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Application News

Nexis™ GC-2030 Gas Chromatograph

High-Sensitivity Analysis of Fragrance Components by Trap Mode of HS-20 Trap Headspace Sampler

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User Benefits

- The trap mode enables analysis of trace components which were undetectable with conventional headspace samplers.
- With the HS-20 Trap, concentration of the headspace gas and high-sensitivity analysis are possible with simple setting and operation.

■ Introduction

Headspace samplers enable high-sensitivity analysis of volatile components by holding the sample injected into the vial under time/temperature-controlled conditions and introducing the gas phase (headspace gas) into a gas chromatograph (GC). In the trap mode of Shimadzu HS-20 Trap headspace sampler, it is possible to concentrate the headspace gas and analyze ultra-trace components that had been impossible to analyze with conventional headspace samplers. In this article, the sensitivity of ordinary headspace analysis (loop mode) and the trap mode was compared by analyzing the fragrance components in a liquid food sample.

■ Sample Concentration by Trap Mode

An electronic cooling trap is built into the trap model of the HS-20, enabling concentration and high-sensitivity analysis of headspace gas, as illustrated in Fig. 1. The headspace gas is transferred to the cooled trap tube and concentrated multiple times. The concentrated headspace gas is then introduced into the GC by heating, making it possible to analyze the sample with high sensitivity. Simple switching between the trap mode and the normal loop mode is possible by using method files, and the trap mode and loop mode can also be combined during continuous analyses.

■ Sample and Analysis Conditions

A 2.5 mL sample of a food product in liquid form was injected as-is into the headspace vial. The vial was then set in the headspace sampler, and a headspace analysis was carried out by the GC. Table 1 shows the instrument configuration and analysis conditions used in this experiment.

Table 1 GC Instrument Configuration and Analysis Conditions

Model	: HS-20 Trap / Nexis GC-2030
HS-20 Trap	
Mode	: Trap (trap tube: Tenax®TA)
Multi Injection Times	: 5 times
Oven Temperature	: 60 ˚C
Sample Line Temperature	: 100 ˚C
Transfer Line Temperature	: 100 ˚C
Trap Cooling Temperature	: -10 ˚C
Trap Heating Temperature	: 280 ˚C
Trap Waiting Temperature	: 25 ˚C
Vial Pressure	: 80 kPa
Dry Purge Pressure	: 60 kPa
Vial Heat-retention Time	: 30 min
Vial Pressurization Time	: 1 min
Vial Pressurization Equilibrating Time	: 0.1 min
Loading Time	: 1 min
Loading Pressurization Time	: 0.1 min
Dry Purge Time	: 1 min
Injection Time	: 3 min
Needle Flush Time	: 5 min
GC	
Injection Mode	: Split
Split Ratio	: 1 : 20
Carrier Gas	: He
Carrier Gas Control	: Constant Linear Velocity (30 cm/s)
Column	: SH-Stabilwax™ (30 m × 0.25 mm I.D., 0.5 µm)
Column Temp.	: 40 ˚C - 5 ˚C/min – 240 ˚C
Detector	: FID-2030
Detector Temp.	: 260 ˚C
Detector Gas	: Make up (He) 24 mL/min H_2 32 mL/min Air 200 mL/min

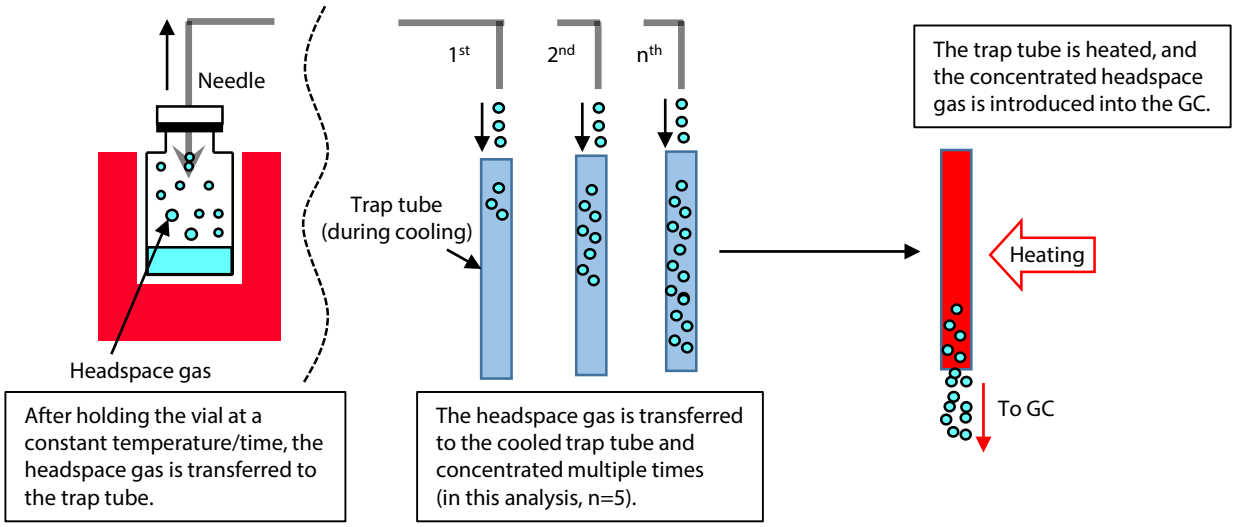

Fig. 1 Image of Sample Concentration in Trap Mode

■ Analysis Results

Fig. 2 shows a comparison of the chromatograms when the same sample was analyzed by the loop mode and the trap mode. Sensitivity was greatly increased by analysis in the trap mode. Here, peaks other than those originating from the sample (e.g., water, vial septum, atmosphere during injection) could also be detected, indicating that it is important to conduct a blank analysis to check for extraneous peaks. Fig. 3 shows a comparison of the chromatograms when three types of food samples were analyzed by the trap mode. The results confirmed that there were differences among the samples.

■ Conclusion

An actual food sample was analyzed by the trap mode and the loop mode of the HS-20 headspace sampler, demonstrating that sensitivity can be improved simply by using the trap mode. In addition, three types of food samples were analyzed using the trap mode, and differences among the samples were confirmed.

<Acknowledgment>
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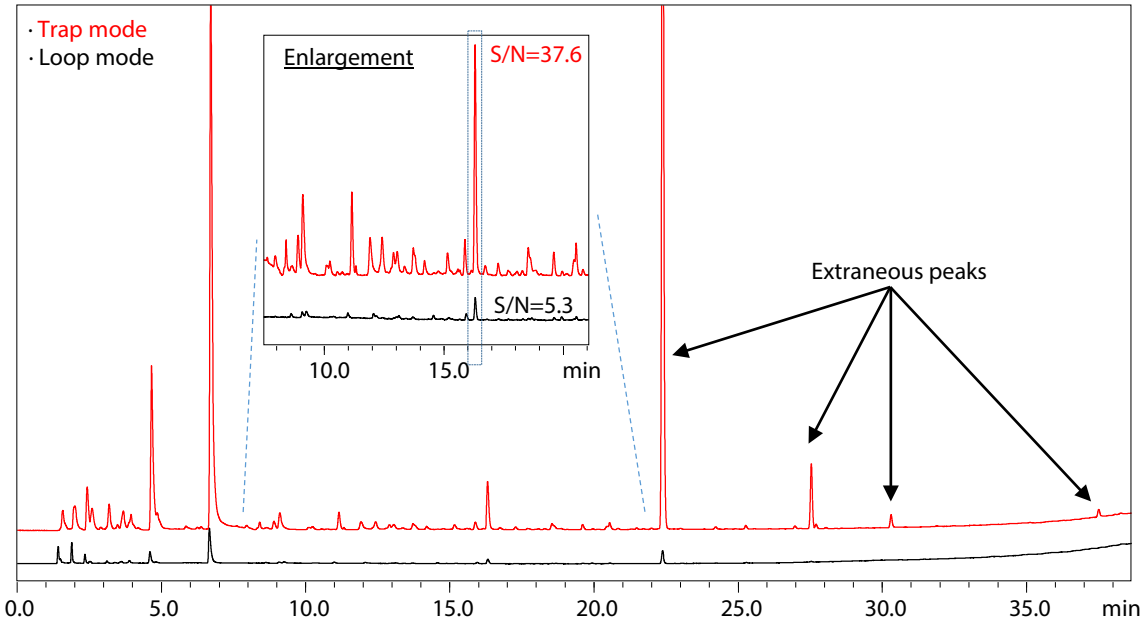

Fig. 2 Comparison of Chromatograms in Loop Mode and Trap Mode

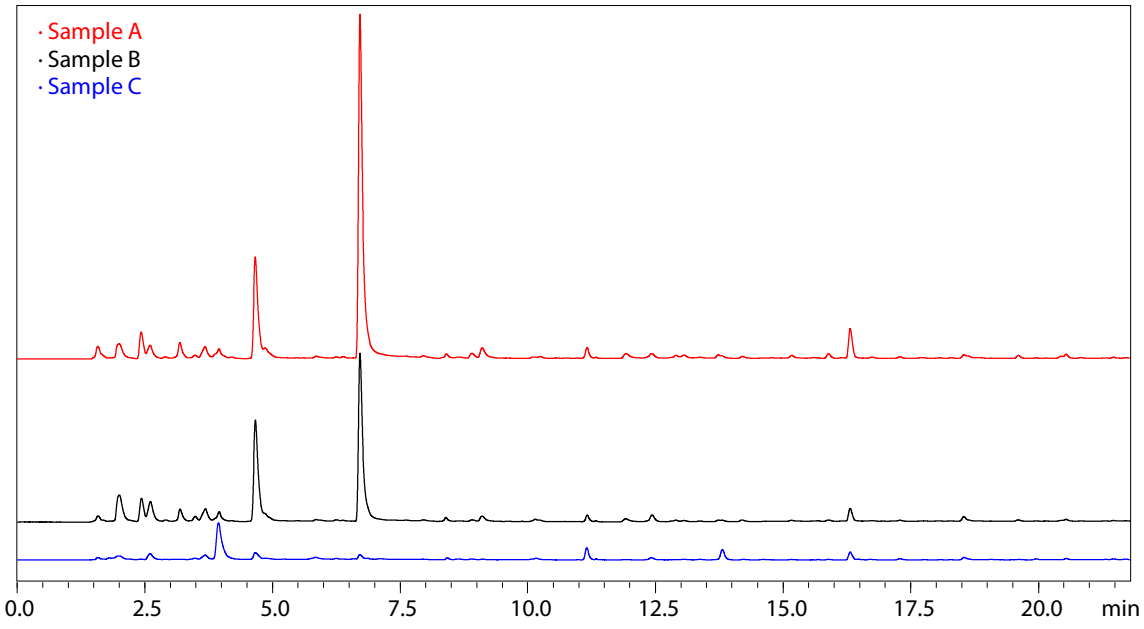

Fig. 3 Comparison of Chromatograms of Analysis of 3 Food Samples by Trap Mod

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