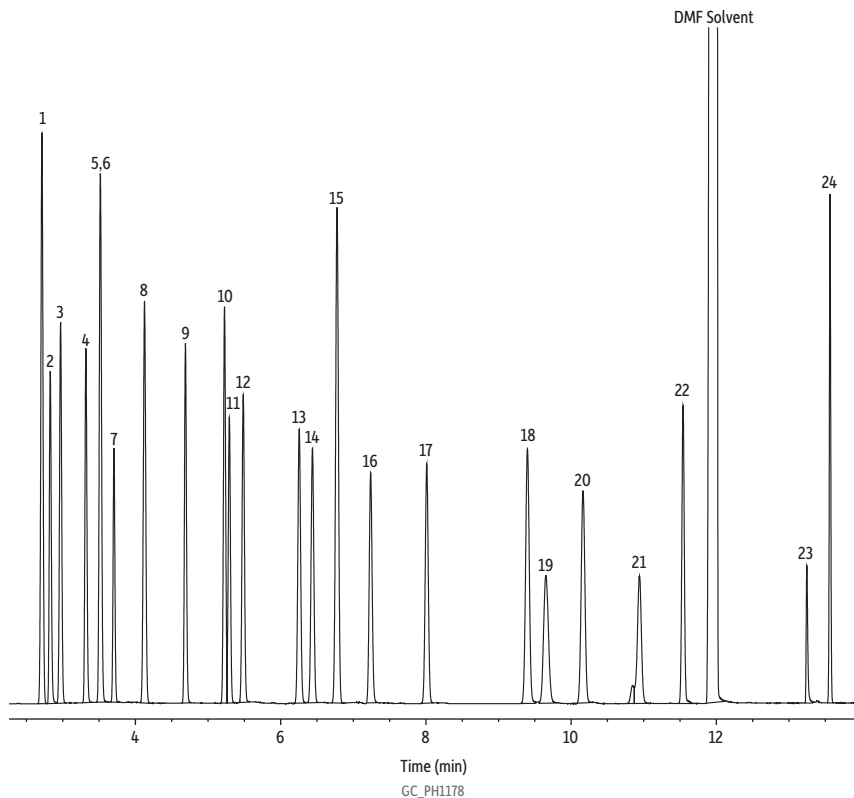


USP <467> Residual Solvents Class 3 Mix A on Rxi-624Sil MS



**Column** Rxi-624Sil MS, 30 m, 0.32 mm ID, 1.80  $\mu$ m (cat.# 13870)  
**Sample** Residual Solvents Class 3 - Mix A (cat.# 36013)  
**Conc.:** 5000  $\mu$ g/mL split 100:1  
**Injection** headspace-loop split (split ratio 100:1)  
**Liner:** Topaz 1 mm Split (cat.# 23333)  
**Headspace-Loop**  
**Inj. Port Temp.:** 200 °C  
**Instrument:** Tekmar HT-3  
**Inj. Time:** 1.0 min  
**Transfer Line Temp.:** 105 °C  
**Valve Oven Temp.:** 105 °C  
**Standby flow rate:** 50 mL/min  
**Sample Temp.:** 80 °C  
**Platen temp equil. time:** 80 min  
**Sample Equil. Time:** 1.0 min  
**Mixer time:** 5.0 min  
**Mixing level:** 5  
**Mixer stabilize time:** 0.50 min  
**Vial Pressure:** 10 psi  
**Pressurize Time:** 5.0 min  
**Pressure Equilibration Time:** 0.20 min  
**Loop Pressure:** 5 psi  
**Loop Fill Time:** 2.0 min  
**Oven**  
**Oven Temp.:** 35 °C (hold 3 min) to 60 °C at 24 °C/min to 80 °C at 3 °C/min to 280 °C at 25 °C/min  
**Carrier Gas** He, constant flow  
**Flow Rate:** 2.7 mL/min  
**Linear Velocity:** 41 cm/sec  
**Detector** FID @ 300 °C  
**Make-up Gas Flow Rate:** 45 mL/min  
**Make-up Gas Type:** N<sub>2</sub>  
**Hydrogen flow:** 40 mL/min  
**Air flow:** 450 mL/min  
**Data Rate:** 20 Hz  
**Instrument** Agilent/HP6890 GC

**Peaks**

1. *n*-Pentane
2. Ethanol
3. Diethyl ether
4. Acetone
5. Ethyl formate
6. 2-Propanol
7. Methyl acetate
8. Methyl *tert*-butyl ether
9. 1-Propanol
10. 2-Butanone
11. Ethyl acetate
12. 2-Butanol
13. Isopropyl acetate
14. Isobutanol
15. *n*-Heptane
16. 1-Butanol
17. Propyl acetate
18. 4-Methyl-2-pentanone
19. 3-Methyl-1-butanol
20. Isobutyl acetate
21. 1-Pentanol
22. Butyl acetate
23. Dimethyl sulfoxide
24. Anisole

<b>Conc.</b>	<b>tr (min)</b>	<b>(<math>\mu</math>g/mL)</b>
5000	2.71	5000
5000	2.82	5000
5000	2.96	5000
5000	3.31	5000
5000	3.51	5000
5000	3.52	5000
5000	3.70	5000
5000	4.12	5000
5000	4.68	5000
5000	5.22	5000
5000	5.29	5000
5000	5.48	5000
5000	6.25	5000
5000	6.43	5000
5000	6.77	5000
5000	7.23	5000
5000	8.01	5000
5000	9.39	5000
5000	9.65	5000
5000	10.16	5000
5000	10.94	5000
5000	11.54	5000
5000	13.24	5000
5000	13.56	5000