Application Brief

Food Testing and Agriculture

Response Comparison of Agilent SPME Arrows and Agilent SPME Fibers with DVB/Carbon WR/PDMS Phase for Free Volatile Phenols

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Abstract

Solid phase microextraction (SPME) has become one of the most widely used extraction technologies for volatile aromatics. Its popularity for use stems from its operational simplicity, suitability for automation, reduced use of organic solvents, and direct thermal desorption into a gas chromatograph. Additionally, SPME combines the matrix separation of analytes with a concentrating step. With the introduction of the Agilent SPME Arrows, the choice between an Agilent SPME fiber or SPME Arrow has become a common application question. This application demonstrates the significant benefit in extraction efficiency of the Agilent DVB/carbon WR/PDMS SPME Arrow (1.1 mm) larger sorption phase volume.

Experimental

For both SPME fibers and SPME Arrows, there are similar parts that are worth noting (Figure 1). The hub is the colored top of the SPME fiber or SPME Arrow, which signifies the phase type. The adjustable needle guide controls the septum piercing needle. The septum piercing needle has two purposes. The first purpose is to pierce the septa for extraction and desorption. The second purpose is to house and protect the phase. For the SPME Arrow, the septum piercing needle is only used for housing and protecting the phase. The arrow-shaped septum-piercing tip pierces the septa when using an SPME Arrow.

SPME fiber properties

The SPME fiber has a 10 mm phase length, a 9.4 mm^2 sorption phase surface area, and a 0.6 µL sorption phase volume.

SPME Arrow properties

SPME Arrows combine trace level sensitivity with high mechanical robustness. The SPME Arrow has an outside diameter of 1.10 or 1.50 mm, resulting in large sorption phase surfaces and volumes (Figure 2). The arrow-shaped tip allows smooth penetration of the vial and injector septa. In contrast to traditional SPME fibers, the SPME Arrow design fully protects the sorptive material, minimizing adverse influences and loss of analytes during the transfer processes.[1]

Method

Headspace solid phase microextraction has been shown to be a fast and effective sampling method for gas chromatography/mass spectrometry (GC/MS) analysis. The technique is used extensively for the determination of volatile compounds in wine. Guaiacol and 4-methylguaiacol are main target compounds implicated in smoke-affected grapes and wines.

The use of the DVB/carbon WR/PDMS SPME phase was chosen due to its selective extraction of odor and flavor compounds. An Agilent PAL3 autosampler with robotic tool change (RTC) was installed on an Agilent 8890 GC system with an Agilent 5977B GC/MSD. The SPME headspace parameters, GC method settings, and MS conditions are listed in Tables 1, 2, and 3, respectively.

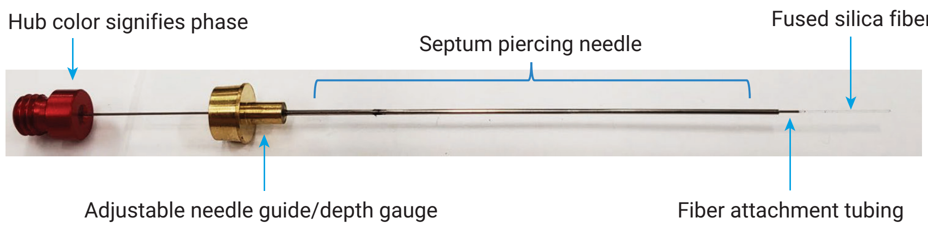

Figure 1. Characteristics of a classical 100 µm PDMS SPME fiber.

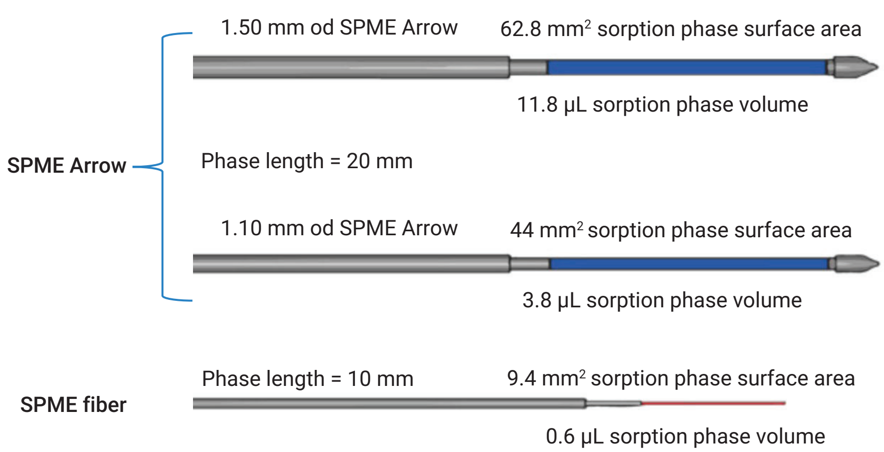

Figure 2. Sorption phase surface area and sorption phase volume comparisons for SPME Arrows and SPME fibers.

Table 1. SPME headspace parameters.

Parameter	Setting
Predesorption Time	3 min
Predesorption Temperature	250 °C
Incubation Time	5 min
Heatex Stirrer Speed	1,000 rpm
Heatex Stirrer Temperature	40 °C
Sample Extract Time	10 min
Sample Desorption Time	3 min

Table 2. Agilent 8890 GC settings.

Parameter	Setting
Inlet Liner	Agilent Ultra Inert inlet liner, splitless, straight, 0.75 mm id, recommended for SPME injections (p/n 5190-4048)
Injection Mode, Temperature	Splitless, 250 °C
Control Mode	Constant flow (1.2 mL/min)
Column	Agilent J&W DB-HeavyWAX GC column, 30 m, 0.32 mm, 0.25 µm (p/n 123-7132)
Oven Program	120 °C (hold 1 min); 10 °C/min to 250 °C (hold 0 min); 60 °C/min to 280 °C (hold 0 min)

Table 3. Agilent 5977B GC/MSD conditions.

Parameter	Setting
Transfer Line	280 °C
Acquisition Mode	SIM
Solvent Delay	3.0 min
Tune File	HES_Atune.u
Gain	1
MS Source Temperature	280 °C
MS Quadrupole Temperature	150 °C

Sample preparation

- 20 mL headspace vial and cap (part numbers 5188-6537 and 5188-2759)
- 10 mL sample with 4 g of NaCl
- Samples spiked at 50 ppb
- Agilent SPME fiber, DVB/C-WR/PDMS/10 (part number 5191-5874)
- Agilent SPME Arrow, DVB/carbon WR/PDMS, 1.10 mm, 120 µm (part number 5191-5861)

Results and discussion

In addition to guaiacol and 4-methylguaiacol, there are other target analytes that are often included the analysis of smoke affected wines (Figure 3). The use of the SPME Arrow was acknowledged for increasing sensitivity. Signal response was compared with extraction via an SPME fiber or an SPME Arrow (1.10 mm), both with the same DVB/carbon WR/PDMS SPME phase. Figure 3 displays the increase of the response with the use of the SPME Arrow (1.10 mm) compared to the SPME fiber. Figures 4 and 5 focus on the response increase of the two main target compounds in smoke impact analysis, guaiacol and 4-methylguaiacol, respectively. With the use of the SPME Arrow (1.10 mm; part number 5191-5861), the response for guaiacol was four times higher than with the SPME fiber (part number 5191-5874). The SPME Arrow response for 4-methylguaiacol was seven times higher than with the SPME fiber.

Additional information regarding this method can be found in Agilent application note *Analysis of Free Volatile Phenols in Smoke-Impacted Wines by SPME*.[2]

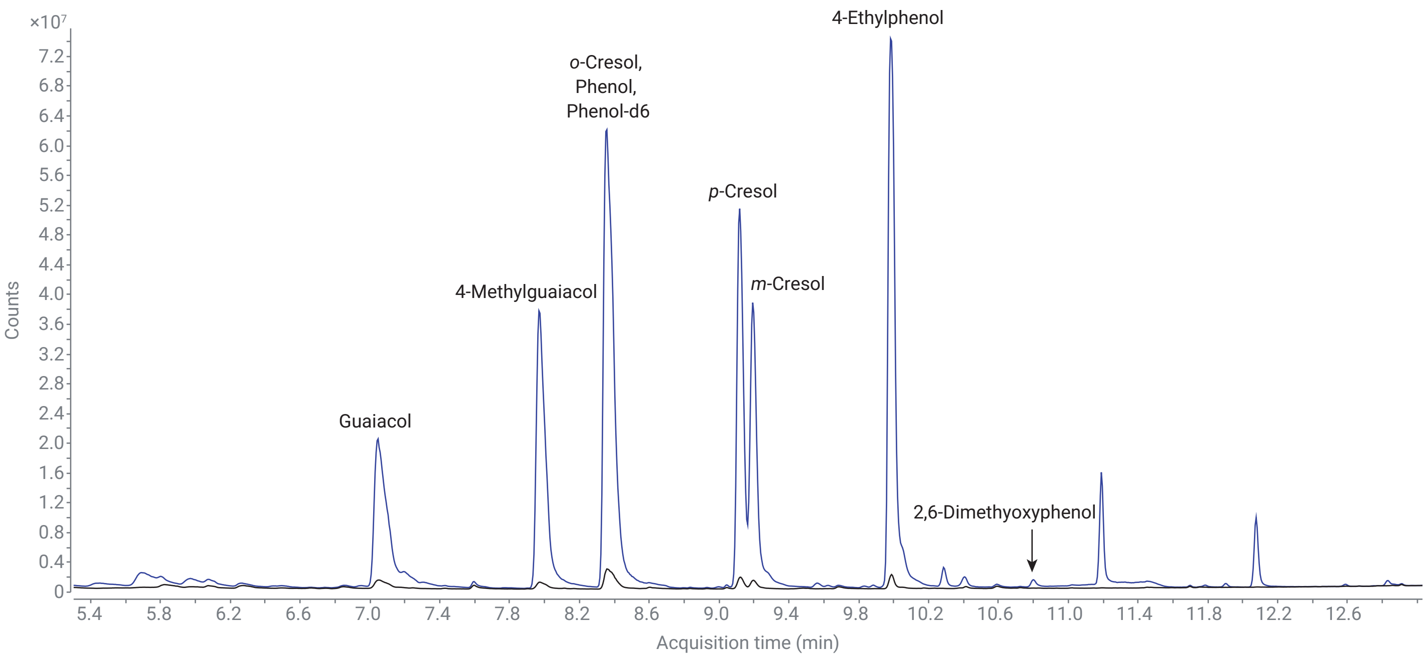

Figure 3. TIC scan of smoke impact compounds at 50 ppb extracted with the Agilent SPME fiber, DVB/C-WR/PDMS/10 (p/n 5191-5874, black trace) and the Agilent SPME Arrow, DVB/carbon WR/PDMS, 1.10 mm, 120 µm (p/n 5191-5861, blue trace).

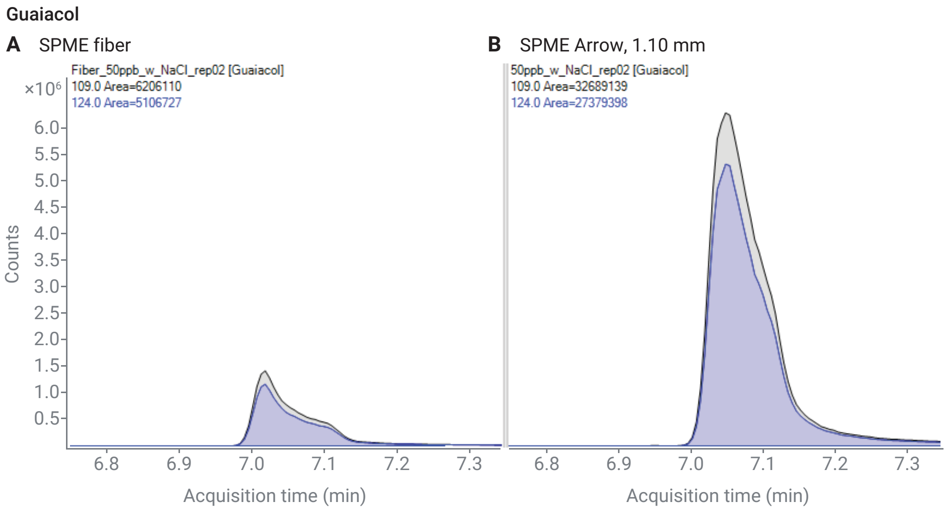

Figure 4. SIM traces of guaiacol at 50 ppb extracted with the A) Agilent SPME fiber, DVB/C-WR/PDMS/10 (p/n 5191-5874) and B) Agilent SPME Arrow, DVB/carbon WR/PDMS, 1.10 mm, 120 µm (p/n 5191-5861).

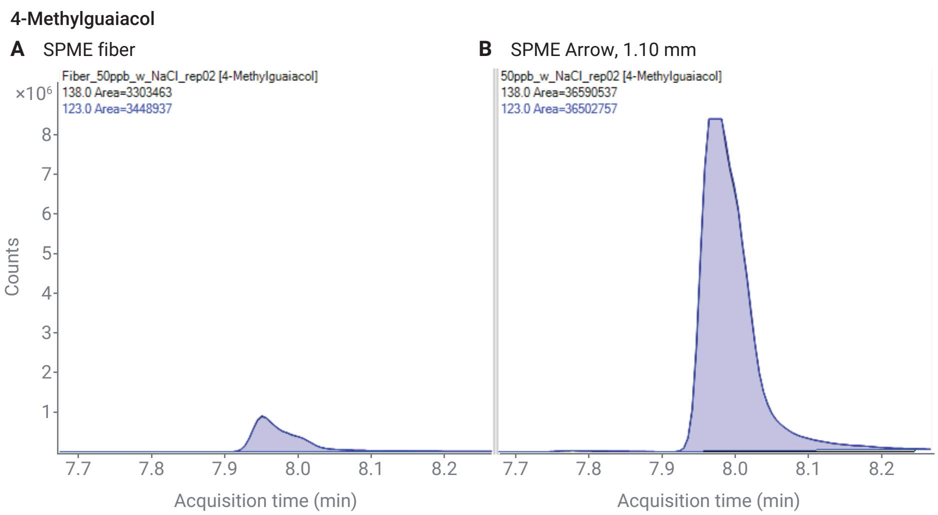

Figure 5. SIM traces of 4-methylguaiacol at 50 ppb extracted with the A) Agilent SPME fiber, DVB/C-WR/PDMS/10 (p/n 5191-5874) and B) Agilent SPME Arrow, DVB/carbon WR/PDMS, 1.10 mm, 120 µm (p/n 5191-5861).

Conclusion

In this application, the Agilent DVB/carbon WR/PDMS SPME Arrow (1.1 mm) demonstrates the significant benefit in extraction efficiency due to its larger sorption phase volume.

References

1. PAL Smart SPME Arrows: Bigger, Smarter, Better. PAL System – Ingenious sample handling, *CTC Analytics AG* **2020**.
2. Westland, J.; Abercrombie, V. Analysis of Free Volatile Phenols in Smoke-Impacted Wines by SPME. *Agilent Technologies application note*, publication number 5994-3161EN, **2021**.

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