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Analysis of Fast Moving Consumer Goods (FMCG) using GCMS with static and dynamic headspace (HS)

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Introduction

Accurate screening, characterization and quantitative analysis of flavour & fragrance (F & F) components is essential for FMCG industries and subsequent reverse engineering, especially when unknown finished good to be analysed is in solid form. Conventional methods of GC analysis often lead to qualitative results and are time consuming and troublesome with regards to sample preparation techniques.

In this analysis, neat consumer products (Figure 1) were analysed directly in HS sampler, applying different analytical techniques like static and dynamic headspace and their results were compared. In both the headspace technique it requires minimal sample preparation that significantly reduces overall analysis time without sacrifice in quality data.

F & F components were determined at trace levels by Shimadzu GCMS-QP2010 Ultra with HS-20 Trap system.

Figure 1. Consumer products

Method of Analysis

Extraction of F & F from consumer sample

Commercially available consumer products like soap, shower gel, tooth paste, body lotion and orange juice were purchased from local market. Static (Loop) and Dynamic (Trap) headspace techniques were employed for qualitative analysis.

For sample preparation (Figure 2), individual products were weighed in HS vial and crimped immediately using Aluminium cap with PTFE/ Silicon septum.

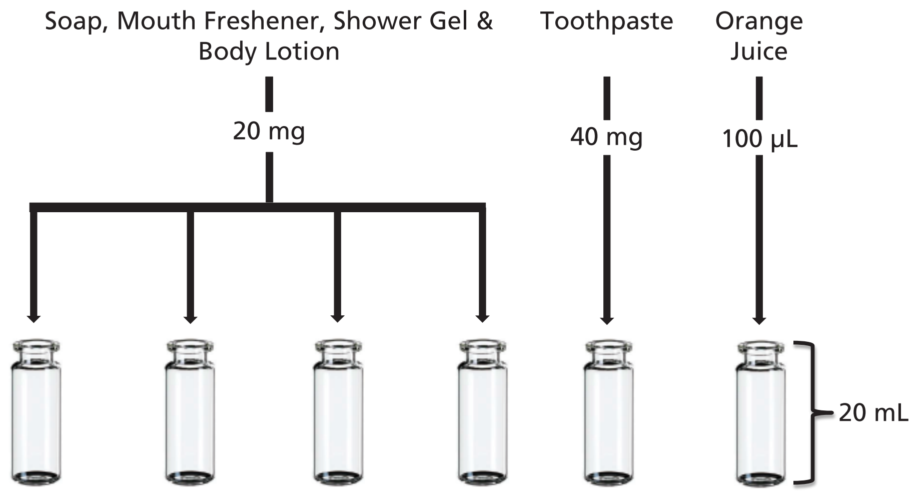

Figure 2. Representation of sample preparation for HS analysis

Loop technique employed single extraction of sample in static mode whereas dynamic technique used multiple extractions to concentrate sample in trap, which were further analysed by GC-MS.

Figure 3. GCMS-QP2010 Ultra coupled with HS-20 System by Shimadzu

HSGC-MS Analytical conditions

The instrument configuration used is shown in Figure 3. Samples were analyzed using HS-20 coupled with GCMS-QP2010 Ultra as per below conditions as shown in Table 1.

Table 1. Analytical conditions

Headspace Parameters

Parameter	Loop	Trap
Mode	Loop	Trap
Oven Temp. (Juice sample)	130 °C (80 °C)	130 °C (80 °C)
Sample Line Temp.	150 °C	150 °C
Transfer Line Temp.	180 °C	180 °C
Trap Cooling Temp.	NA	-20 °C
Trap Desorb Temp.	NA	300 °C
Trap Equilib. Temp.	NA	-10 °C
Multi Injection Count	1	5
Pressurizing Gas Pressure	103 kPa	103 kPa
Equilibrating Time	30.0 min	30.0 min
Pressurizing Time	1.0 min	1.0 min
Load Time	0.50 min	0.50 min
Injection Time	1.0 min	10.0 min
Needle Flush Time	45.0 min	45.0 min
GC Cycle Time	55.0 min	55.0 min

Chromatographic Parameters

Column : Rxi-5Sil MS (30 m L x 0.25 mm ID x 0.25 µm)
Injection Mode : Split
Split Ratio : 100 (5.0 for Juice sample)
Carrier Gas : Helium
Flow Control Mode : Linear Velocity
Linear Velocity : 36.3 cm/sec
Pressure : 53.6 kPa
Column Flow : 1.00 mL/min
Total Run Time : 45.0 min
Column Oven Temp :

Rate °C/min	Temperature °C	Hold time (min)
----	50.0	0.0
5.0	250.0	5.0

Mass Spectrometry Parameters

Ion Source Temp : 200 °C
Interface Temp : 250 °C
Ionization Mode : EI
Event Time : 0.30 sec
Mode : Scan
Start m/z : 40
End m/z : 400

Results

Sample analysis using HS Loop and Trap technique

Same amount of samples of different products were analyzed on HSGC-MS loop and trap mode to compare the sensitivity. Difference in number of peaks and their comparative areas using two techniques are shown in Table 2 and 3 respectively. The chromatograms are shown in figure 4.

Table 2. Comparative result of Loop and Trap mode analysis

Summary of Comparison Between Loop and Trap for Different Products					
Sr. No.	Product	Vial Temp °C	Split Ratio	HS Mode	HS Mode
1	Orange Juice	80	5	Loop	3
				Trap	28
2	Toothpaste	130	100	Loop	66
				Trap	183
3	Mouth Freshner	130	100	Loop	56
				Trap	175
4	Shower Gel	130	100	Loop	86
				Trap	136
5	Body Lotion	130	100	Loop	33
				Trap	82
6	Soap	130	100	Loop	58
				Trap	107

Table 3. Area comparison from Loop and Trap Mode for Major Components in Different Products

Sr. No.	Product	Components	Area Loop (x)	Area Trap	Increased Area in Trap
1	Orange Juice	Limonene	1505291	51859804	34x
		Pentane, 2,2,4-trimethyl-	27205	3670523	135x
		Terpineol <alpha->	59883	2782693	46x
2	Toothpaste	Menthol	26911667	469422497	17x
		Camphor	32963052	437964646	13x
		Eugenol	21062093	391114898	19x
3	Mouth Freshner	Menthol	24864908	488158291	20x
		Propylene Glycol	2806236	223693653	80x
		Terpinyl acetate	4796880	221454317	46x
4	Shower Gel	Benzeneethanol	5047951	77003506	15x
		Linalool	5608303	68041272	12x
		Acetic acid, phenylmethyl ester	4480261	55205250	12x
5	Body Lotion	Phenoxyethanol	12877588	176739640	14x
		Isopropyl palmitate	8890582	133791601	15x
		Heptadecanol <n->	2986315	67216756	23x
6	Soap	Dihydromyrcenol	2977853	48902570	16x
		Cyclohexanol <2-tert-butyl-, trans-> acetate	1968123	39508536	20x
		Linalyl acetate	762749	34916396	16x

Table 4. Reproducibility data for Limonene from orange juice in Trap mode

Sr. No.	Product	Component	% RSD (n=6)
1	Orange Juice	Limonene	7.0

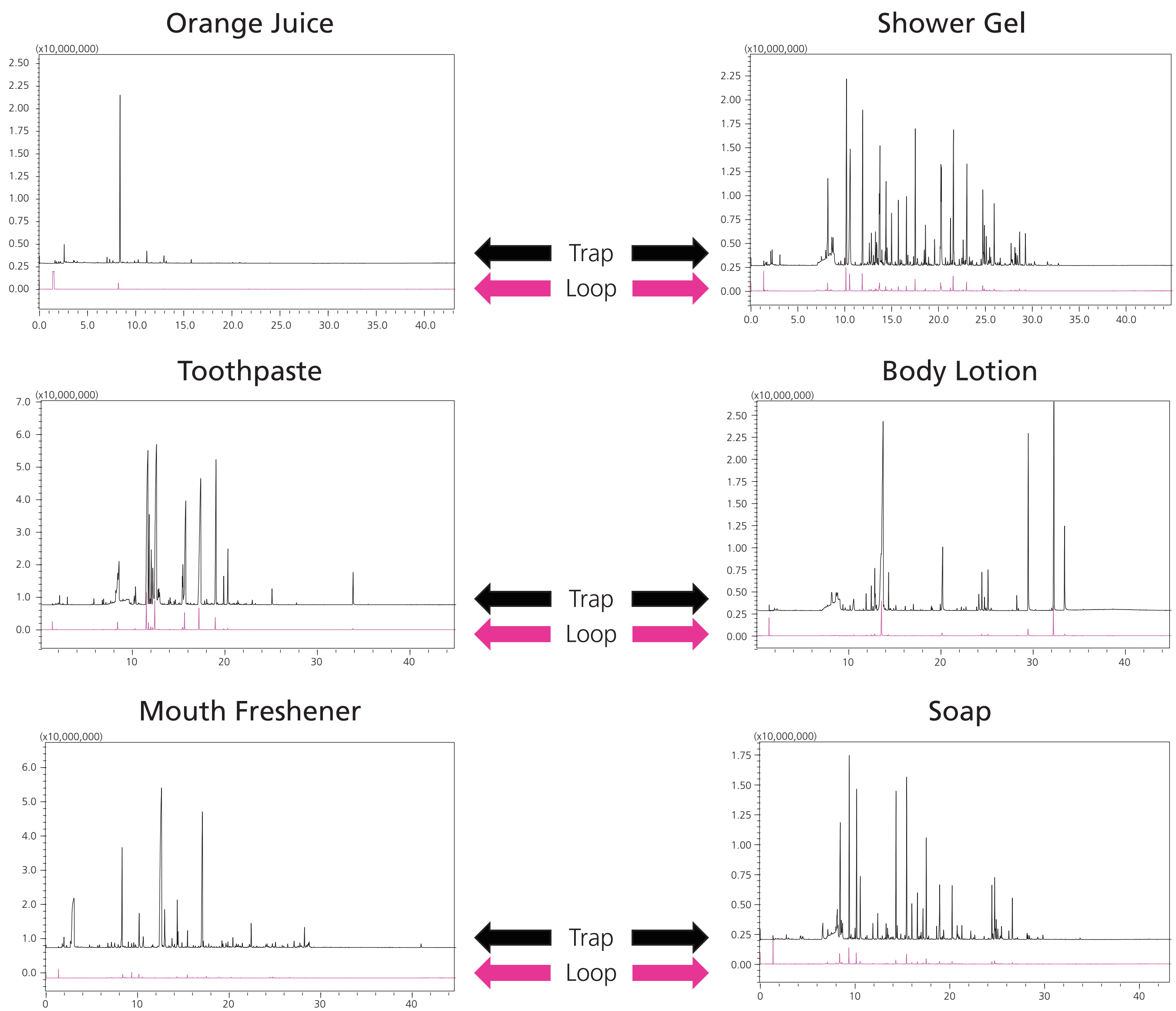

Figure 4. Overlay of Loop and Trap chromatograms for different products.

Conclusions

- HSGC-MS method was developed for qualitative of consumer products. Comparative data was generated using static and dynamic headspace techniques for consumer products.
- Both technique can be used as valuable tools for analyzing a variety of matrices. Statistical evaluation of the data showed that the dynamic HS technique method was more superior with respect to sensitivity and reliability as compared to static HS technique.
- The unique configuration of flow lines and the HS oven enable the analysis of high boiling point compounds while minimizing carryover. In addition, by using a trap function that incorporates an electronic cooling mechanism, it is possible to concentrate the headspace gas, which enables high-sensitivity analysis of low to high boiling point compounds.

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