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Application News

No. M278A

Gas Chromatography Mass Spectrometry

High Sensitivity Analysis of White Wine Aroma Components Using ITEX DHS

Gas chromatograph mass spectrometers (GC-MS) capable of excellent qualitative measurements are used in the analysis of aroma components in foods and beverages. The convenient sampling methods of SPME (solid-phase microextraction) and HS (headspace extraction) are increasingly used for sample introduction. However, sample introduction methods such as these can suffer from insufficient sensitivity when analyzing some aroma components.

The ITEX DHS (in-tube extraction dynamic headspace) method was developed as a new sample introduction option for the AOC-6000 Multifunctional Autosampler to address this shortcoming. ITEX DHS allows analysis at higher sensitivities compared to the conventional headspace method by enriching headspace components contained in a vial into an adsorbent-filled syringe.

This article presents the results of analyzing white wine aroma components using ITEX DHS.

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Sample Introduction Using ITEX DHS

ITEX DHS involves repeatedly pumping a syringe inserted into the headspace area of a heated vial to enrich the adsorbent that fills the syringe's needle with volatile components.

Next, the adsorbent is heated above the GC injection port and then the volatile organic compounds are introduced into the GC injection port for analysis (Fig. 1). Higher sensitivity than the conventional HS method can be obtained by increasing the number of pumping repetitions during extraction.

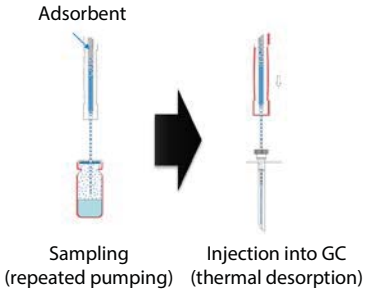

Fig. 1 **ITEX DHS Sample Introduction**

Sample and Analysis Conditions

A volume of 10 mL of commercially-available white wine poured into a 20 mL glass vial was used as the measurement sample. Table 1 lists the instruments and conditions used for analysis. For comparison, analysis was also performed using the conventional HS method.

Table 1 **Analysis Conditions**

GCMS	: GCMS-QP2020
Autosampler	: AOC-6000
Column	: DB-WAXetr (length: 60 m, 0.25 mm I.D., df = 0.25 µm)

ITEX DHS conditions

Trap	: Tenax® TA
Pre Cleaning Temp.	: 270 °C
Pre Cleaning Time	: 5 min
Incubation Temp.	: 60 °C
Incubation Time	: 10 min
Agitator Speed	: 500 rpm
Syringe Temp.	: 90 °C
Trap Temp.	: 40 °C
Extraction Strokes	: 50
Extraction Volume	: 1000 µL
Desorb Temp.	: 250 °C
Desorb Flow Rate	: 100 µL/sec
Injection Volume	: 500 µL

HS conditions

Incubation Temp.	: 60 °C
Incubation Time	: 10 min
Agitator Speed	: 500 rpm
Syringe Temp.	: 90 °C
Injection Flow Rate	: 10 mL/min
Injection Volume	: 500 µL

GC conditions

Vaporizing chamber temperature	: 250 °C
Injection mode	: Split (split ratio: 15)
Purge flow rate	: 3.0 mL/min
Control mode	: Linear Velocity (45 cm/sec)
Column oven temperature	: 40 °C (3 min) → 10 °C/min → 250 °C (10 min)

MS conditions

Interface temperature	: 230 °C
Ion source temperature	: 200 °C
Ionization method	: EI
Measurement mode	: Scan
Event time	: 0.3 sec

Analysis Results

Fig. 2 shows the analysis results for HS and ITEX DHS. While the HS method only detected major components such as alcohols, esters, and carboxylic acids, the ITEX DHS method additionally detected peaks of other trace components. In addition to alcohols, esters, and carboxylic acids, the peaks of aldehydes, furanones, and sulfur-based compounds were detected. Moreover, sensitivity in the ITEX DHS method can be increased further by increasing the number of pumping repetitions during extraction. Fig. 3 shows a comparison of the peak areas of dimethyl disulfide, known for its sulfurous odor, and furfural, a known barrel aroma, across several pumping repetitions for HS and ITEX DHS.

Conclusion

Trace components that prove difficult to detect using conventional HS can be analyzed with high sensitivity by employing ITEX DHS, which allows enrichment of volatile components

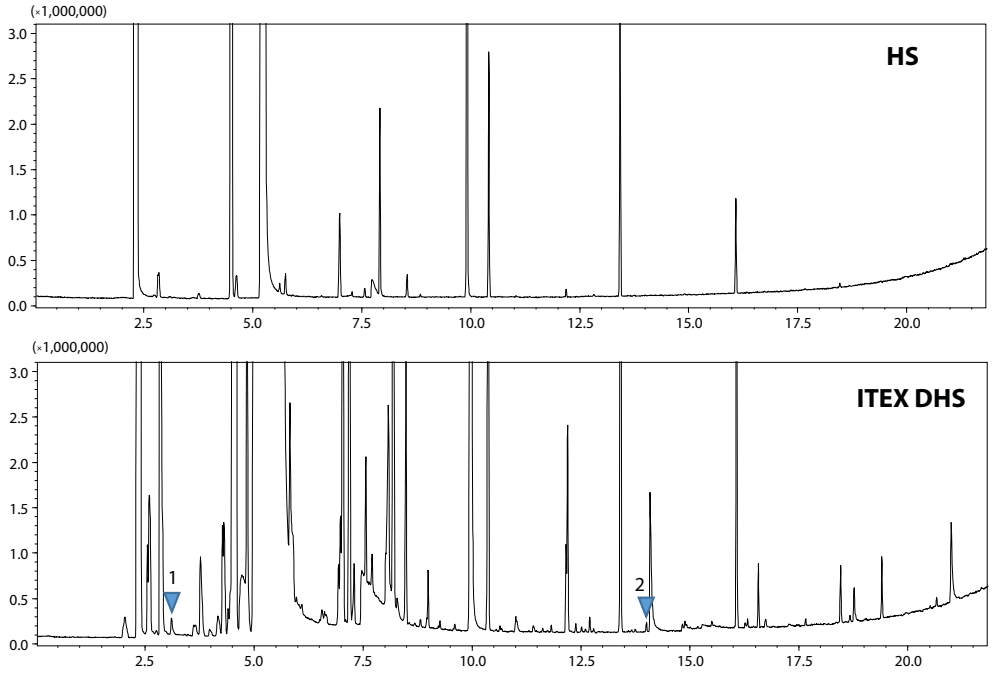

Fig. 2 **TIC Comparison of HS and ITEX DHS**

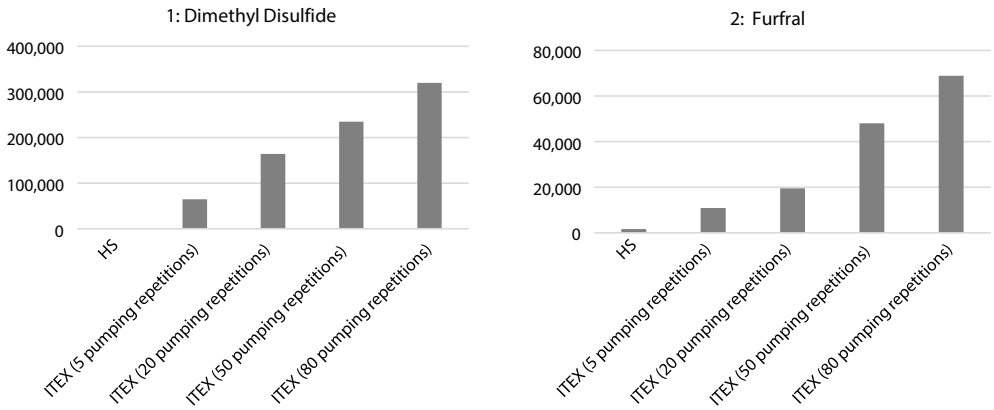

Fig. 3 **Comparison of Peak Areas For Each Pumping Repetition for HS and ITEX DHS**

Tenax is a registered trademark of Buchem B.V.

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