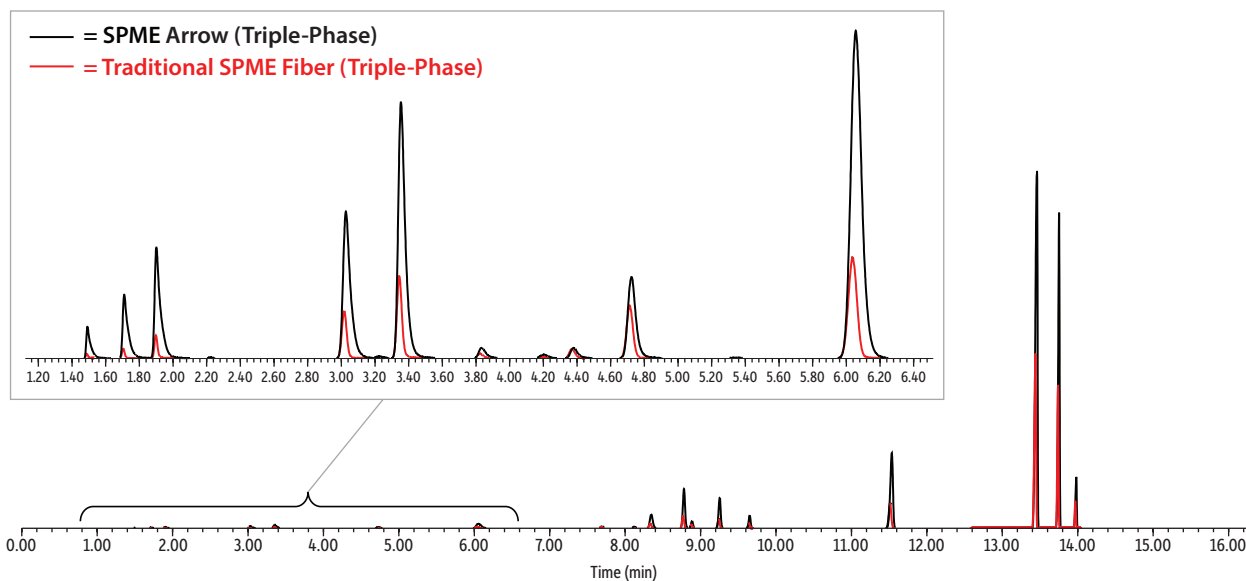


Triple-Phase SPME Arrow vs. Triple-Phase SPME Fiber

- Triple-phase SPME Arrows provide increased sensitivity on a more durable platform.
- Increased phase area and volume compared to traditional SPME fibers.



GC_GN1204

Peaks

1. *n*-Propane
2. Isobutane
3. *n*-Butane
4. Methanol
5. *n*-Pentane
6. Ethanol
7. Diethyl ether
8. Acetone
9. 2-Propanol
10. Acetonitrile
11. Methylene chloride
12. *n*-Hexane
13. Ethyl acetate
14. Chloroform
15. Cyclohexane
16. Benzene
17. 1,2-Dichloroethane
18. *n*-Heptane
19. Trichloroethene
20. Toluene
21. *m*-Xylene
22. *p*-Xylene
23. *o*-Xylene
24. N,N-Dimethylacetamide (solvent)

Column Sample

Rxi-624Sil MS, 30 m, 0.25 mm ID, 1.40 μ m (cat.# 13868)
Residual solvents #1 (cat.# 34105)

n-Propane, isobutane, *n*-butane (Emerald Scientific)
N,N-Dimethylacetamide

Diluent: 10 μ g/mL

Conc.: Direct

Injection

Liner: Topaz 1.8 mm ID straight/SPME inlet liner (cat.# 23280)

Inj. Temp.: 280 °C

Oven

Oven Temp.: 30 °C (hold 6 min) to 85 °C at 15 °C/min (hold 2 min) to 250 °C at 35 °C/min

Carrier Gas

Carrier Gas: He, constant flow

Flow Rate: 2 mL/min

Detector: FID @ 320 °C

Make-up Gas

Flow Rate: 40 mL/min

Make-up Gas Type: N₂

Hydrogen flow: 45 mL/min

Air flow: 450 mL/min

Data Rate: 20 Hz

Instrument

Agilent 7890B GC

Notes

A 6 mL sample for residual solvents analysis based on USP <467>

was prepared for headspace solid phase microextraction (HS-SPME) as follows:

1. 3 g sodium chloride was weighed into a 20 mL amber headspace vial (cat. # 23086) with screw top cap (cat. # 23090).
2. 6 mL of deionized water was added to the vial.
3. The sample was fortified at 10 μ g/mL with the residual solvents, *n*-propane, isobutane, and *n*-butane standards.
4. The vial was capped and vortexed at 3000 rpm for 10 seconds, inverted, then vortexed again for 10 seconds at 3000 rpm.
5. The sample was analyzed using the following CTC RTC parameters.

CTC RTC Parameters

HS-SPME Arrow

Tool: SPME Arrow and SPME fiber
 SPME Arrow: Restek PAL SPME Arrow, DVB/C-WR/PDMS, 1.1 mm, 120 μ m (cat.# 27875)
 SPME Fiber: Restek SPME fiber, DVB/C-WR/PDMS (cat.# 27873.1)
 Agitator Speed: 250 rpm
 Agitator Temp: 30 °C
 Incubation Time: 120 sec
 Heatex Stirrer Speed: 1000 rpm
 Heatex Stirrer Temp.: 30 °C
 Vial Penetration
 Depth: 35 mm
 Extraction Time: 120 sec
 Injector
 Penetration Depth: 50 mm
 Desorption Time: 10 sec
 Pre-Conditioning: True
 Post-Conditioning: False
 Conditioning Time: 60 sec
 Conditioning Temp.: 250 °C