SHIMADZU
Excellence in Science

Application News

No. 277A

Gas Chromatography Mass Spectrometry

High Sensitivity Analysis of Coffee Aroma Components Using the SPME Arrow

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 SPME Arrow method was developed as a new sample introduction option for the AOC-6000 Multifunctional Autosampler to address this shortcoming. The larger sorption phase volume compared to conventional SPME fibers allows the SPME Arrow to achieve high enrichment of volatile components and serve as a solution for applications where sensitivity was previously lacking.
This article presents the results of analyzing coffee aroma components using the SPME Arrow.

K. Kawamura

Sample Introduction Using the SPME Arrow

The SPME Arrow enables analysis at high sensitivities due to approximately 5 to 20 times more sorption phase than conventional SPME fibers (Fig. 1).

The thick and robust structure of the SPME Arrow also provides higher durability over conventional SPME fibers.

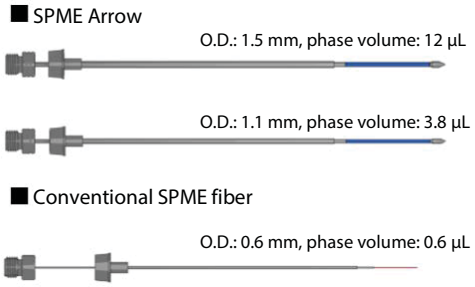

Fig. 1 **Comparison of SPME Arrow and Conventional SPME Fibers**

Sample and Analysis Conditions

A sample of 2 g of commercially-available ground coffee beans were weighed out, placed into a 20 mL vial, and set in the AOC-6000. Table 1 lists the analysis conditions. For comparison, analysis was also performed using the conventional SPME 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)

SPME Arrow conditions

SPME Arrow	: PDMS (O.D.: 1.1 mm, film thickness: 100 µm, length: 20 mm)
Conditioning Temp.	: 270 °C
Pre Conditioning Time	: 5 min
Incubation Temp.	: 60 °C
Incubation Time	: 8 min
Stirrer Speed	: 250 rpm
Sample Extract Time	: 30 min
Sample Desorb Time	: 2 min (250 °C: GC vaporizing chamber temperature)

SPME conditions

SPME fiber	: PDMS (film thickness: 100 µm, length: 10 mm)
Conditioning Temp.	: 270 °C
Pre Conditioning Time	: 5 min
Incubation Temp.	: 60 °C
Incubation Time	: 8 min
Agitator Speed	: 250 rpm
Sample Extract Time	: 30 min
Sample Desorb Time	: 2 min (250 °C: GC vaporizing chamber temperature)

GC conditions

Vaporizing chamber temperature	: 250 °C
Injection mode	: Splitless
Purge flow rate	: 5.0 mL/min
Control mode	: Linear Velocity (25,5 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 the SPME Arrow and SPME fibers. Known aroma components of coffee that were detected include short-chain aldehydes, phenols, pyridines, and pyrazines in addition to multiple sulfur-based compounds. Compared to the conventional SPME fiber, the SPME Arrow enabled high enrichment and analysis of the aroma components. Fig. 3 shows a comparison of the mass chromatogram peak area of several aroma components.

Conclusion

In contrast to conventional SPME fibers, analysis of trace components that prove difficult with SPME can be achieved using the SPME Arrow, which is coated with a larger volume of sorption phase.

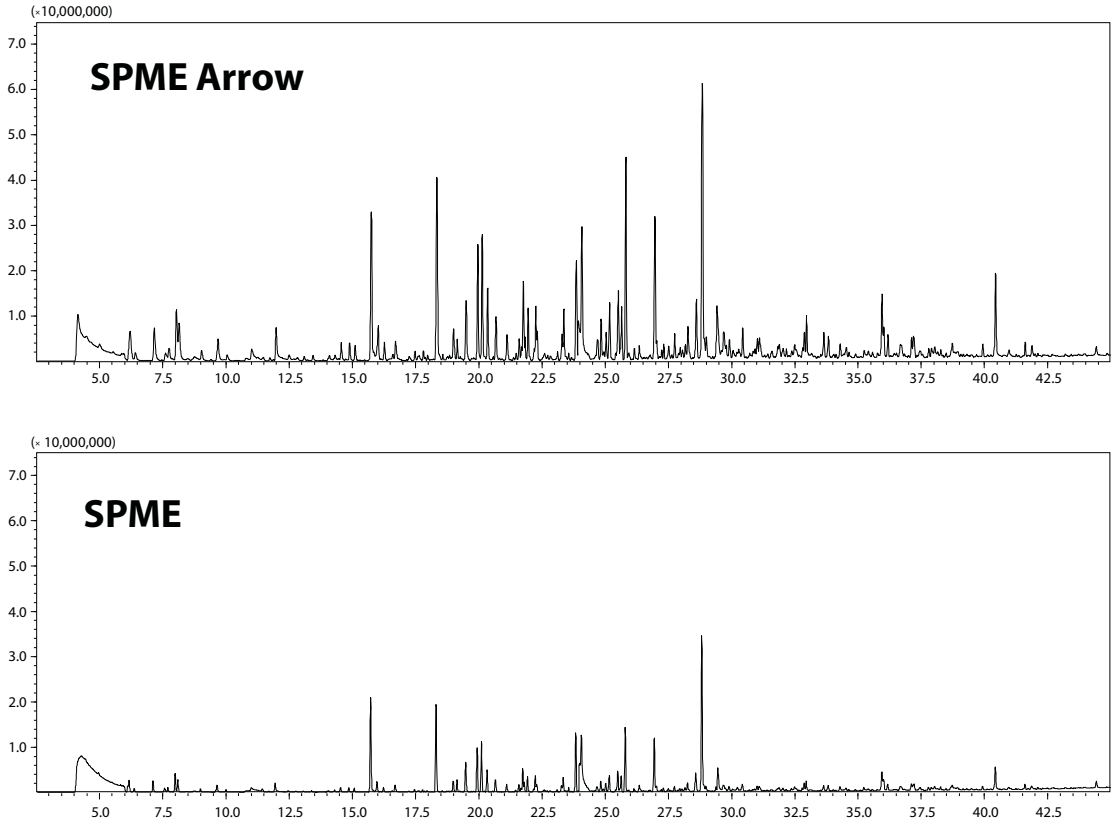

Fig. 2 **Comparison of SPME Arrow and Conventional SPME Fibers**

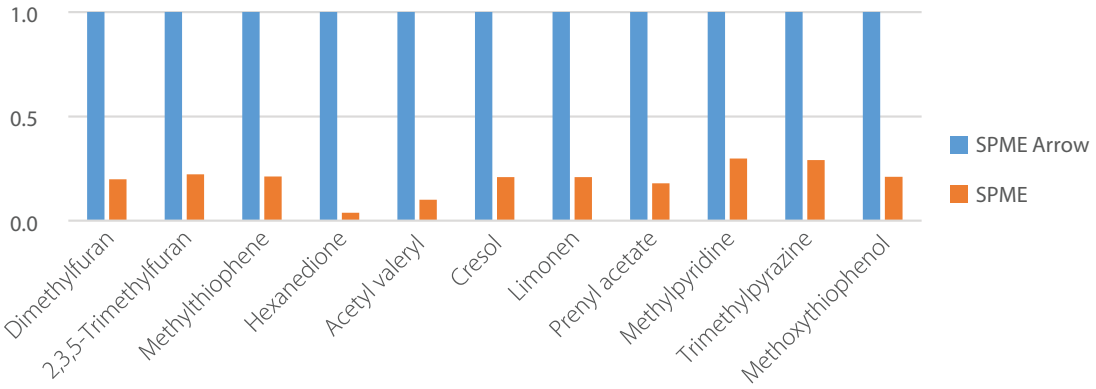

Fig. 3 **Peak Area Comparison of Typical Aroma Components (Area Comparison Where SPME Arrow Value is 1)**

First Edition: Jan. 2019
Second Edition: Oct. 2019

Shimadzu Corporation
www.shimadzu.com/an/