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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 $H_2$ 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 $H_2$ 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.

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.

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<br>$H_2$ 32 mL/min Air 200 mL/min |

## ■ Results

### 1. Standard Samples

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

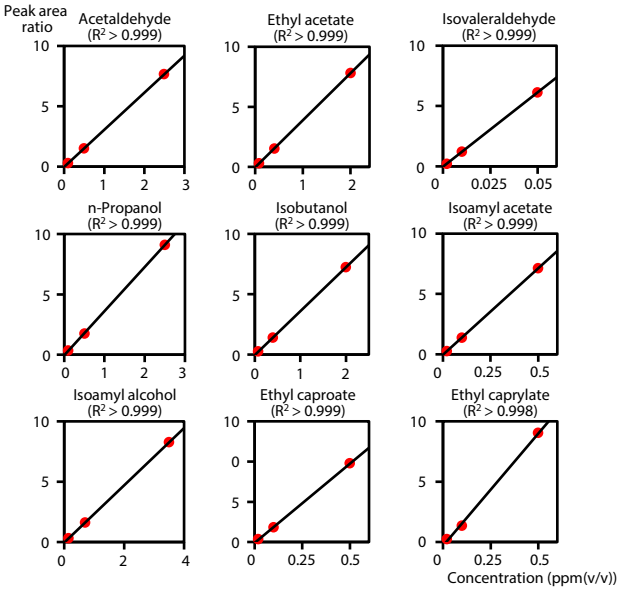


Fig. 2 Calibration Curves for Each Aroma Compound

## 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.

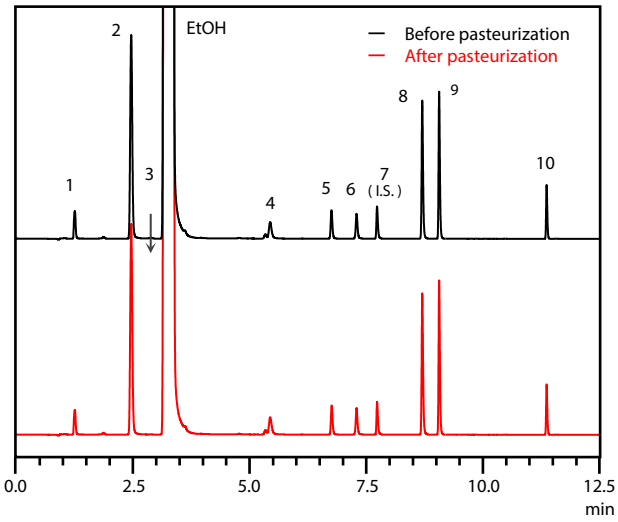


Fig. 3 Chromatograms of Sake Before and After Pasteurization

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 |

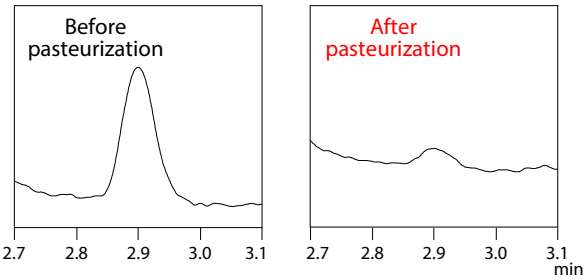


Fig. 4 Isovaleraldehyde Peaks

## ■ 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 $H_2$ 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 $H_2$ carrier gas can be performed safely.

Acknowledgements

We would like to express our sincere gratitude to Dassai Inc. for its extensive support in preparing this application note, including providing samples and assisting with interpretation of the results.

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



01-00886-EN First Edition: Apr 2026

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