Application News

GC-MS GCMS-QP2020 NX

Analysis of Aroma for Beverage R&D Using Smart Aroma Database™ and an SPME Arrow

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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.
- This means methods can be created easily using the Smart Aroma Database without having to reconsider analytical conditions.
- Compounds can be analyzed with high sensitivity by concentrating compounds with an SPME Arrow. That is especially useful for research and development of aroma compounds.

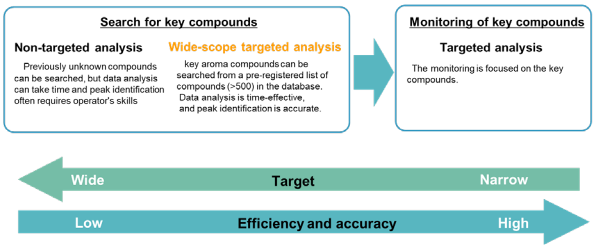
■ Introduction

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 used. 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 an R&D application, this article describes using an SPME Arrow to extract aroma compounds from samples of different types of beer made by different manufacturers using different methods, and then analyzing the aroma compounds by GC-MS. Results were also analyzed using the Smart Aroma Database, which contains information for over 500 compounds related to aroma. Identified compounds were analyzed by principal component analysis using SIMCA® 17 (Infocom) software to characterize and compare differences in aroma from the different types of beers made using different methods.

■ Key Features of the Smart Aroma Database

Attempting to comprehensively analyze samples with non-targeted analysis would require a tremendous amount of work to check the vast number of peaks detected and identification accuracy would decrease. In contrast, targeted analysis of only key compounds would increase identification accuracy but reduce the number of compounds targeted. Using the Smart Aroma Database enables the respective advantages of targeted and non-targeted analysis indicated above to achieve wide-scope targeted analysis. The database includes information about the retention times, mass spectra, and aroma characteristics of over 500 important compounds that contribute to aroma, and enables efficient analysis. This means the database can be used to easily create methods, without having to reconsider analytical conditions, by following the steps shown in Fig. 2. It also automatically detects aroma compounds registered in the database based on TIC chromatogram results from scan mode analysis.



Previously unknown compounds can be searched, but data analysis can take time and peak identification often requires operator's skills

key aroma compounds can be searched from a pre-registered list of compounds (>500) in the database. Data analysis is time-effective, and peak identification is accurate.

The monitoring is focused on the key compounds.

Fig. 1 Wide-Scope Targeted Analysis

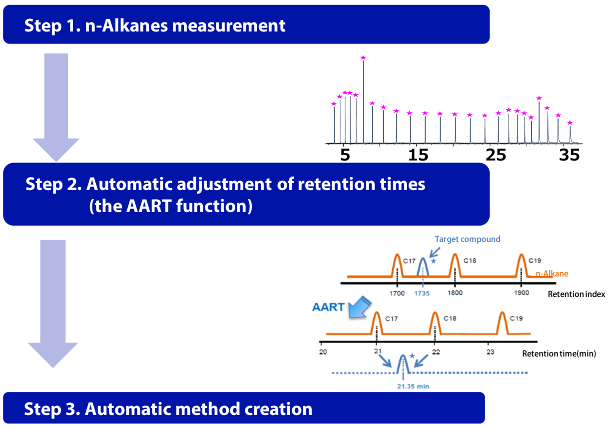


Fig. 2 Analysis Process Flow Using Smart Aroma Database

■ SPME Arrow Method

Solid phase microextraction (SPME) is a method used to adsorb compounds into a fiber to concentrate them before injection into a GC unit. That offers the advantage of higher sensitivity analysis. In this case, a large volume of sorbent was retained and an SPME Arrow was used to enable even higher sensitivity analysis compared with regular SPME. By using an AOC™-6000 Plus pretreatment system (Fig. 3), all steps from sample pretreatment with the SPME Arrow to sample analysis can be performed automatically.

Fig. 3 AOC™-6000 Plus + GCMS-QP2020 NX

■ Beer Analysis Results Using the SPME Arrow

Seven types of commercially sold beers were used as samples. 8 g of beer and 3 g of NaCl were sealed in each vial and measured. Analytical conditions are indicated in Table 1. Compounds were identified based on the retention time information, ion information, and mass spectrum information registered for each compound in the Smart Aroma Database. As a result, 204 aroma compounds were identified. Results from principal component analysis of the detected compounds are shown in Fig. 4. This enabled classification of each beer based on score plot results. In combination with the loading plot, the results showed which relatively high-concentration compounds each beer contained.

The relatively high-concentration compounds and the aroma characteristics of barrel-aged and IPA beers are indicated in Table 2. The results show that the barrel-aged beer contains higher concentrations of rich sweet aroma compounds, such as honey, vanilla, and coconut, whereas the IPA has higher concentrations of herb and grass aroma compounds. FASST analysis could also be effective, by only monitoring the important compounds identified above with SIM measurements and monitoring all other compounds with scan measurements. For an example of using FASST analysis to analyze aroma compounds, refer to Application News 01-00317.

Table 1 Analytical Conditions

Model:	GCMS-QP2020 NX	[GC conditions]	
Autosampler:	AOC-6000 Plus	Injection Mode:	Splitless
[SPME Arrow conditions]		Carrier Gas:	He
SPME Arrow:	DVB/Carbon WR/PDMS (O.D.: 1.1 m, Film thickness: 120 µm, length: 20 mm)	Carrier Gas Control:	Linear Velocity (25.5 cm/s)
		Column:	SH-PolarWax (60 m × 0.25 mm I.D., 0.25 µm)
Conditioning Temp.:	270 °C	Column Temp.:	40 °C (5 min) – 3 °C/min – 250 °C (15 min)
Pre Conditioning Time:	10 min	[MS conditions]	
Incubation Temp.:	60 °C	Ion Source Temp.:	200 °C
Stirrer Speed:	250 rpm	Interface Temp.:	250 °C
Sample Extract Time:	30 min	Acquisition Mode:	Scan
Sample Desorb Time:	1 min (250 °C: GC injection temperature)	Event Time:	0.3 sec
Post Conditioning Time:	5 min	*m/z* Range:	*m/z* 45-400

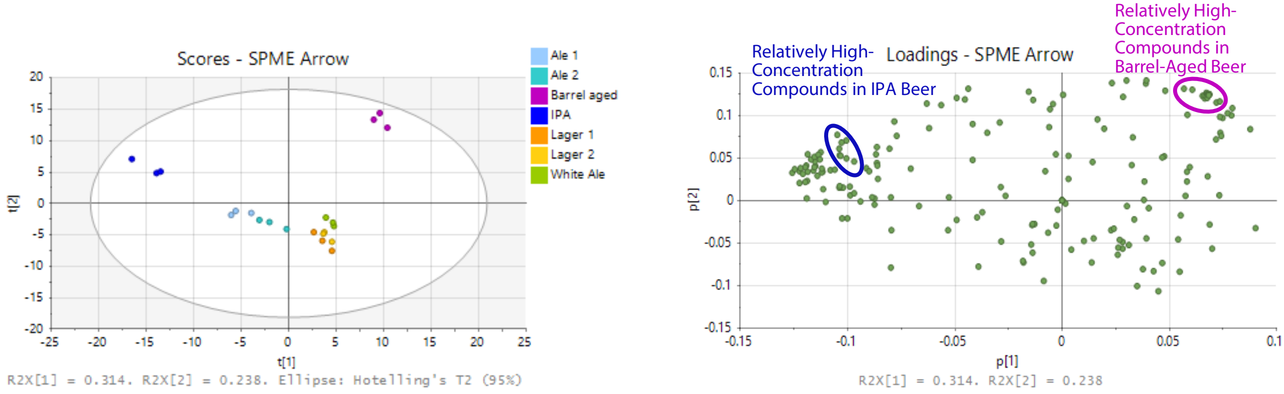


Fig. 4 Score Plot (Left) and Loading Plot (Right) of Results Measured with SPME Arrow

Table 2 Relatively High-Concentration Compounds in Each Beer

Barrel aged		IPA	
Compound	Aroma Characteristics	Compound	Aroma Characteristics
Ethyl lactate	Fruit	3-Methyl-2-buten-1-ol	Herb
4-Ethyl-2-methoxyphenol	Spice, clove	1-Hexanol	Resin, flower, green
3-Ethylphenol	Must	trans-Rose Oxide	Flower
Diethyl succinate	Wine, fruit	3-Ethoxy-1-propanol	Fruit
Benzyl alcohol	Sweet, flower	cis-3-Hexen-1-ol	Grass
Eugenol	Clove, honey	Geranyl acetate	Rose
(E)-Whiskey lactone	Flower, lactone	Methyl salicylate	Peppermint
(Z)-whiskey lactone	Coconut	Ethyl salicylate	Wintergreen, mint
gamma-Decalactone	Peach, fat		
Ethyl vanillate	Flower, fruit, sweet, Vanilla		
Benzaldehyde	Almond, burnt sugar		

■ Conclusion

This article describes R&D analysis of beer aroma compounds using an SPME Arrow, which enables pretreating samples to highly concentrate aroma compounds, in combination with the Smart Aroma Database, which enables efficient detection of aroma compounds. This resulted in identifying 204 aroma compounds. Then a principal component analysis of those results confirmed the characteristic aroma compounds of each beer.

Whereas Application News 01-00317 assumes analysis is being performed for quality control purposes, this Application News bulletin assumes aroma compounds are being analyzed for R&D purposes. Thus, the Smart Aroma Database can be used for a wide range of applications from R&D to quality control. Using the database for a wide-scope targeted analysis eliminates the need for time-consuming reconsideration of analytical conditions and checking data. Consequently, it can simplify acquisition and data analysis.

Acknowledgments
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