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Application News

GC-MS GCMS-QP2020 NX

Analysis of Aroma for Beverage Quality Control Using Smart Aroma Database™ and the Headspace Method

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User Benefits

- The Smart Aroma Database enables efficient analysis of aroma compounds using information for over 500 aroma-related compounds registered in the database.
- With the HS-20 NX, preparing samples and configuring analytical conditions are simple. It is especially useful for quality control of aroma compounds, which requires performing a large number of analyses.
- With FASST analysis, principal compounds can be monitored with SIM mode measurements, while other compounds can be simultaneously monitored with scan mode measurements.

■ Introduction

Beer, a malted beverage loved throughout the world, is manufactured by fermenting malt, but the aroma and taste vary depending on the type of malt and fermentation method. Aroma compounds in food and beverages are analyzed using a GC-MS system, which offers superior qualitative analysis capabilities, but determining which of the hundreds of detected compounds affect aroma requires a lot of work processing vast amounts of data. However, the amount of work involved in searching through that data can be significantly reduced by using a database containing previously registered compound information.

Assuming analysis for quality control purposes, this article describes using the headspace (HS) trap to collect aroma compounds from different types of beer made by different manufacturers using different methods and then analyzing the compounds by GC-MS.

The Smart Aroma Database was used for GC-MS analysis in the simultaneous scan/SIM mode (FASST analysis). While target compounds were analyzed with high sensitivity in the SIM mode, other aroma compounds were qualitatively analyzed based on scan results.

■ Key Features of the Smart Aroma Database

The Smart Aroma Database includes analytical condition settings and sensory information for over 500 important compounds that contribute to aroma. This means the database can be used to easily create methods without having to consider analytical conditions, by following the steps indicated in Fig. 2. For scan mode measurements, the presence of compounds registered in the database can be determined automatically based on retention time information, ion information, and mass spectrum information. For SIM and MRM mode analysis, methods can be created for high-sensitivity accurate analysis of compounds targeted for quality control.

Compound Name	Ret. Time	Comment Odor Quality
2-Methylfuran	4.946	chocolate, cocoa
Ethyl formate	5.145	pungent
Butanal	5.801	pungent, green
Ethyl acetate	5.972	pineapple
Acetal	6.172	fruit, cream
2-Butanone	6.172	ether

Compounds | Retention times | Aroma characteristics

Fig. 1 Information Registered in Smart Aroma Database

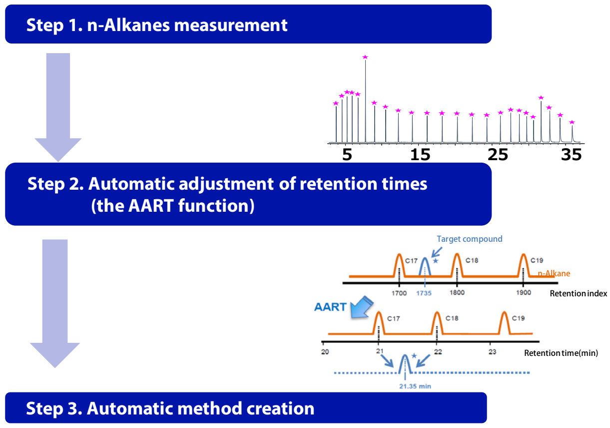

Fig. 2 Analysis Process Flow Using Smart Aroma Database

■ FASST Analysis and High-Speed Scan Control Technology

By using FASST analysis with high-speed switching between scan and SIM modes, target compounds can be measured with high sensitivity in the SIM mode while other compounds can be simultaneously monitored in the scan measurement mode. Furthermore, with Advanced Scanning Speed Protocol (ASSP™) high-speed scan control technology, scan data with excellent sensitivity and mass spectra can be obtained using FASST analysis.

■ Headspace (HS) Method

The headspace (HS) method is a technique for pretreating vaporized volatile compounds before injection into a GC unit. Due to the ease and simplicity of sample preparation for the HS method and the good reproducibility it provides, the HS method is commonly used for routine analysis in quality control applications. In this example, a trap model of the HS-20 NX (Fig. 3) was used to pretreat samples by the trap headspace method. The trap headspace method enables high sensitivity analysis of aroma compounds because it concentrates compounds in an electronically-cooled trap.

Fig. 3 HS-20 NX + GCMS-QP2020 NX

■ Beer Analysis Results Using the HS Method

In this example of routine quality control analysis, an HS-20 NX + GC-MS system was used to analyze seven key aroma compounds (ethyl acetate, isobutanol, isoamyl acetate, isoamyl alcohol, ethyl hexanoate, ethyl octanoate, and 2-phenylethanol) as target compounds by the SIM mode and analyze the other compounds by scan measurements in the FASST analysis mode (simultaneous scan/SIM measurements). 8 g of beer and 3 g of NaCl were placed in each vial. Then a 3-octanol internal standard was added to make a final concentration of 1 ppm and the solutions were measured. Analytical conditions are indicated in Table 1.

Area ratio comparison results from SIM measurement results for the seven types of beer are shown in Table 2. The results confirm that the white ale tends to contain higher concentrations of the seven key beer compounds. If target compounds are predetermined, as in this example, then SIM measurements can be used to monitor and compare concentrations with high sensitivity. In addition, the Smart Aroma Database was used to qualitatively detect 141 aroma compounds from FASST analysis scan measurement results. Results from principal component analysis of the detected compounds using SIMCA® 17 software (Infocom) are shown in Fig. 4. This enabled classification of each beer based on score plot results, as described in Application News 01-00316-JP.

Table 1 Analytical Conditions

Model:	GCMS-QP2020 NX	Loading Pressurization Time:	0.1 min
Autosampler:	HS-20 NX	Dry Purge Time:	10 min
[HS conditions]		Injection Time:	3 min
Mode:	Trap (Tenax®TA 60/80 mesh)	Needle Flush Time:	5 min
Multi Injection Times:	5	[GC conditions]	
Oven Temperature:	60 °C	Injection Mode:	Split
Sample Line Temperature:	100 °C	Split Ratio:	5
Transfer Line Temperature:	100 °C	Carrier Gas:	He
Trap Cooling Temperature:	-10 °C	Carrier Gas Control:	Pressure (83.5 kPa)
Trap Heating Temperature:	280 °C	Column:	InertCap Pure-wax (30 m × 0.25 mm I.D. 0.25 µm)
Trap Waiting Temperature:	25 °C	Column Temp.:	50 °C (5 min) – 10 °C/min – 250 °C (10 min)
Vial Pressure:	80 kPa	[MS conditions]	
Dry Purge Pressure:	60 kPa	Ion Source Temp.:	200 °C
Vial Heat-retention Time:	30 min	Interface Temp.:	250 °C
Vial Pressurization Time:	1 min	Acquisition Mode:	Scan/SIM
Vial Pressurization: Equilibrating Time:	0.1 min	Event Time:	0.3 sec (Scan), 0.2 sec (SIM)
		m/z Range:	*m/z* = 35-400 (Scan)
Loading Time:	1 min		Refer to Table 2 (SIM)

Table 2 Summary of SIM Measurement Results for Key Beer Compounds

Compound	Aroma Characteristics	*m/z*	Average Area Ratio for SIM Measurements (n = 3)						
			Ale 1	Ale 2	Barrel aged	IPA	Lager 1	Lager 2	White Ale
Ethyl acetate	Pineapple	43.0	16.5	16.3	23.0	18.1	17.2	17.0	23.2
Isobutanol	Wine, solvent, bitter	43.1	2.7	2.0	3.0	3.5	2.2	2.4	3.9
Isoamyl acetate	Banana	87.1	0.4	0.4	0.2	0.3	1.2	0.8	1.0
Isoamyl alcohol	Whiskey, malt, burnt	70.1	18.5	15.4	14.5	19.8	18.0	16.0	20.9
Ethyl hexanoate	Apple peel, fruit	70.0	0.3	0.3	0.7	0.5	0.3	0.2	0.5
Ethyl octanoate	Fruit, fat	127.0	1.3	0.8	2.0	1.3	1.2	0.7	1.6
2-Phenylethanol	Honey, spice, rose, lilac	92.0	2.6	2.3	2.0	2.8	2.5	1.9	2.3

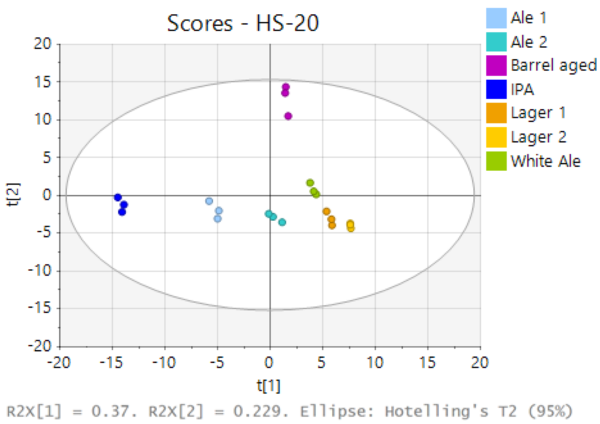

Fig. 4 Score Plot of HS-20 Measurement Results

■ Conclusion

Assuming a quality control application, this article describes using the HS method to pretreat samples for simultaneous SIM/scan analysis by the FASST analysis mode in a GC-MS system. Using SIM measurements to monitor key compounds and scan results to also confirm other compounds enabled confirmation of uncertain peaks. Furthermore, with 35 minutes spent per analysis, the target compounds can be monitored efficiently.

Whereas Application News 01-00316 assumes analysis is being performed for R&D, this Application News bulletin assumes aroma compounds are being analyzed for quality control purposes. Thus, the Smart Aroma Database can be used for a wide range of applications from R&D to quality control.

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