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Application News

Gas Chromatograph

Simple Aroma Component Analysis Using Nexis GC-2060 with a Multi-Mode Inlet (MMI)

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User Benefits

- By installing an MMI in a Nexis GC-2060 system, a wide variety of injection methods can be used .
- By using a MonoTrap silica monolith sorbent to collect aroma components, highly sensitive analysis can be achieved without cumbersome extraction procedures.
- With the MMI thermal desorption/extraction mode, aroma components can be analyzed without dedicated sample-preparation equipment.

■ Introduction

Given that aroma is an important attribute of foods, gas chromatography (GC) and gas chromatography–mass spectrometry (GC-MS) are often used for qualitative and quantitative analysis of aroma components. Aroma components are typically collected from foods using headspace sampling, solid-phase microextraction (SPME), or liquid extraction methods, but for this article, a MonoTrap silica monolith sorbent was used to collect aroma components from alcoholic beverages. With the MonoTrap, aroma components can be collected and concentrated without cumbersome extraction procedures, enabling highly sensitive analysis.

In this case, aroma components were analyzed using a Nexis GC-2060 gas chromatograph system equipped with a Multi-Mode Inlet (MMI). The MMI is a newly developed inlet that supports a wide range of injection methods. This article describes using the thermal desorption/extraction mode, one of the methods available with the MMI, to desorb the aroma components collected on the MonoTrap sorbent and inject them into the GC unit. That enabled highly sensitive aroma analysis while reducing analysis costs, without using dedicated sample-preparation equipment.

Fig. 1 (Left) Exterior View of the Nexis™ GC-2060
(Right) Exterior View of the MMI

■ Overview of the Multi-Mode Inlet (MMI)

The Nexis GC-2060 can be equipped with a Multi-Mode Inlet (MMI) (Fig. 1 (right)), a new type of inlet . The MMI is a newly developed inlet that allows many injection methods to be used with a single inlet—not only the commonly used split/splitless injection methods, but also programmed temperature vaporizing (PTV) injection, direct injection, large volume injection, and thermal desorption/extraction methods . It supports a wide range of applications across many fields, including analyzing pesticide residues in foods using large-volume injection and aroma analysis using thermal desorption.

Because the MMI enables rapid temperature ramping at rates up to 1200 °C/min, it can suppress peak broadening. In addition, by selecting optional cooling with compressed air or liquid nitrogen, analysis cycle times can be shortened with fast cooling. The GC-2060 also comes standard with the "Easy sTop" function, which allows inlet maintenance to be performed immediately with simple steps, enabling smooth maintenance operations, such as insert replacement, even after cooling.

■ Aroma Component Analysis Using an MMI and MonoTrap

The MonoTrap silica monolith sorbent enables highly efficient compound collection, due to its large surface area that provides a high sample loading capacity. In this article, a MonoTrap RGPS TD (GL Sciences) sorbent was used for its comprehensive collection performance. An example of a MonoTrap analysis workflow using the MMI thermal desorption/extraction mode is shown below.

[1] Collect aroma components on the MonoTrap
Collect volatile components by placing the MonoTrap in an MT h older (GL Sciences) and suspending the holder in the headspace of a vial.

[2] Load the MonoTrap into the MMI
Use the GC-2060 Easy sTop function to stop the carrier gas, remove the insert from the MMI, and load the MonoTrap into the insert . No tools are required to open the MMI.

[3] Analyze components
Return the insert containing the MonoTrap back into the GC-2060 system. Once the Easy sTop function is finished, start the analysis.

[4] Perform the next analysis
To continue with subsequent analyses, wait for the MMI temperature to decrease and then use the Easy sTop function again to replace the MonoTrap sorbent.

In this example, compressed air was used to cool the MMI. After each analysis was finished, about 10 minutes was required for cooling the MMI to a temperature that enables the next analysis. That resulted in rapid cooling and analysis with a short cycle time. Note that cooling times can vary depending on analysis conditions and other factors.

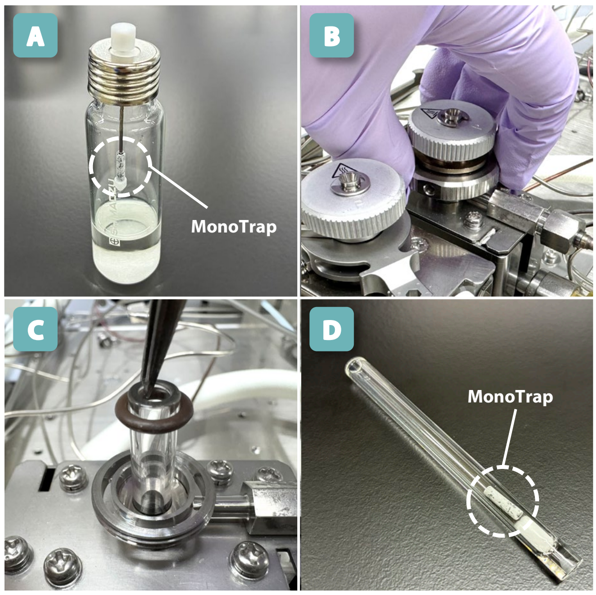

Fig. 2 (A) Aroma Collection on MonoTrap, (B) Tool-Free Opening of the MMI, (C) Insert Removal, and (D) MonoTrap in the Insert.

Analysis Conditions

Two types of sake samples (regular sake and daiginjo sake) and three types of beer samples (pilsner, saison, and IPA*) were prepared. For each type of alcoholic beverage, 5 g of sample and 3 g of sodium chloride were placed into a 20 mL vial, 50 µL of a 1 % (v/v) 1-pentanol solution was added as an internal standard (IS), and the vial was thoroughly mixed. Next, MonoTrap RGPS TD sorbent (GL Sciences) was suspended, as shown in Fig. 2A, and the vial was promptly kept warm to collect aroma components. The incubation temperature during collection was set to 40 °C and the collection time was set to 30 minutes. Immediately after collection, the MonoTrap was placed into an insert (P/N: 227-36701-01), as shown in Fig. 2D, and analyzed using the MMI. Hydrogen was used as the carrier gas to reduce analysis costs. The analysis conditions are shown in Table 1.

* For the IPA sample, the "Kōchō" aroma profile jointly developed by Shimadzu Corporation and ISEKADO was used.

Table 1 Analysis Conditions

Model:	Nexis GC-2060 + MMI	
MMI Mode:	Thermal desorption/extraction	
Purge Program:	Time	5 min
	Temperature	35 °C
	Split ratio	1:20
Desorb Program:	Time	5 min
	Temperature	250 °C
	Heating rate	1000 °C/min
	Split ratio	1:20
Carrier Gas:	H_2	
Line Velocity:	60 cm/s	
Septum Purge Flowrate:	3 mL/min	
Column:	SH-PolarWax (30 m, 0.25 mm I.D., 0.50 µm) (P/N 227-36248-01)	
Column Temp.:	40 °C (10 min) – 10 °C/min – 250 °C (3 min)	
Detector:	FID	
Detector Temp.:	250 °C	
Detector Gas Flowrate:	Make up	30 mL/min (N_2)
	H_2	40 mL/min
	Air	170 mL/min

Results 1 – Comparison of Aroma Components in Sake

The aroma components in the two types of sake (regular sake and daiginjo sake) were compared. Fig. 3 shows chromatograms obtained from the analysis of a MonoTrap sample with no collection (blank) and MonoTrap samples used to collect aroma components from each sake. Table 2 shows peak area ratios relative to the IS for representative aroma components.

In general, it can be said that sake made with a higher rice polishing ratio tends to have a stronger fruity aroma, known as "ginjo-ka" (ginjo aroma). In this analysis, daiginjo sake showed larger area ratios for esters, including ethyl caproate (apple-like aroma) and isoamyl acetate (banana-like aroma), which are representative ginjo aroma components. Fig. 4 shows an overlay of chromatograms for these representative ginjo aroma components. In particular, a marked difference in peak area was observed for ethyl caproate. These results can be considered to reflect aroma differences attributable to differences in the rice polishing ratio.

Table 2 Area Ratios of Aroma Components in Sake (IS: 1-Pentanol)

No.	Components	Regular sake	Daiginjo sake
1	Isobutyl alcohol	0.067	0.057
2	Isoamyl acetate	0.164	0.287
3	Isoamyl alcohol	0.952	1.090
4	Ethyl caproate	0.013	2.018
5	Ethyl caprylate	0.001	0.132
6	2-Phenylethyl acetate	0.003	0.050
7	2-Phenylethyl alcohol	0.156	0.299

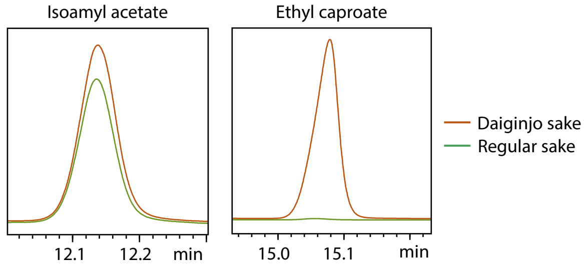

Fig. 4 Overlaid Chromatograms of Isoamyl Acetate and Ethyl Caproate

Results 2 – Comparison of Aroma Components in Beer

Next, the aroma components among the three different beer styles (pilsner, saison, and IPA) were compared. Fig. 5 shows chromatograms obtained from analyzing the blank and MonoTrap samples used to collect aroma components from each beer. Table 3 shows area ratios relative to the IS for representative aroma components.

In general, when comparing "lager" produced by bottom fermentation with "ale" produced by top fermentation, ales are considered to produce more esters due to the activity of yeast. In this example, the results confirmed that saison and IPA beers (types of ales) had larger area ratios for ethyl caproate than the pilsner (a type of lager). In contrast, for acetate esters such as isoamyl acetate and β-phenethyl acetate , the pilsner showed larger area ratios. In addition, differences in aroma components among beer styles were observed. For example, the peak area ratio of ethyl caprylate in the saison was detected as approximately 250 % higher than that in the IPA, which is also an ale.

Furthermore, peaks that are barely visible in the pilsner chromatogram (#8, #9, and #10) can be observed in saison and IPA chromatograms . Analysis using TD-GC-MS identified these peaks as myrcene, linalool, and geraniol, respectively. Fig. 6 shows an overlay of chromatograms for those aroma components. All of the components are representative hop-derived terpenes and are known as aroma components that impart bright fruity and floral notes to beer. These results are consistent with the fact that the saison and IPA used in this experiment are both beers with sweet, fragrant, citrus-like aromas.

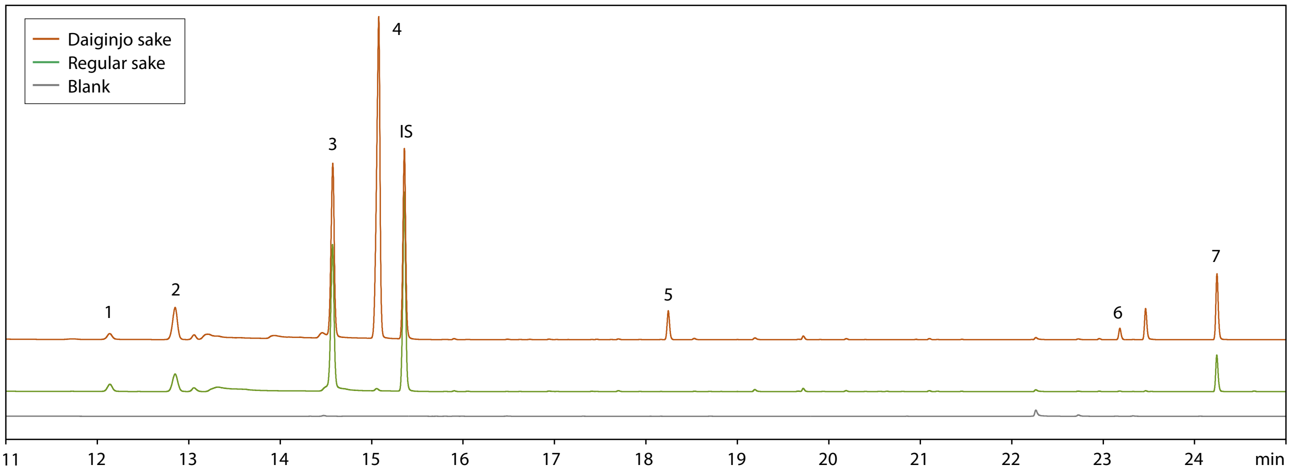

Fig. 3 Chromatograms Obtained from the Analysis of Each Sake

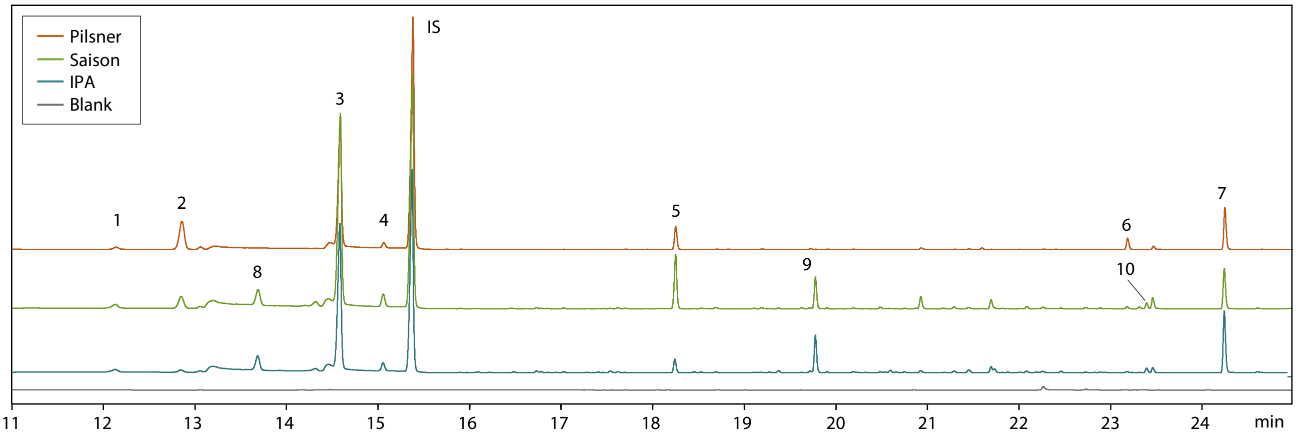

Fig. 5 Chromatograms Obtained from the Analysis of Each Beer Sample

In addition, Fig. 7 shows expanded chromatograms in the mid- to high-boiling range. Many more peaks were observed in the saison and IPA chromatograms than in the pilsner chromatogram, experimentally confirming the differences between the pilsner, characterized by a dry aroma, and the saison and IPA, characterized by rich aroma profiles. In addition, in the IPA chromatogram, the peak area ratio of 2-phenylethyl alcohol (rose-like aroma) was approximately 80 % higher than for the saison, which is also an ale (Fig. 6), demonstrating that characteristic aroma components unique to each beer could also be identified.

Table 3 Area Ratios of Aroma Components in Beer (IS: 1-Pentanol)

No.	Components	Pilsner	Saison	IPA
1	Isobutyl alcohol	0.015	0.031	0.027
2	Isoamyl acetate	0.189	0.080	0.018
3	Isoamyl alcohol	0.623	0.958	0.848
4	Ethyl caproate	0.022	0.054	0.040
5	Ethyl caprylate	0.077	0.180	0.052
6	2-Phenylethyl acetate	0.037	0.007	0.005
7	2-Phenylethyl alcohol	0.132	0.129	0.231
8	Myrcene	0.000	0.086	0.089
9	Linalool	0.001	0.099	0.139
10	Geraniol	0.000	0.018	0.017

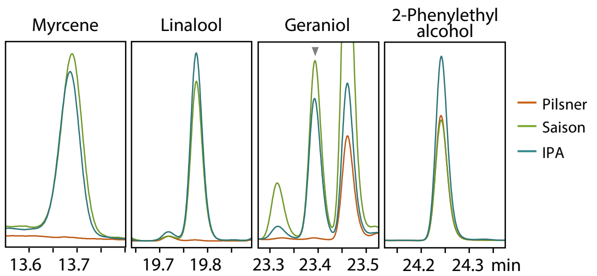

Fig. 6 Overlays Chromatograms of Characteristic Aroma Components

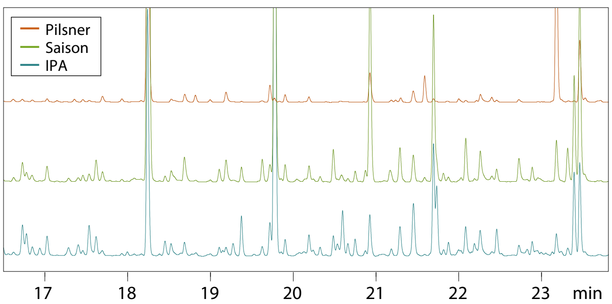

Fig. 7 Chromatograms Obtained from Beer Aroma Analysis (Expanded View)

Results 3 – Comparison of Aroma Components in the Same Beer Type Differing Only in Hop Contact Time

Finally, aroma components were compared in two otherwise identical saison beers that differed only in hop contact time ("saison 24 h" vs. "saison 48 h"). Table 4 shows area ratios relative to the IS for representative aroma components.

For higher alcohols, no major differences were observed due to hop contact time. In contrast, esters and terpenes showed higher area ratios in the saison 48 h chromatogram than in the saison 24 h chromatogram, where terpenes in particular increased by about 30 %. These results confirm that hop contact time has a pronounced impact on hop-derived aroma.

Using this simple aroma analysis approach using MonoTrap sorbent and an MMI, enabled evaluation of how differences in the production process affect aroma components in the same beer type.

Table 4 Area Ratios of Aroma Components in Various Saison Samples (IS: 1-Pentanol)

No.	Components	Saison 24 h	Saison 48 h
1	Isobutyl alcohol	0.031	0.031
2	Isoamyl acetate	0.070	0.080
3	Isoamyl alcohol	0.964	0.958
4	Ethyl caproate	0.046	0.054
5	Ethyl caprylate	0.122	0.180
6	2-Phenylethyl acetate	0.007	0.007
7	2-Phenylethyl alcohol	0.120	0.129
8	Myrcene	0.065	0.086
9	Linalool	0.074	0.099
10	Geraniol	0.014	0.018

Conclusion

By analyzing aroma components using the Nexis GC-2060 in combination with a Multi-Mode Inlet (MMI) and a MonoTrap silica monolith sorbent, aroma characteristics of respective alcoholic beverages could be analyzed and evaluated easily without dedicated sample-preparation equipment and without performing cumbersome extraction procedures.

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