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Packaged Food Aroma Profiling with the Smart Aroma Database on GCMS

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Introduction

SPME sample preparation paired with the QP2020NX Shimadzu GCMS was used to analyze flavor components in food. The Smart Aroma Database was used to create a SIM method to browse for 489 specific food flavor targets using hydrogen carrier gas.

Smart Aroma Database

The Smart Aroma Database, containing over 500 flavor and fragrance components was used to develop SIM methods to isolate the database components in each sample. Information such as target ions, retention indices, and aroma descriptions are included for each component, making it easy to apply the data to any method. As many laboratories move towards helium alternatives, this demonstrates the capabilities of smart databases with hydrogen.

Serial#	Type	Acq. Mode	Compound Name (E)	Ret. Index 1	Ret. Time	Comment (E)	Ion1			Ion2			Ion3		
				SH-I-5-Sil M		Odor Quality	Type	m/z	Rati	Type	m/z	Rati	Type	m/z	Rati
1	Target	SIM	3-Methylbutanal	678	0.252	malt	T	71.0	100.00	Ref.1	58.0	313.23	Ref.2	86.0	37.38
2	Target	SIM	3-Methyl-2-butanone	679	0.269	camphor	T	86.1	100.00	Ref.1	43.1	544.90	Ref.2	41.1	75.38
3	Target	SIM	1-Butanol	679	0.269	medicine, fruit	T	56.1	100.00	Ref.1	41.1	66.67	Ref.2	55.1	20.11
4	Target	SIM	2-Methylbutanal	683	0.336	cocoa, almond	T	86.1	100.00	Ref.1	57.1	664.54	Ref.2	58.1	498.46
5	Target	SIM	Thiophene	685	0.370	garlic	T	84.0	100.00	Ref.1	58.0	65.51	Ref.2	45.0	31.90
6	Target	SIM	Propanoic acid	686	0.387	pungent, rancid, soy	T	73.0	100.00	Ref.1	74.0	144.91	Ref.2	45.0	90.75
7	Target	SIM	Methyl isobutyrate	690	0.455	flower	T	102.1	100.00	Ref.1	71.1	345.53	Ref.2	87.1	289.85
8	Target	SIM	1-Penten-3-ol	690	0.455	butter, pungent	T	57.1	100.00	Ref.1	67.1	1.53	Ref.2	71.1	1.80
9	Target	SIM	Ethyl vinyl ketone	691	0.472	fish, pungent	T	84.1	100.00	Ref.1	55.0	548.39	Ref.2	83.1	52.34
10	Target	SIM	2-Pentanone	692	0.489	ether, fruit	T	86.1	100.00	Ref.1	43.1	538.84	Ref.2	41.1	53.49
11	Target	SIM	3-Pentanone	697	0.574	ether	T	86.1	100.00	Ref.1	57.1	462.60	Ref.2	56.1	22.99
12	Target	SIM	2,3-Pentanedione	697	0.574	cream, butter	T	100.1	100.00	Ref.1	43.1	432.75	Ref.2	57.1	232.25

Method information

Compound information

Mass spectral information

Figure 1: A snapshot of the Smart Aroma database showing sections of instrument condition selections in the method information section, compound names, retention indices and corresponding calculated retention times, as well as qualitative odor descriptions in the compound information section, and finally the mass spectral information here showing the optimum SIM target ions used in this method.

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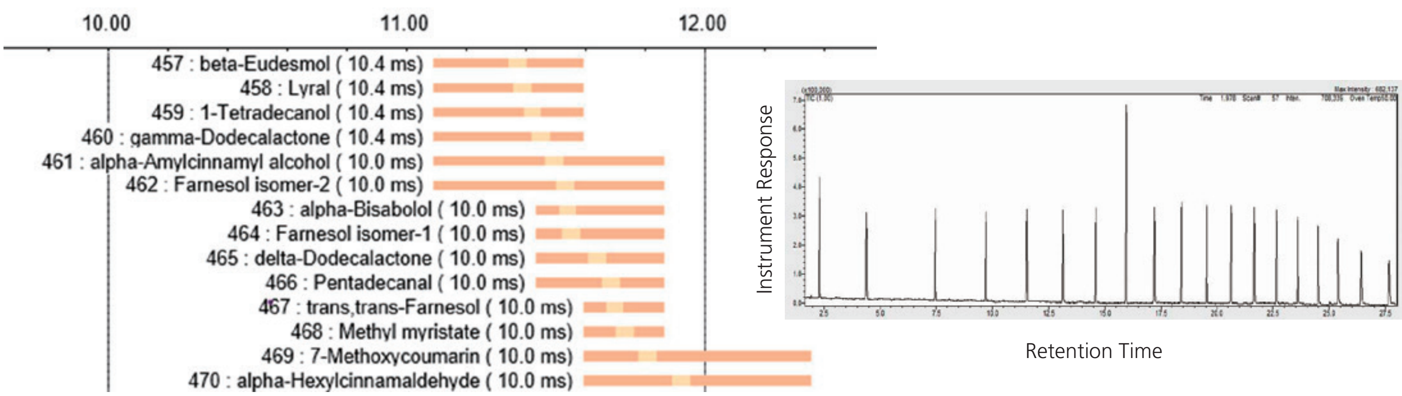

Figure 2: Left: Graphed results of the automatic SIM method creation using the Smart Aroma Database. Dark orange bands show the SIM window, while the light shaded section shows the estimated elution time of the compound. Right: Chromatogram of a mixture of n-alkanes using the same conditions as in the flavor data acquisition method.
This data is applied to the Smart Aroma Database preloaded with retention index information to generate the results on the left.

Method conditions from the Smart Aroma Database recommended methods were followed and are described along with the SPME conditions in Table 1. An alkane standard was used to automatically create a SIM method using the retention index information stored in the database, a snapshot of which can be found in Figure 2. The resulting method is visualized in Figure 2 as created by the Smart Aroma Database and described in detail in Table 1. Highlights in the graph show the expected compound elution time to best optimize the SIM method.

Table 1 Instrument Conditions

GCMS	: GCMS-QP2020 NX
Software	: GCMS Solution 4.53
Autosampler	: AOC-6000
Column	: Rxi-5ms capillary (30 m x 0.25 mm i.d. ft: 0.25 µm)

SPME Method Conditions		GCMS Conditions	
Fiber coating	: Carbon WR/PDMS	Carrier Gas	: Hydrogen
Temperature	: 60 °C	SPL gas control	: Constant Linear Velocity 72.3 cm/s
Incubation Time	: 5 minutes	SPL Temperature	: 250 °C
Inc. Agitation Speed	: 250 rpm	SPL mode	: Splitless; sampling time: 1 minute, 5:1 split afterwards
Extraction Time	: 10 minutes		
Ext. Agitation Speed	: 250 rpm	Purge flow rate	: 3.0 mL/min
Desorption Time Oven Program	: 10 minutes	Ion source	: 200 °C
Start Temperature	: 45 °C	Interface	: 250 °C
Hold Time	: 7 min	Ramp	: 10 °C/min
		End Temperature	: 250 °C
		Hold Time	: 10 min

SPME and Sampling

SPME or solid phase microextraction was used for this analysis on the food products as it is a reusable qualitative and quantitative sampling technique that can collect volatile flavor components. It helps pre-concentrate target analytes for injection into the GCMS while avoiding many matrix components. This is particularly useful when sampling food products that pose difficulties in more conventional sample introduction, like ice cream and chips.

Food samples of cheese flavored chips and vanilla ice cream with chocolate cookies were obtained from local markets. Ice cream was melted and weighed into headspace vials to reach 1g per vial. The melted cream was mixed to ensure distribution of particulates in the sample. Chips were crushed on wax coated weigh-paper and poured into vials, also to reach 1g each.

Ice Cream Aroma Profiling

Ice cream was profiled using both an extended Scan and the SIM method designed using the Smart Aroma Database. Key flavor components like hexanal, benzaldehyde, terpinen-4-ol, and vanillin were easily identified by the SIM method, integration extracted chromatograms for which are shown in Figure 3. Each of these is an expected aroma in ice cream, corresponding to aromas of fat, sweet almond, warm spices and vanilla, respectively. The database allows for a browse of presence or absence of peaks in SIM mode, based on expected elution time and target ions, and also provides an odor description.

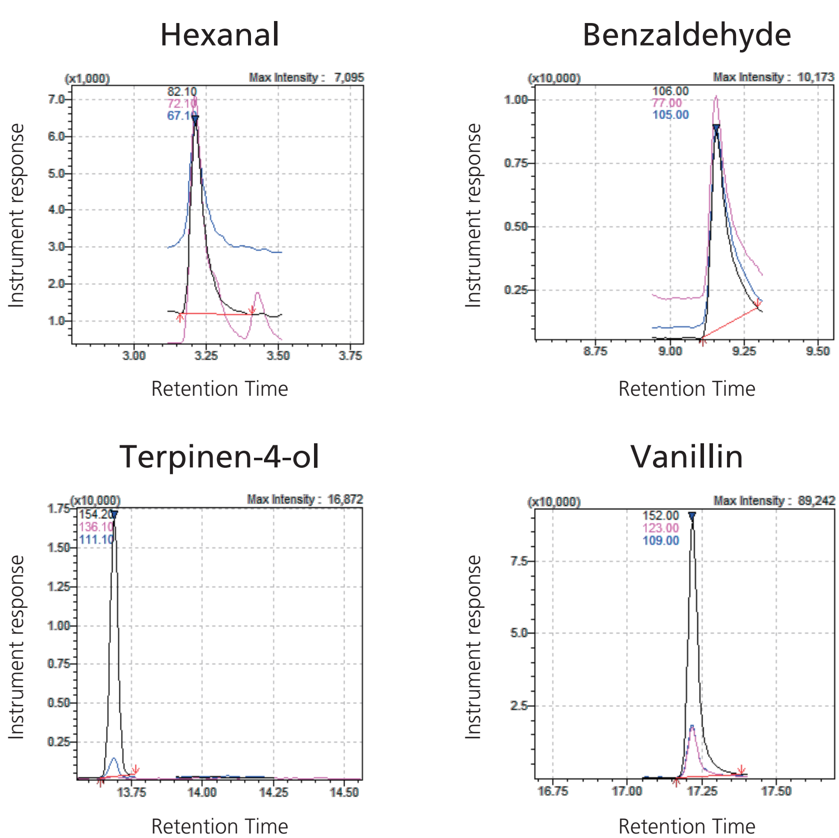

Fig. 3 Select compounds showing automatic identification of aroma in ice cream

Fresh vs Stale Chip Aroma Comparison

Cheese flavored chips were profiled using the same method conditions. Notable components included Z-β-ocimene, S-ethyl thioacetate, 2,4-decadienal and acetophenone, shown in figure 5. These compounds correspond to flavors that typically would be expected in a seasoned fried chip, such as alum flavors of garlic and onions coming from S-ethyl thioacetate, nutty and fatty fried flavors from 2,4-decadienal and acetophenone, and herb seasoning from the remaining Z-β-ocimene. The same chips underwent an aging process and were tested for differences in flavor profiles to monitor a change in results as they became stale. Figure 4 shows the differences in chromatograms obtained from both stale (pink) and freshly opened (black) chips. While many components appear to remain constant, some decrease significantly in response, while a select few increase and likely contribute to a stale odor. Figure 5 shows 4 components that showed changes.

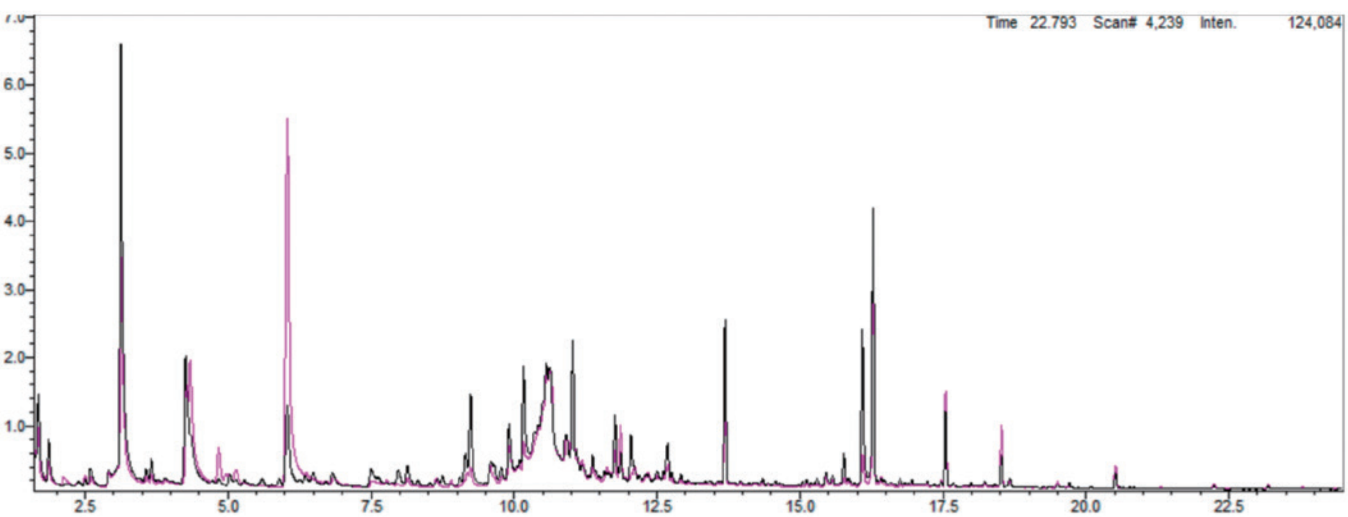

Fig. 4 Scan chromatograms of freshly opened (black) and stale (pink) chips showing some constant, yet some changing aroma components after product aging

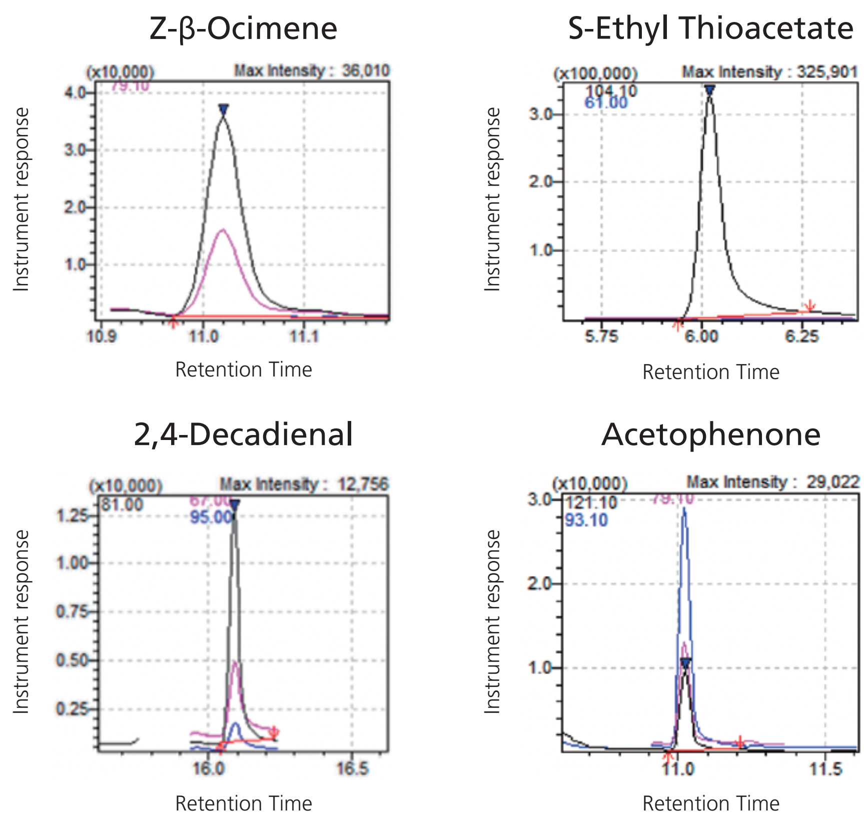

Fig. 5 Select compounds identified in chips which changed in concentration when the product underwent aging.

Over time, the chips resulted in about an 80% loss in response from S-Ethyl Thioacetate, responsible for seasoning flavors expected from the product description. Other components like 2,4-Decadienal increased in response, corresponding to greasy or fatty flavor markers. Trends for select compounds are shown in Table 2.

Table 2: Direct comparison of targeted compounds in fresh and stale chips

No.	Name	Retention Time	M/Z ratio	Fresh Chip Area Count	Aged Chip Area Count	Aroma
43	S-Ethyl Thioacetate	6.018	104	1355699	307112	onion, garlic
179	(Z)-beta-Ocimene	11.021	93	72890	542	citrus, herb, flower
199	Acetophenone	11.868	105	67380	13678	must, floral, almond
362	2,4-Decadienal	16.089	81	28751	137742	fried, wax, fat

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Conclusions

The techniques used in this qualitative work demonstrate the use of SPME GCMS with targeted screening for flavor profiling, comparison, and monitoring of product degradation and shelf life. Clear reduction in response was seen for select compounds in stale chips when compared to a fresh package. Ice cream was scanned for presence of select aroma components which can help in new product development and manufacture. The approach used is a reusable technique that prevents matrix introduction into the GCMS, improving instrument uptime.

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