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Efficient Comprehensive Analysis of Beer Aroma by SPME Arrow-GC/MS and Smart Aroma Database™

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Yui Higashi[1]; Emiko Shimbo[1]; Yusuke Takemori[1]; Kuhn Eberhardt[2]; Shiro Yamada[3], Takuma Yamamiya[4]; Narihiro Suzuki, Ph.D.[4]
[1] Shimadzu Corporation, Kyoto, Japan, [2] Shimadzu Scientific Instruments, Columbia, MD, [3] Far Yeast Brewing Company, Yamanashi, Japan, [4] Ise Kadoya Brewery, Ise, Japan

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1. 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. A wide-scope target analyses using the special database was done for the comprehensive analysis of aroma compounds. A wide-scope target analysis is based on the database that contains pre-registered compounds' characteristics (e. g., retention time, target ion, mass spectrum, odor quality) and achieves both efficiency and accuracy. Acquired data were processed and analyzed by multivariate data analyses in order to calibrate and visualize beer qualifications.

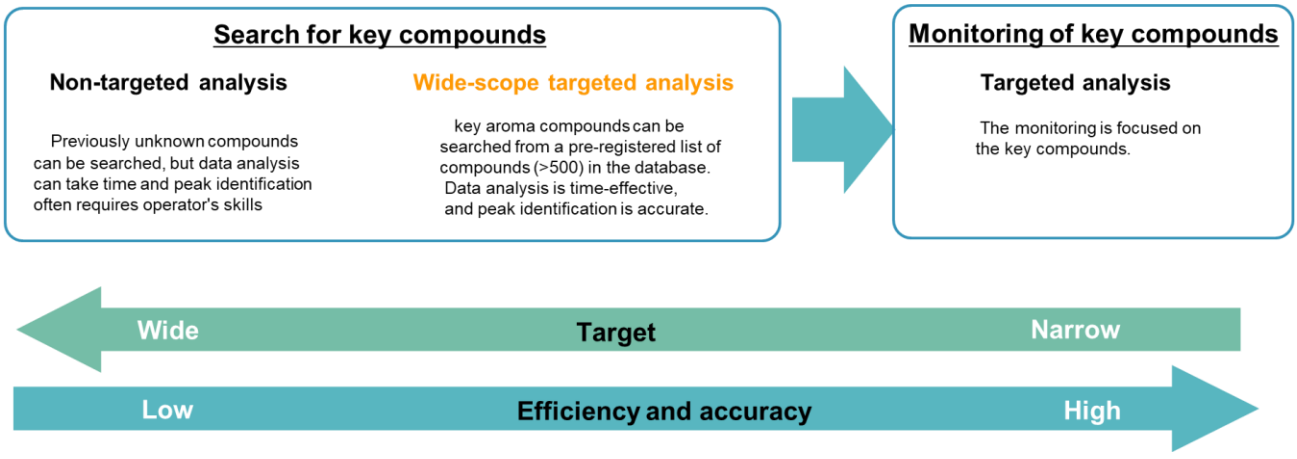

Figure 1 Wide-Scope Targeted Analysis

2. Special Database

In this study, we used GC-MS with Special Database for aroma in order to evaluate beer samples. The Smart Aroma Database was used for comprehensive analysis of aroma components. This database can be used to easily create methods without having to consider analytical conditions, by following the steps indicated in Figure 2.

The Smart Aroma Database includes analytical condition settings and sensory information for over 500 important compounds that contribute to aroma. 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.

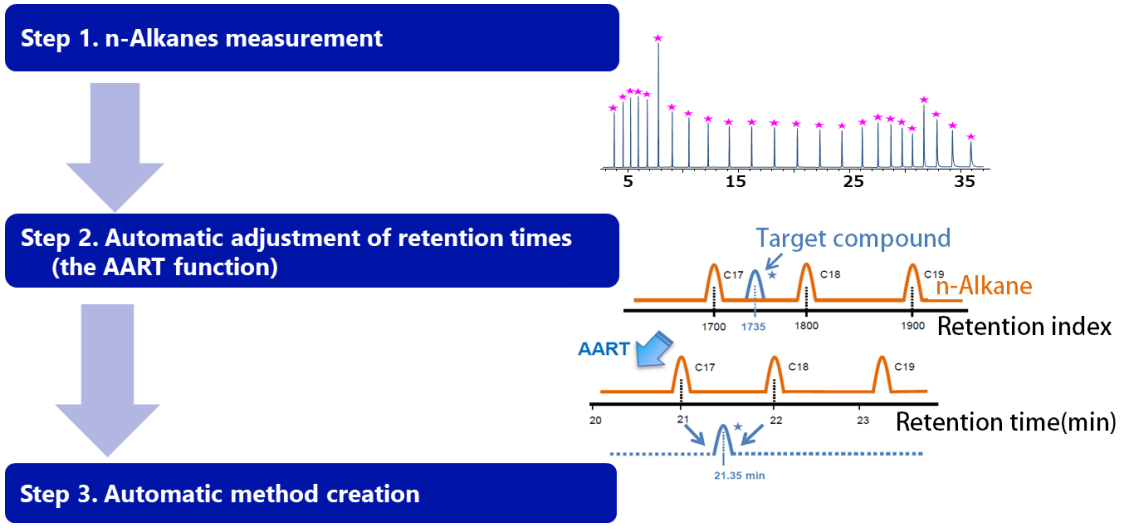

Figure 2 Analysis Process Flow Using Smart Aroma Database

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

Figure 3 Information Registered in Smart Aroma Database

3. Methods

In this study, in order to evaluate the difference of brands, 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.

Solid Phase Microextraction (SPME) Arrow was used at the pre-concentration. 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. Table 1 shows Analytical Conditions.

Table1 Analytical condition for aroma analysis

GC-MS : GCMS-QP 2020 NX
Autosampler : AOC™-6000 Plus
Software : Smart Aroma Database

SPME Arrow conditions
SPME Arrow:DVB/Carbon WR/PDMS (O.D.: 1.1 m, Film thickness: 120 µm,length: 20 mm)
Conditioning Temp.:270 °C
Pre Conditioning Time:10 min
Incubation Temp.:60 °C
Stirrer Speed:250 rpm
Sample Extract Time:30 min
Sample Desorb Time:1 min (250 °C: GC injection temperature)
Post Conditioning Time:5 min

GC conditions
Column: InertCap Pure-wax (30 m × 0.25 mm I.D. 0.25 µm)
Carrier Gas: He
Control Mode : Pressure (83.5 kPa)
Injection Method : Split (1:5)
Oven Temperature : 40°C (5 min) - 10 °C/min - 250 °C (10 min)

MS conditions
Ion Source Temperature : 200 °C
Interface Temperature : 250 °C
Acquisition mode : Scan
Event time : 0.3 sec
m/z range : m/z=35-400 (Scan)

Figure 4 AOC™-6000 Plus + GCMS-QP2020 NX

4. Results

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. Acquired data were processed and analyzed by Principal Component Analysis (PCA) in order to calibrate and visualize beer qualifications. Results from principal component analysis(PCA) of the detected compounds are shown in Figure 5 and Figure 6. 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.

SIMCA 17 software (INFOCOM CORPORATION) was used for PCA.

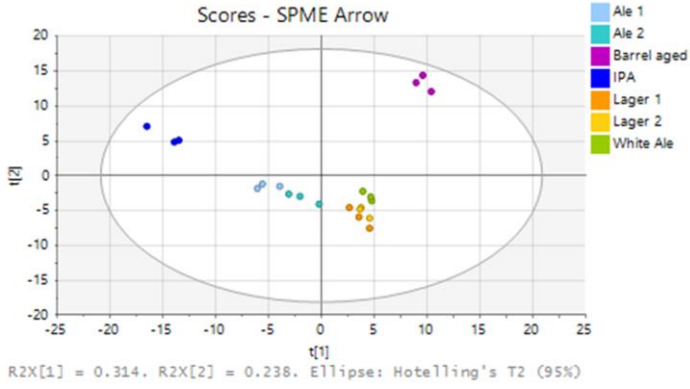

Figure 5 Score Plot

Figure 6 Loading Plot

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		

5. Conclusions

- 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.
- Using The Smart Aroma 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.

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Shimadzu Corporation
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