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Application News

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Gas Chromatograph GC-2030

Quantitative Analysis of Hydrogen Sulfide in Beer Using Nexis™ SCD-2030

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User Benefits

- A state-of-the-art sulfur chemiluminescence detector (SCD) enables highly sensitive analysis of sulfur components.
- Quality control with higher accuracy is realized by monitoring hydrogen sulfide in beer.
- Use of a headspace sampler eliminates the need for sample preparation in analysis of samples.

■ Introduction

When brewing beer, various volatile sulfur compounds are formed in the yeast fermentation process. Hydrogen sulfide (H_2S) is one sulfur compound that has a particularly strong effect on the flavor of beer and is one off-flavor that should be removed as far as possible, depending on the product. Thus, quantitation of hydrogen sulfide in beer is a crucial element for control of beer quality and the beer manufacturing process.

The sulfur chemiluminescence detector (SCD) is capable of detecting selectively sulfur compounds with high sensitivity. Use of an SCD in combination with the headspace (HS) method makes it possible to analyze volatile sulfur compounds in beverages. In this article, the hydrogen sulfide contents of several commercial beer products were measured and quantified by using an SCD and HS.

■ Samples and Analysis Conditions

Standard reagents of hydrogen sulfide are extremely difficult to obtain in the market. Owing to the high ionization of hydrogen sulfide in aqueous solutions, volatilization to the gas phase is poor. Therefore, carbonated water and sodium sulfide enneahydrate were used in this Application News. Hydrogen sulfide is generated by adding water-soluble sodium sulfide to carbonated water, which is weakly acidic. Standards were prepared by taking 5 g of carbonated water in a 20 mL headspace vial and adding an aqueous solution of sodium sulfide enneahydrate to obtain a S^{2-}concentration of 0.1, 0.2, 0.5, 1, 2, 5, or 10 ng/mL.

As beer samples, five types of beer were prepared, including three lager beers and two ale beers. As in the case of the standards, the samples were prepared by taking 5 g of each beer in a 20 mL headspace vial. Because the condition of beer changes quickly after the container is opened, the measurements were carried out within 1 h after the beer was enclosed in the vial. Table 1 shows the analysis conditions.

Fig. 1 Nexis™ GC-2030/SCD-2030/HS-20

Table 1 Analysis Conditions

Model	: Nexis GC-2030/SCD-2030/HS-20
<HS>	
Mode	: Loop
Oven Temperature	: 60 ˚C
Sample Line Temp.	: 120 ˚C
Transfer Line Temp.	: 150 ˚C
Vial Pressure	: 80 kPa
Vial Heat-retention Time	: 10 min
Vial Pressurization Time	: 1 min
Vial Pressurization Equil. Time	: 0.1 min
Loading Time	: 1 min
Loading Pressurization Time	: 0.1 min
Injection Time	: 1 min
Needle Flush Time	: 5 min
<GC>	
Injection Mode	: Split
Split Ratio	: 5
Carrier Gas	: He
Carrier Gas Control	: Constant Pressure (20 kPa)
Column	: SH-Rtx™-1 (60 m x 0.53 mm I.D., df = 7 µm)
Post Column	: Deactivated fused silica tubing (0.3 m x 0.32 mm I.D.)
Oven Temperature	: 30 ˚C (1.5 min), 10 ˚C/min to 50 ˚C, 25 ˚C/min to 220 ˚C (2 min)
Detector	: SCD
Interface Temp.	: 200 ˚C
Electric Furnace Temp.	: 850 ˚C
Detector Gas	: H_2 100 mL/min N_2 10 mL/min O_2 12 mL/min O_3 25 mL/min

■ Calibration Curve

Fig. 2 shows the calibration curve. Satisfactory linearity was obtained in the concentration range from 0.1 to 10 ng/mL, as the coefficient of correlation was R = 0.999 or higher.

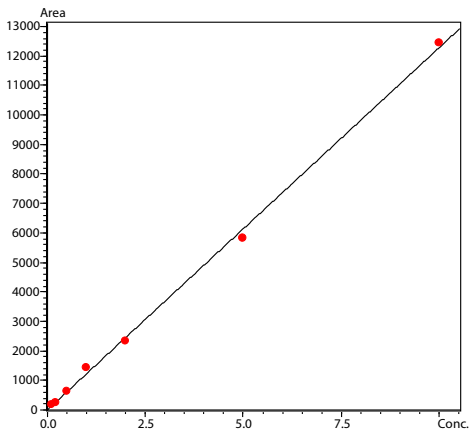

Fig. 2 Calibration Curve of Hydrogen Sulfide

■ Results of Analysis of Beer Samples

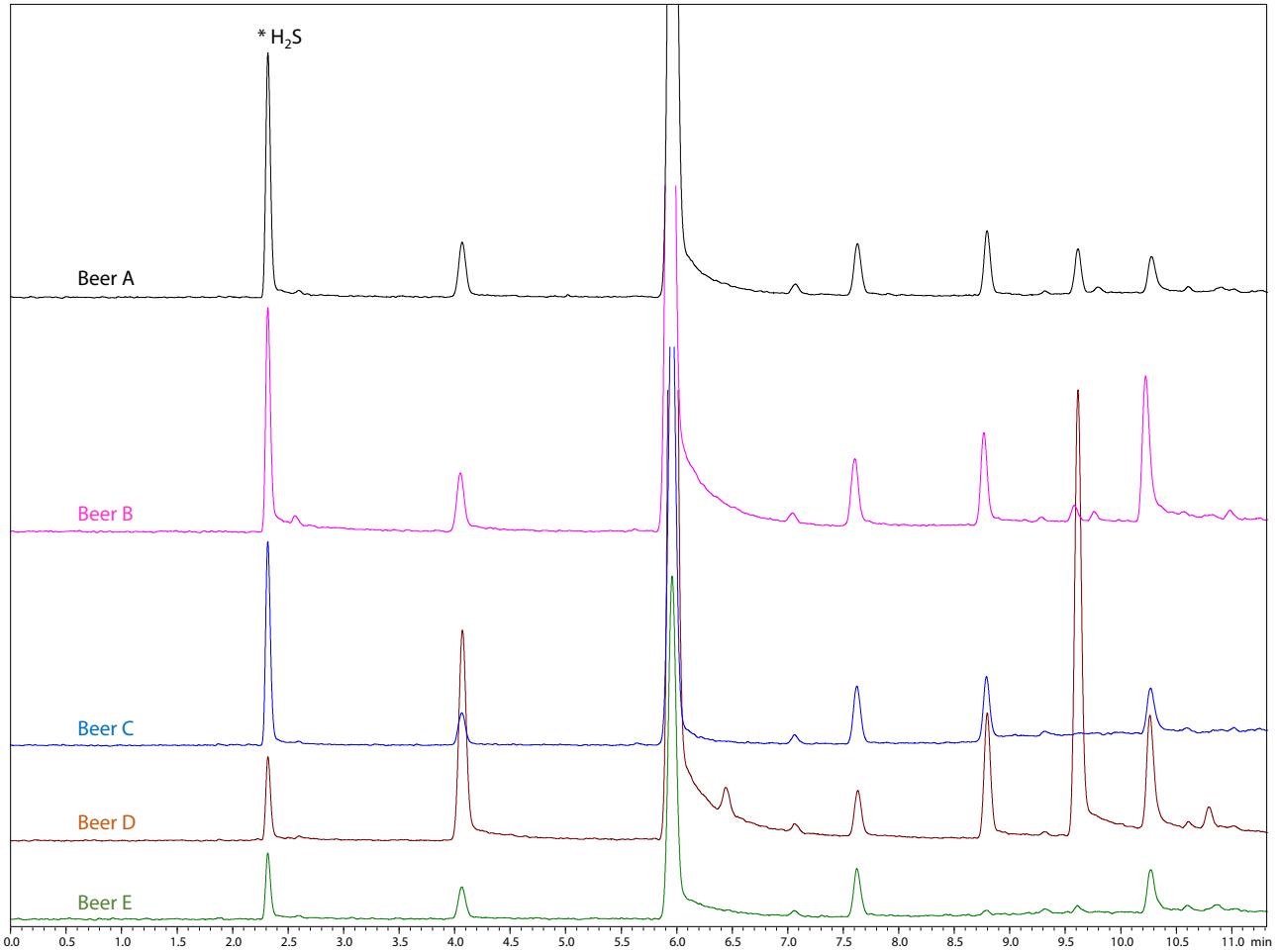

Fig. 3 Chromatograms of Beer Samples

Fig. 3 shows the chromatograms of each beer sample. Although a large number of volatile sulfur compounds were detected, the peaks indicated by the * mark are hydrogen sulfide.

As shown in Table 2, the hydrogen sulfide concentrations obtained in this experiment were in the range of 0.87 to 3.49 ng/mL. Beer samples A, B, and C are lager beers, and D and E are ale beers. A difference in the hydrogen sulfide concentrations of these two types of beer can confirmed, suggesting that the difference in the fermentation process and the effect on flavor can be seen from the hydrogen sulfide concentration.

Table 2 Hydrogen Sulfide Concentrations of Beer Samples

Sample	Concentration of H_2S (ng/mL)
Beer A	3.49
Beer B	3.41
Beer C	2.65
Beer D	1.20
Beer E	0.87

■ Conclusion

Hydrogen sulfide at or below the ng/mL level was analyzed and quantified by using a Shimadzu Nexis SCD-2030 sulfur chemiluminescence detection system and HS-20 headspace analysis system.

This experiment confirmed that a difference in the concentration of hydrogen sulfide between beer samples exists, depending on the beer manufacturing process. Because the concentration of hydrogen sulfide effects the aroma and flavor of beer, monitoring of the hydrogen sulfide concentration is expected to provide a useful index for control of product quality and the product manufacturing process.

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