compounds. Figure 5 gives a zoom of the total ion chromatogram illustrating the low-level fragrant content of the facial cleanser bar. While the concentration is substantially less their presence is clearly detected. As discussed in the introduction, the use of the term "fragrance-free" is left to the description of the manufacturer and is not regulated and the consumer must be aware of these discrepancies.

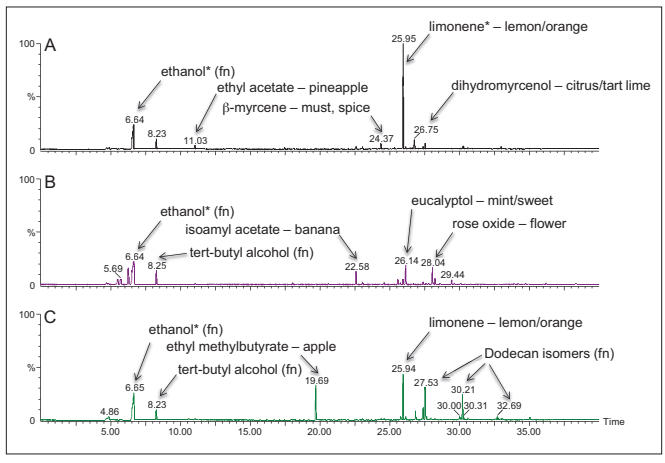

Figure 1. Full Total Ion Chromatograms obtained from (A) laundry detergent, (B) liquid fabric softener and (C) fruit scented dishwashing liquid with major fragrance compounds and scents labeled.

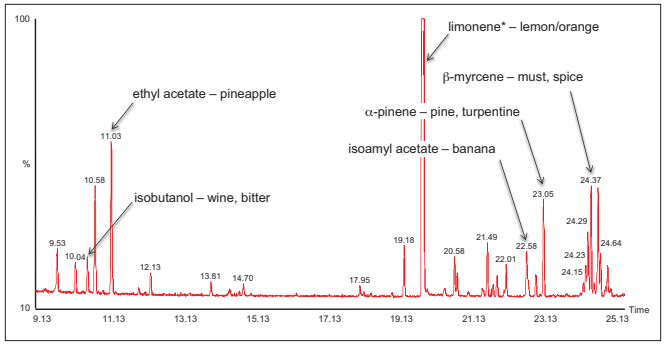

Figure 2. Zoom of Total Ion Chromatogram obtained from liquid hand soap.

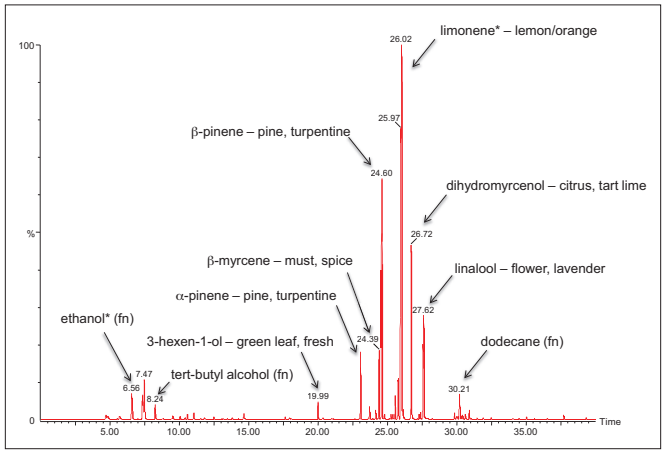

Figure 3. Full Total Ion Chromatogram obtained from fruit scent liquid hand soap.

Conclusions

In this application brief we outline a simple and comprehensive technique for the analysis of fragrance causing organic compounds in various consumer goods. The combination of GC/MS with HS Trap delivers enhanced sensitivity that provides for positive identification of individual components at low concentration levels. This technique has application in both formulation and regulation of these products and can help bring clarity to the issue of "fragrance-free" products.

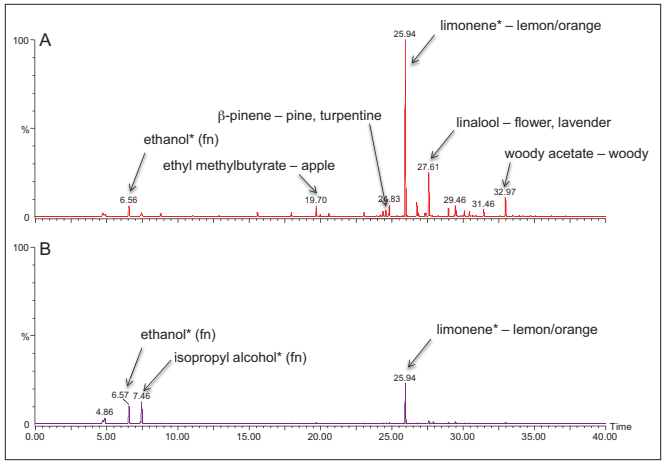

Figure 4. Full Total Ion Chromatograms obtained from (A) fruit scented bar soap and (B) "fragrance-free" facial cleanser bar.

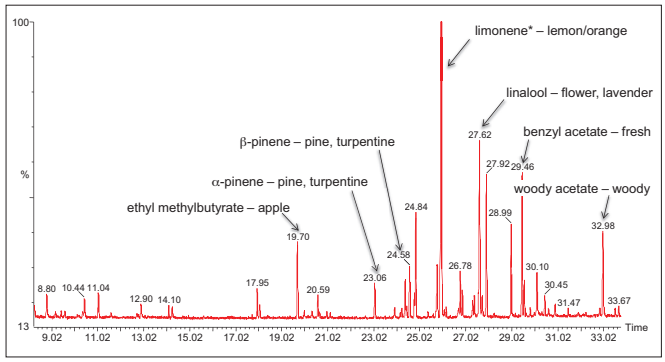

Figure 5. Zoom of Total Ion Chromatogram obtained from "fragrance-free" facial cleanser bar.

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