Application News

GC Nexis™ GC-2030/SCD-2030/HS-20 NX

Highly Sensitive Analysis of Sulfur Compounds in Beer Using the Trap Mode of a Headspace Sampler

Yuto Nakasuji, Akara Hashimoto

User Benefits

- Using the Trap mode of HS-20 NX allows for a simple and highly sensitive analysis of concentrated headspace gas.
- By utilizing the Trap mode and SCD-2030, it becomes possible to detect trace sulfur compounds that were difficult to analyze in the Loop mode.
- This system is compatible with N2 carrier gas, allowing analysis to be conducted without the use of helium.

■ Introduction

Volatile sulfur compounds, which are generated during the fermentation process of beer yeast, significantly contribute to the flavor and quality of beer. However, as these compounds exist in very small amounts, their detection requires pre-concentration techniques or the use of high-sensitivity detectors.

In a previous Application News titled "Analysis of Volatile Sulfur Compounds in Beer Using Nexis™ SCD-2030", we introduced an analysis method combining SCD (Sulfur Chemiluminescence Detector) and the Loop mode of the headspace sampler for the analysis of volatile sulfur compounds in beer.

In this Application News, we will present the results of further investigation into achieving higher sensitivity using the Trap mode of the headspace sampler.

Fig. 1 HS-20 NX + Nexis™ GC-2030 + SCD-2030

■ Samples and Analytical Conditions

We used two test-brewed beers as samples, differing only in the yeast (Yeast A and Yeast B).

In the procedure, 3 g of NaCl was measured and added into a vial, then 3 g of the sample was measured, added into the same vial, and sealed. The analysis was conducted by concentrating the headspace gas in the vial through five multi-injections using the Trap mode of HS-20 NX, and injection into the GC. The detailed analytical conditions are shown in Table 1.

■ Concentrate Headspace Gases in a Trap

The HS-20 NX can operate in loop mode or trap mode. These two modes use different methods for sampling the headspace gas. In the Loop mode, the headspace gas is collected in a measuring tube and injected into the GC. On the other hand, in the Trap mode, the headspace gas is collected in a trap tube, followed by thermal desorption and injection into the GC. The Trap mode enables high sensitivity by multiple injections from the same vial to concentrate the gas in the trap tube. The flow of analysis by the Trap mode is shown in Fig. 2.

With the HS-20 NX, users can easily switch between Loop mode and Trap mode in the software, allowing for flexible usage depending on the sample and concentration.

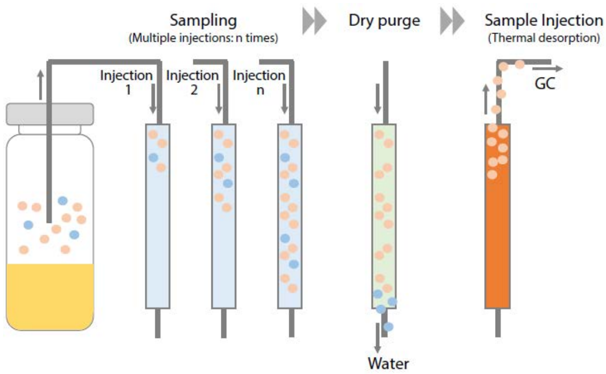

Fig. 2 Headspace Sampling in Trap Mode

Table 1 System Configuration and Analytical Conditions

System	HS-20 NX Nexis GC-2030/SCD-2030		
HS		**GC**	
Mode:	Trap (Tenax TA)	Injection Mode:	Split
Oven Temp.:	80 °C	Split Ratio:	5
Sample Line Temp.:	100 °C	Carrier Gas:	N_2
Transfer Line Temp.:	100 °C	Carrier Gas Control:	Const. Linear velocity (45 cm/sec)
Trap Cooling Temp.:	10 °C	Column:	DB-1 (60 m × 0.32 mm I.D., 5 µm)
Trap Heating Temp.:	250 °C	Oven Program:	60 °C (3 min)_15 °C/min_240 °C (20 min)
Trap Waiting Temp.:	25 °C		
Multi Injection:	5	**SCD**	
Vial Pressure:	80 kPa	Interface Temp.:	200 °C
Dry Purge Pressure:	20 kPa	Electric Furnace Temp.:	850 °C
Vial Heating Time:	35 min	Detector Gas:	H_2 100 mL
Vial Pressurization Time:	1 min		N_2 10 mL
Pressure Equilibrating Time:	0.1 min		O_2 12 mL
Loading Time:	0.5 min		O_3 25 mL
Load Equilibrating Time:	0.1 min		
Dry Purge Time:	10 min		
Injection Time:	10 min		
Needle Flush Time:	45 min		

■ Results

Fig. 3 shows the results obtained from analyzing the beers in trap mode and loop mode. An examination of the enlarged chromatogram from 9 to 19 minutes reveals a substantial improvement in sensitivity in trap mode compared to loop mode, with multiple components that were barely detected in loop mode readily detected in trap mode.

The S/N ratios of the major peaks (peaks A to J in Fig. 3) are also shown in Table 2. The S/N results showed that trap mode improved sensitivity by a factor of 6 to 20 times compared to loop mode.

The results also showed a substantial difference in the amount of S-methylthioacetate present in the two beers (Fig. 4). S-Methylthioacetate is produced by yeast during fermentation and is known to contribute to the aroma of beer.[1)] The results of this analysis reveal that S-methylthioacetate potentially plays an important role in the difference in the aroma of the two beers.

■ Conclusion

This article examined using the HS-20 NX headspace sampler in trap mode with the SCD-2030 detection system to further improve the sensitivity of analysis of sulfur components in beer.

By analyzing in trap mode, we confirmed an improvement in sensitivity of 6 to 20 times compared to loop mode and enabled the detection of multiple compounds that were not detected in loop mode.

Analyzing two test beers brewed under identical conditions except for the brewing yeast (yeast A and yeast B) in trap mode revealed a much larger amount of S-methylthioacetate in the beer brewed with yeast A, and it identified S-methylthioacetate as the compound potentially responsible for the difference in the aroma of the two beers.

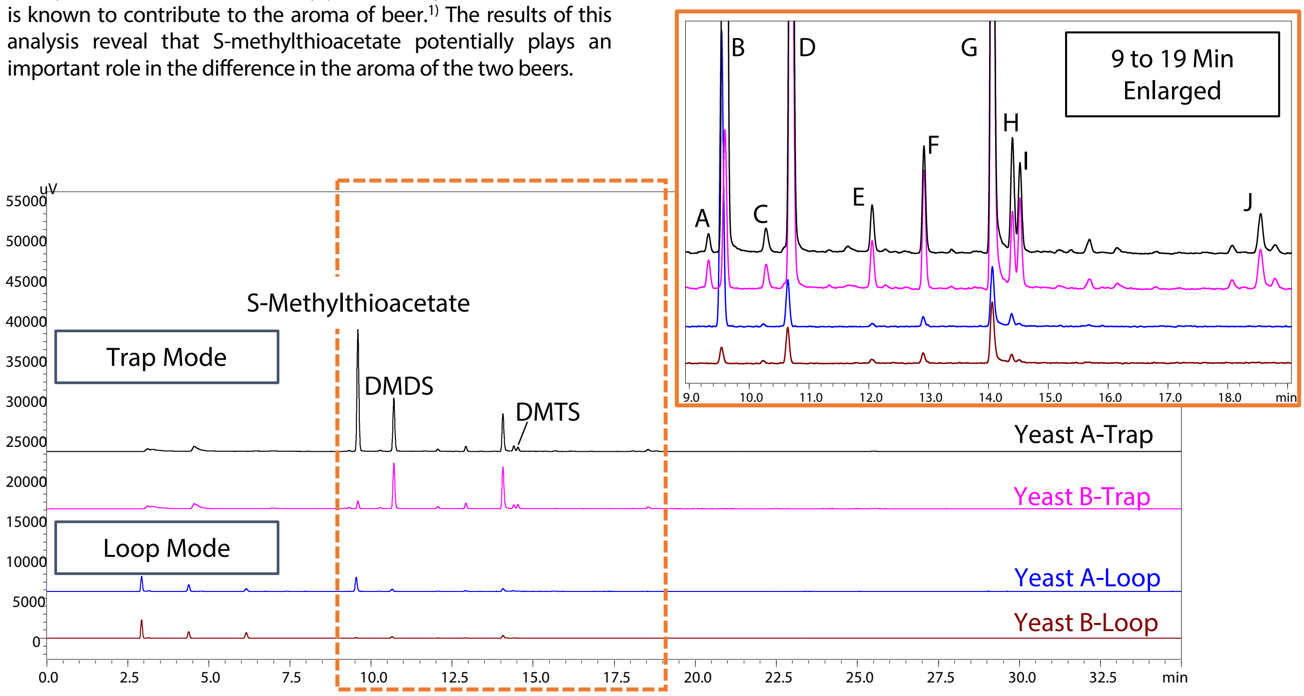

Fig. 3 Chromatograms of Trap Mode and Loop Mode Results for Each Beer

Table 2 S/N Ratio of Major Peaks (A-J) in Trap Mode and Loop Mode

Peak	Yeast A		Yeast B	
	Trap	Loop	Trap	Loop
A	13.6	N.D.	18.1	N.D.
B	1896.7	287.0	104.2	14.1
C	17.6	N.D.	15.5	N.D.
D	828.5	45.0	627.2	30.9
E	34.4	N.D.	30.7	N.D.
F	79.7	9.4	78.2	8.9
G	586.8	57.5	573.7	51.9
H	76.6	10.8	42.9	6.2
I	57.3	N.D.	52.0	N.D.
J	28.3	N.D.	24.8	N.D.

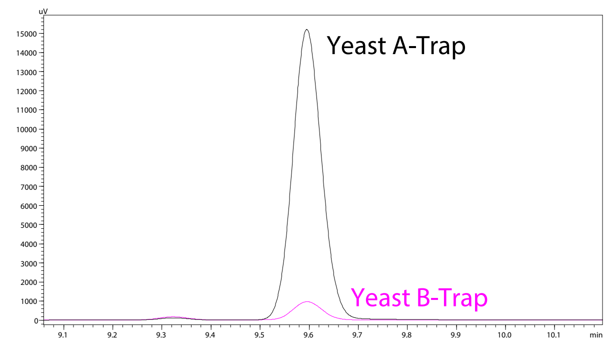

Fig. 4 Enlarged Chromatogram Showing S-Methylthioacetate

< References >
1) Identification and Determination of S-Methyl Thioacetate in Beer, Nippon Nogeikagaku Kaishi, Vol. 54, No. 9, 1980

< Acknowledgments >
We would like to thank Narihiro Suzuki (CEO), Takuma Yamamiya, and Ren Takasaki of ISEKADO, and Hironori Maruyama, principal researcher at Mie Prefecture Industrial Research Institute, for their assistance with this research.

01-00642A-EN
First Edition: Dec. 2023
Revision A: Feb. 2024

Shimadzu Corporation
www.shimadzu.com/an/

> Please fill out the survey

Related Products Some products may be updated to newer models.

> Nexis™ GC-2030
Gas Chromatograph

> Nexis™ SCD-2030
Sulfur Chemiluminescence Detection Gas Chromatogra…

> HS-20 NX series
Headspace Sampler

Related Solutions

> Food and Beverages

> Food and Nutrition

> Flavor Analysis

> Price Inquiry

> Product Inquiry

> Technical Service / Support Inquiry

> Other Inquiry