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Application News

GC-MS GCMS-QP2020 NX, AOC™-6000 Plus

Improving Analytical Efficiency and Reliability of SPME Analysis through Optimal Selection of Vial Septa

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User Benefits

- The detection of cyclic siloxanes from vial septa can be minimized by using the low bleed septum for SPME.
- Reducing the detection of cyclic siloxanes makes data analysis easier and more efficient.
- It helps prevent overlooking compounds with retention times that overlap with cyclic siloxanes, thereby supporting more reliable analysis.

■ Introduction

SPME (Solid Phase Micro Extraction) is a method that allows for convenient and highly sensitive analysis of volatile organic compounds. It is used in a wide range of analyses that target volatile compounds, including odor analysis and outgas analysis from chemical products.

One of the challenges in SPME analysis has been the detection of cyclic siloxanes from vial septa, which often interfere with the analysis. For instance, the co-elution of sample-derived compounds and cyclic siloxanes can obscure sample-derived compounds, complicate the analysis, and potentially lead to overlooked trace compounds.

To address these issues, a low bleed septum for SPME (P/N: S225-47192-91, Fig. 1) that maximally suppresses the elution of cyclic siloxanes was developed. In this article, the fundamental performance of this low bleed septum for SPME is presented, and examples of its use in the analysis of actual products are provided.

Fig. 1 Low Bleed Septum for SPME and Cap

■ Samples and Analytical Conditions

The multifunctional autosampler AOC-6000 Plus and the GCMS-QP2020 NX (Fig. 2) were used for the analysis.

For blank analysis, ultra-pure water was utilized, 10 mL sealed in a 20 mL vial, and an empty 20 mL vial. As an example of actual product analysis, food container samples (PET resin products) were analyzed. The food containers were finely cut, and 0.5 g was sealed in a 20 mL vial for analysis.

The analytical conditions were individually set according to each evaluation purpose. The details of the analytical conditions are shown in Table 1.

Additionally, for the vial septum, either the newly developed low bleed septum for SPME or the standard septum for SPME was used, and the results from each were compared.

Fig. 2 GCMS-QP2020 NX + AOC™-6000 Plus

Table 1 System Configuration and Analytical Conditions

System:	GCMS-QP2020 NX / AOC-6000 Plus
◆ Evaluation of Blanks	
SPME Arrow:	PDMS (O.D.: 1.1 m, film thickness: 100 µm)
Vial Incubation Temp.:	80 °C, 150 °C
Vial Incubation Time:	5 min
Sample Extract Time:	30 min
Sample Desorb Time:	2 min
Column:	InertCap 5MS/Sil (30 m, 0.32 mm i.d., df = 0.5 µm)
Injection Mode:	Split
Split Ratio:	5
Carrier Gas:	He
Carrier Gas Control:	Constant pressure (83.5 kPa)
Oven Program:	50 °C (5 min)→10 °C/min→250 °C (10 min)
Ion Source Temp.:	200 °C
Interface Temp.:	250 °C
Event Time:	0.3 sec
Data Acquisition Mode:	Scan (*m/z* 45 - 500)
◆ Analysis of Food Container	
SPME Arrow:	DVB/Carbon WR/PDMS (O.D.: 1.1 m, film thickness: 120 µm)
Vial Incubation Temp.:	80 °C
Vial Incubation Time:	5 min
Sample Extract Time:	30 min
Sample Desorb Time:	2 min
Column:	InerCap Pure-WAX (30 m, 0.25 mm i.d., df = 0.25 µm)
Injection Mode:	Split
Split Ratio:	5
Carrier Gas:	He
Carrier Gas Control:	Constant pressure (83.5 kPa)
Oven Program:	50 °C (5 min)→10 °C/min→250 °C (10 min)
Ion Source Temp.:	200 °C
Interface Temp.:	250 °C
Event Time:	0.3 sec
Data Acquisition Mode:	Scan (*m/z* 45 - 500)

■ Blank Evaluation under Various Conditions

In order to evaluate the basic performance of the low bleed septum, a blank evaluation was conducted under various conditions typically used. The TIC chromatogram is shown in Fig. 3. Fig. 3 displays the results of the blank analysis when each type of septum was used with ultra-pure water (10 mL, heated to 80 °C) and in an empty vial (heated to 80 °C and 150 °C). With the standard septum, multiple peaks of cyclic siloxanes were detected under all conditions. In particular, it was observed that the detection intensity of cyclic siloxanes tended to increase when the heating temperature in the empty vial was higher. Conversely, when using the low bleed septum, no significant peaks of cyclic siloxanes were detected on the TIC chromatogram under any of the conditions. These results confirmed the superior low bleed and heat resistant characteristics of the low bleed septum.

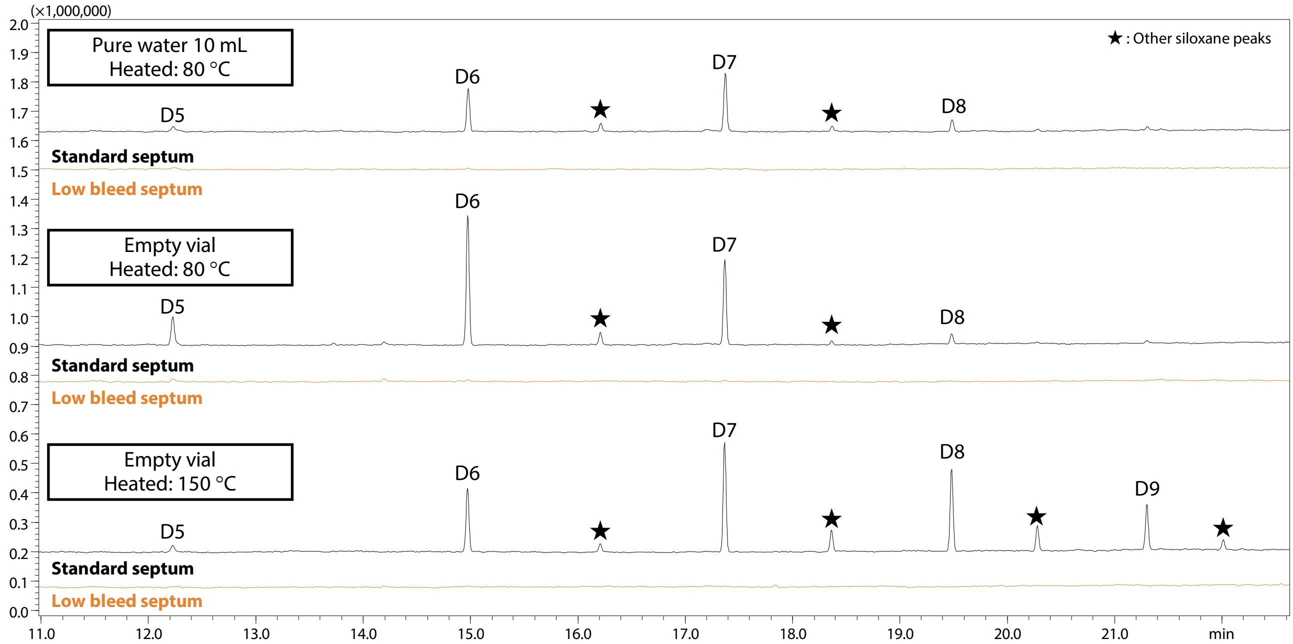

Fig. 3 Comparison of TIC Chromatograms of Blanks under Various Conditions (Displayed with Base Shift)

■ Product Analysis Using a Low Bleed Septum

A food container was analyzed as an example of product analysis using a low bleed septum. When analyzing aroma compounds or outgases from solid samples like food containers, the SPME method allows for a simple analysis, requiring only sealing in a vial as sample preparation. For this evaluation, an SPME Arrow coated with DVB/Carbon WR/PDMS was used, which easily concentrates a wide range of compounds. The TIC chromatogram is displayed in Fig. 4. In cases where a standard septum was used, cyclic siloxanes were detected with signal strengths similar to the peaks from the sample.

Conversely, when the low bleed septum was used, detection of cyclic siloxanes was suppressed, and only peaks from the sample were clearly identifiable. Additionally, Fig. 5 presents an enlarged view of the chromatogram around D8. As shown in Fig. 5, if the retention times of cyclic siloxanes and target compounds overlap, the target compounds may be obscured by the peaks of the cyclic siloxanes, posing a risk of overlooking them in cases of trace compounds. By using a low bleed septum, interference from cyclic siloxanes is minimized, thereby reducing the risk of overlooking compounds and enhancing the reliability of the analysis.

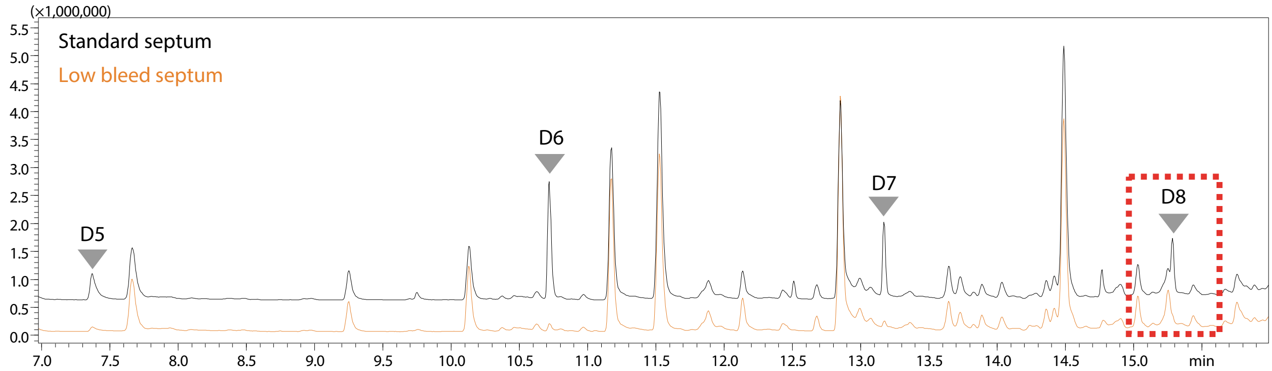

Fig. 4 TIC Chromatograms of Food Container with Each Septum

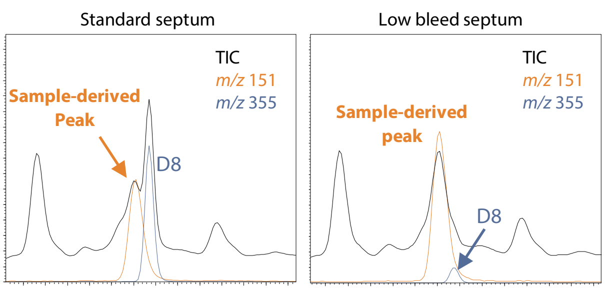

Fig. 5 Enlarged View of the Chromatograms around D8

■ Conclusion

The low bleed septum for SPME has significantly reduced the elution of cyclic siloxanes from the septum, an issue that was prevalent with conventional septa. This leads to more efficient analysis and improved data reliability when compared with traditional septa.

It can be effectively employed across a wide range of fields, including the analysis of aroma and volatile compounds in water, beverages, food, and resin materials.

< Related Materials >
Smart SPME Fibers and Arrow Selection Guide
[c146e424.pdf (shimadzu.com)]

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