Agilent CrossLab
From Insight to Outcome

Analysis of Free Volatile Phenols in Smoke-impacted Wines using GC/MS

Consumable Workflow Ordering Guide

Agilent

With the increase in wildfires over various regions around the globe, many growers and wineries continue to worry about smoke impact on grapes causing off-flavors in their wine.

Guaiacol and 4-methylguaiacol have been identified as the primary volatile aromatics that contribute to the undesirable smoke impact characteristic. While aging wine in oak barrels can also contribute to the concentration of guaiacol and 4-methylguaiacol, the ratio of these two compounds differ in smoke-impacted berries. While the aroma contributed by oak barrels is perceived as smoke and char, smoke impact is more reminiscent of campfires and ashtrays, which is not desirable in wine.

Detection limits for the analysis of smoke impact compounds must be sensitive enough to detect below 1 ppb, which is why selected ion monitoring (SIM) or multiple reaction monitoring (MRM) are commonly used in GC/MS analyses.

Direct analysis of wine can be challenging because of the sugars, organic acids, and other aromatic compounds with higher retentions. To simplify the extraction and analysis of these volatiles, solid phase microextractions (SPME) has become the extraction method of choice. Its popularity stems from:

- operational simplicity
- suitability for automation
- reduced use of organic solvents, and
- direct thermal desorption into a gas chromatograph

The Agilent SPME-GC/MS/MS method for the analysis of free-form volatile phenols associated with smoke impact allows for confident identification and reliable quantitation.[1]

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, Temp	Splitless, 250 °C
Control Mode	Constant flow (1.2 mL/min)
Column	Agilent J&W DB-HeavyWAX GC column, 30 m × 0.25 mm, 0.25 µm (p/n 122-7132)
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 7000D triple quadrupole GC/MS conditions.

Parameter	Setting
Transfer Line	280 °C
Acquisition Mode	dMRM
Solvent Delay	3.0 min
Tune File	Atune.eiex
Gain	10
MS Source Temperature	280 °C
MS Quadrupole Temperature	150 °C

The choice between an Agilent SPME fiber or SPME Arrow (Figure 1) is a common application question.

A comparison of Agilent's SPME Arrow and the SPME fiber, both with the same DVB/carbon WR/PDMS SPME phase demonstrated the SPME arrow to possess a greater extraction efficiency than SPME Fiber (Figure 2).[2] With the SPME arrow, the response was 4x higher for guaiacol and 7x higher for 4-methylguaiacol than the corresponding SPME fiber.

Moreover, the arrow shaped tip allowed smooth penetration of the vial and injector septa and in contrast to traditional SPME fibers, the SPME Arrow design fully protected the sorptive material, minimizing adverse influences and loss of analytes during the transfer processes.

To maximize the concentration of volatile components in the headspace, consider adding salt to the sample matrix to lower the partitioning coefficient (K) for some target analytes. An increased response of smoke impact volatiles was seen with the addition of 4 g of NaCl (Figure 3).[3]

References

1. Analysis of Free Volatile Phenols in Smoke-Impacted Wines by SPME, 5994-3161
2. Response Comparison of Agilent SPME Arrows and Agilent SPME Fibers with DVB/Carbon WR/PDMS Phase for Free Volatile Phenols 5994-3160EN
3. Use of Salt to Increase Analyte Concentration in SPME Headspace Applications, 5994-3159EN

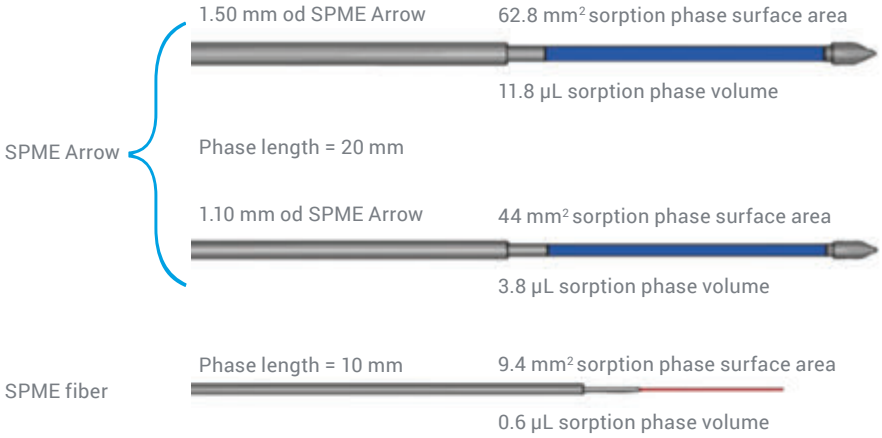


Figure 1. Sorption phase surface area and sorption phase volume comparisons for SPME Arrows and SPME fibers.

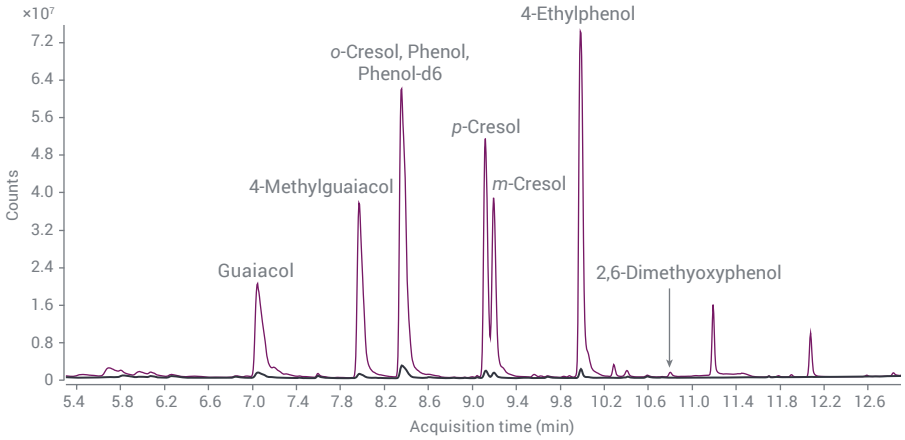


Figure 2. TIC scan of smoke impact compounds at 50 ppb extracted with the Agilent SPME fiber, DVB/C-WR/PDMS/10 (p/n 5191-5874, black trace) and the Agilent SPME Arrow, DVB/carbon WR/PDMS, 1.10 mm, 120 µm (p/n 5191-5861, purple trace).

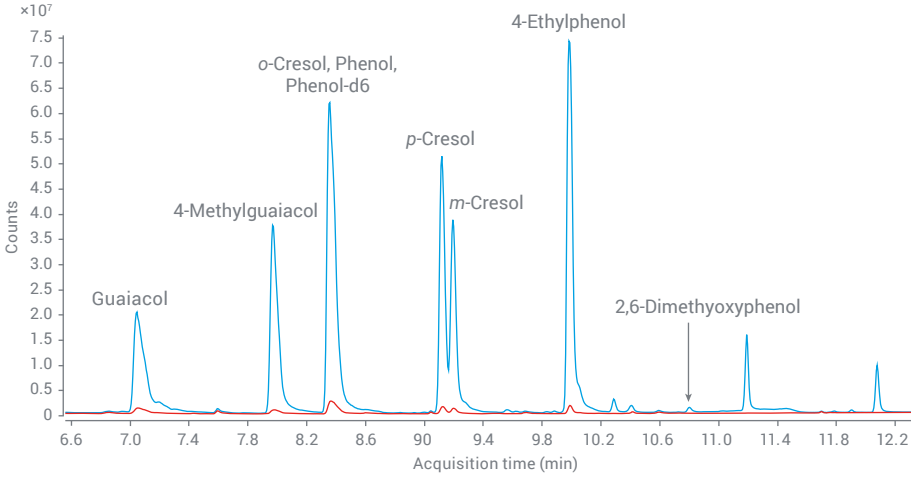


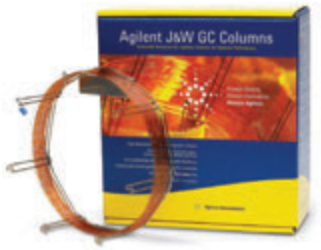
Figure 3. TIC scan of smoke impact compounds at 50 ppb extracted with the Agilent SPME Arrow, DVB/carbon WR/PDMS, 1.10 mm, 120 µm (p/n 5191-5861). The red trace indicates standards that were run without salt, and the blue trace indicates standards that were run with 4 g NaCl.

Ordering information

This guide provides recommendations for Agilent products used in this analysis, so you can find what you're looking for quickly. Click the MyList* links in the header below to add items to your "Favorite Products" list at the Agilent online store. Then, enter the quantities for the products you need. Your list will remain under "Favorite Products" for your use with future orders.

MyList of Columns and Supplies for analysis of free volatile phenols in smoke-impacted wines

Description	Part No.
Sample Preparation	
Agilent SPME Arrow DVB/carbon WR/PDMS, 1.10 mm, 120 µm (recommended)	5191-5861
Agilent SPME fiber, DVB/C-WR/PDMS/10	5191-5874
Standards	
Smoke Standard‡ Solution in MeOH; 10 µg/mL, 1 mL	CUS-00004677†
Internal Standard§ in MeOH, 10 µg/mL, 1 mL	CUS-00004678†
InfinityLab Ultrapure LCMS water	5191-4498
GC Columns	
Agilent J&W DB-HeavyWAX GC column, 30 m, 0.25 mm, 0.25 µm	122-7132
GC Supplies	
Agilent Ultra Inert inlet liner, splitless, straight, 0.75 mm id, recommended for SPME injections	5190-4048
Inlet septa, Advanced green, non-stick, 11 mm, 50/pk	5183-4759
Ultra Inert Gold seal, with washer, 1/pk	5190-6144
Self-Tightening column nut, collared, inlet	G3440-81011
Self-Tightening column nut, collared, MSD	G3440-81013
15%Graphite/85% Vespel Ferrules, 0.4 mm id, 10/pk	5181-3323
Vials & Caps	
20 mL amber headspace vials, screw top, 18 mm cap, 100/pk	5188-6537
Headspace Screw Cap, PTFE/silicone septa, 18 mm, 100/pk	5188-2759
MS Supplies	
EI Filament (for 7000A/B/C/D, 5977B Inert Plus, 5977A Extractor, Inert or Stainless steel and 5975 systems)	G7005-60061
HES Filament for 7010 Triple Quadrupole GC/MS	G7002-60001
Gas Filters	
Gas Clean Carrier Gas Kit for 7890	CP17988
Gas Clean Carrier Gas Kit for 8890 and 8860	CP179880
Gas Clean carrier gas purifier replacement cartridge	CP17973



* *If this is your first time using the Agilent Online Store, you will need to enter your email address for account verification. If you don't have a registered Agilent account, you will need to register at www.agilent.com/en/promotions/onlinestore-videos for one. This feature is valid only in regions that are e-commerce enabled. All items can also be ordered through your regular sales and distributor channels.*

† *To order these custom standard part numbers please visit www.agilent.com/chem/standards. Standards may not be available in some countries.*

‡ *Contains: 2,4-Dimethylphenol (CAS#105-67-9), 3,5-Xylenol (CAS#108-68-9), 4-Ethylguaiacol (CAS#2785-89-9), Creosol (2-Methoxy-4-Methylphenol) (CAS#93-51-6), Eugenol (CAS#97-53-0), Guaiacol (CAS#90-05-1), m-Cresol (CAS#108-39-4), o-Cresol (CAS#95-48-7), o-Ethylphenol (CAS#90-00-6), p-Cresol (CAS#106-44-5), Phenol (CAS#108-95-2), 4-Ethylphenol (CAS#123-07-9), 4-ethyl-p-Xylenol (CAS#95-87-4), Syringol (CAS#91-10-1), 2,6-Dimethoxy-4-methylphenol (CAS#6638-05-7).*

§ *Contains: Phenol-d6 (CAS#13127-88-3), Guaiacol-d3 (CAS#74495-69-5).*

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