

Application News

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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.

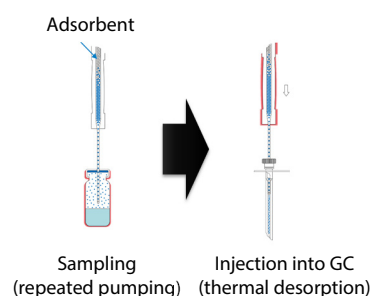


Fig. 1 ITEX DHS Sample Introduction

■ 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		HS conditions	
Trap	: Tenax® TA	Incubation Temp.	: 60 °C
Pre Cleaning Temp.	: 270 °C	Incubation Time	: 10 min
Pre Cleaning Time	: 5 min	Agitator Speed	: 500 rpm
Incubation Temp.	: 60 °C	Syringe Temp.	: 90 °C
Incubation Time	: 10 min	Injection Flow Rate	: 10 mL/min
Agitator Speed	: 500 rpm	Injection Volume	: 500 μL
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		
GC conditions		MS conditions	
Vaporizing chamber temperature	: 250 °C	Interface temperature	: 240 °C
Injection mode	: Split (split ratio: 15)	Ion source temperature	: 200 °C
Purge flow rate	: 3.0 mL/min	Ionization method	: EI
Control mode	: Constant pressure (83.5 kPa)	Measurement mode	: Scan
Column oven temperature	: 50 °C (5 min) → 10 °C/min → 250 °C (10 min)	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

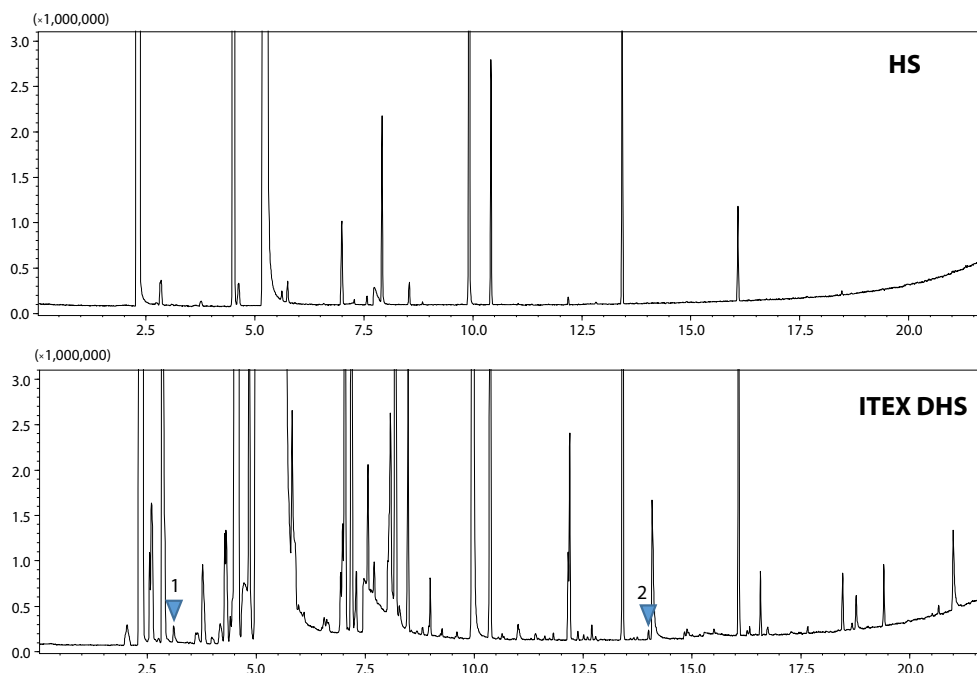


Fig. 2 TIC Comparison of HS and ITEX DHS

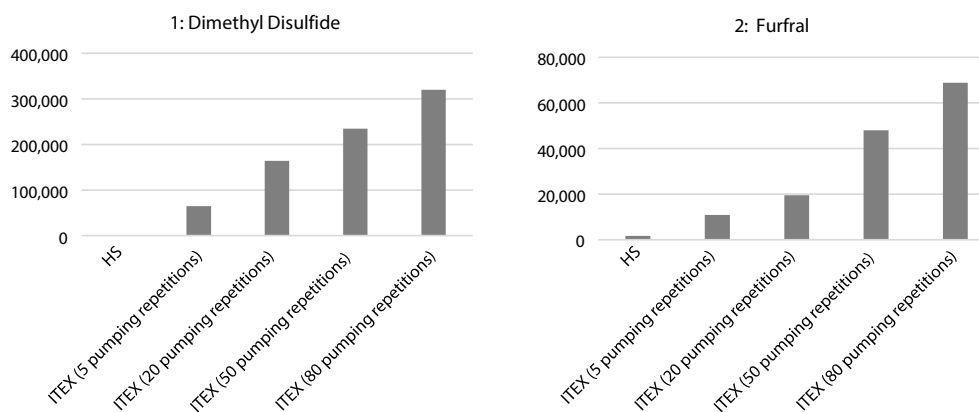


Fig. 3 Comparison of Peak Areas For Each Pumping Repetition for HS and ITEX DHS

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