

Application News

Gas Chromatograph Nexis™ GC-2030
Headspace Sampler HS-20 NX

HS-GC Analysis of Sake Aroma Compounds Using Hydrogen Carrier Gas

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User Benefits

- ◆ Aroma compounds in alcoholic beverages can be quantified easily and cost-effectively.
- ◆ The HS-20 NX headspace sampler enables simple, high-sensitivity analysis of aroma compounds.
- ◆ Using hydrogen as the carrier gas reduces costs while maintaining separation performance.

■ Introduction

Aroma is a crucial component of sake. Therefore, during the sake production process, it is essential to preserve the sake's aroma profile. One key step in sake production is pasteurization ("hiire"), in which unpasteurized sake is heated to sterilize it, deactivate enzymes, and halt fermentation. This pasteurization step is known to impair the aroma of sake if it is carried out for too long or at too high a temperature. In this analysis, a Nexis GC-2030 gas chromatograph was combined with an HS-20 NX headspace sampler (Fig. 1) to quantify individual aroma compounds in sake before and after pasteurization.

Headspace gas chromatography (HS-GC) is an effective method for analyzing volatile compounds, including aromas. HS-GC requires no pretreatment (e.g., derivatization), and sample preparation is straightforward: simply place the sample in a vial and perform the analysis.

Hydrogen, which is inexpensive and readily available, was used as the carrier gas. Using H₂ gas as the carrier reduces analysis costs while maintaining high separation performance over a wide range of linear velocities. However, because hydrogen is flammable, it must be handled with care. The Nexis GC-2030, Brevis™ GC-2050, and Nexis GC-2060 gas chromatographs can be equipped with an optional hydrogen sensor that measures H₂ concentration in the column oven. If the hydrogen sensor detects a leak, the system shifts to standby mode or automatically powers off, helping prevent accidents before they occur.

Table 1 Analysis Conditions

HS-20 NX	
Mode:	Loop
Oven Temp.:	40 °C
Sample Line Temp.:	90 °C
Transfer Line Temp.:	100 °C
Vial Pressure:	60 kPa (N ₂)
Vial Heat-Retention Time:	45 min
Vial Press. Time:	1 min
Equilibration Time:	0.1 min
Loading Time:	0.5 min
Loading Press. Time:	0.1 min
Injection Time:	0.5 min
Needle Flush Time:	5 min
Nexis GC-2030	
Injection Mode:	Split (1:10)
Carrier Gas:	H ₂
Carrier Gas Control:	Linear velocity (60 cm/s)
Column:	SH-PolarWax (30 m, 0.25 mm ID, df 0.50 μm) (P/N : 227-36248-01)
Column Temp.:	40 °C (3 min) — 5 °C/min — 55 °C — 15 °C/min — 190 °C (3 min)
Detector:	FID
Detector Temp.:	220 °C
Detector Gas:	Make up (N ₂) 24 mL/min H ₂ 32 mL/min Air 200 mL/min



Fig. 1 Nexis™ GC-2030 and HS-20 NX

■ Samples and Analytical Conditions

For analysis, 5 mL of either a standard sample or an unknown sample was placed in a 20 mL headspace vial, and an internal standard solution was added to give a concentration of 50 ppm (v/v). The standard samples used for calibration curve preparation were prepared by diluting a mixture of nine aroma compounds in 16 % aqueous ethanol. Three calibration points were used. n-Butanol was used as the internal standard. The unknown samples were sampled before and after pasteurization (alcohol content: 16 %). The analytical conditions are shown in Table 1.

■ Results

1. Standard Samples

Fig. 2 shows the calibration curves for the standard samples, along with their R² values. All calibration curves showed good linearity.

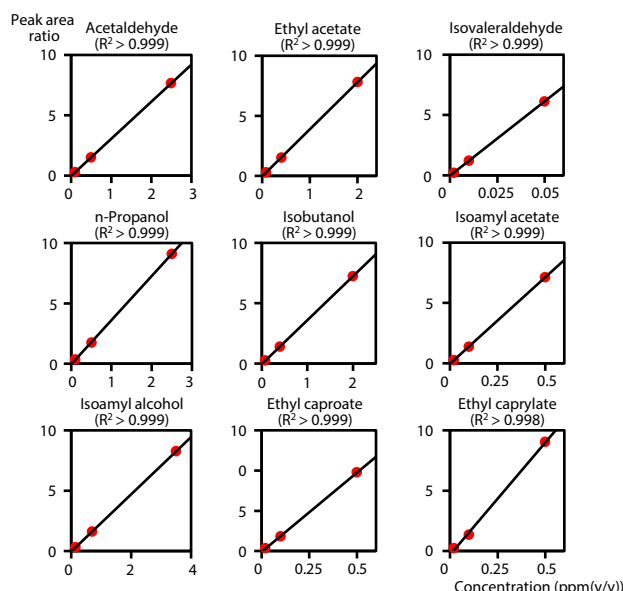


Fig. 2 Calibration Curves for Each Aroma Compound

2. Unknown Samples

Fig. 3 shows the chromatograms obtained from the analysis of sake before and after pasteurization. Table 2 shows the average quantified values for each aroma compound (N = 5), calculated using the prepared calibration curves.

By calculating the quantified values for each aroma compound, it was possible to quantitatively evaluate differences in aroma before and after pasteurization. The results showed that there were no substantial differences in the concentrations of the aroma compounds examined in this study.

The sake used in this analysis was pasteurized using a method known as “bin-kan hiire” (bottle pasteurization). Bin-kan hiire is a pasteurization method in which unpasteurized sake is gradually heated after being filled into bottles, and compared with the conventional pasteurization method using a heat exchanger, it can better suppress the loss of aroma compounds. In this analysis, no substantial differences were observed in the concentrations of aroma compounds before and after pasteurization, suggesting that bottle pasteurization enabled sterilization and enzyme deactivation without impairing aroma.

On the other hand, isovaleraldehyde, produced by enzymatic degradation of isoamyl alcohol, was detected only before pasteurization (Fig. 4). The isovaleraldehyde detected in the pre-pasteurization sample is thought to have formed during storage after bottling. Because it was not detected in the post-pasteurization sample, it was confirmed that pasteurization had properly deactivated the enzymes.

Conclusion

By combining the Nexis GC-2030 gas chromatograph with the HS-20 NX headspace sampler, the aroma compounds can be easily quantified without pretreatment such as derivatization. In addition, using inexpensive, readily available H₂ as the carrier gas reduces analysis costs while maintaining high separation performance. Because the Nexis GC-2030 can be equipped with an optional hydrogen sensor, analyses using H₂ carrier gas can be performed safely.

Acknowledgements

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Related Applications

1. Analysis of Aroma Components in Beer Using HS-20 and Nexis™ GC-2030, [Application News No. G317](#)
2. Analysis of Diacetyl and 2,3-Pentanedione in Beer Using Brevis GC-2050, [Application News 01-00652](#)
3. High-Sensitivity Analysis of Fragrance Components by Trap Mode of HS-20 Trap Headspace Sampler, [Application News 01-00138A](#)

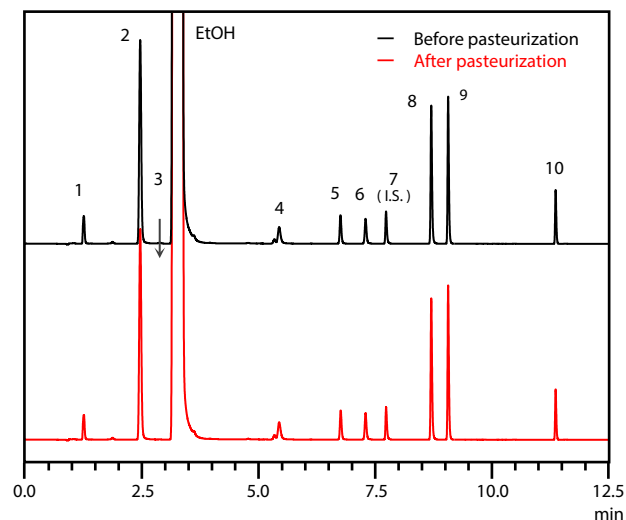


Fig. 3 Chromatograms of Sake Before and After Pasteurization

Table 2 Quantitative Values for Each Aroma Compound (Unit: ppm (v/v))

No.	Compounds	Before pasteurization	After pasteurization
1	Acetaldehyde	16.59	14.41
2	Ethyl acetate	59.46	58.94
3	Isovaleraldehyde	0.19	N.D.
4	n-Propanol	67.03	68.44
5	Isobutanol	35.45	35.43
6	Isoamyl acetate	1.79	1.80
7	n-Butanol (Internal standard)	-	-
8	Isoamyl alcohol	148.84	149.02
9	Ethyl caproate	5.65	5.61
10	Ethyl caprylate	1.81	1.70

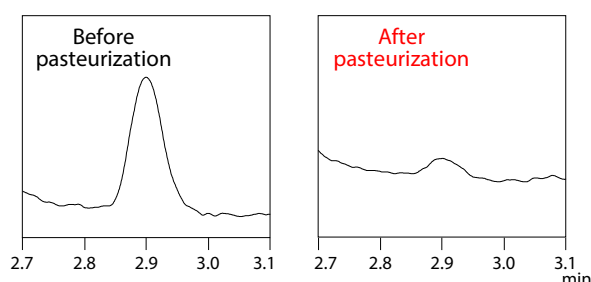


Fig. 4 Isovaleraldehyde Peaks

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