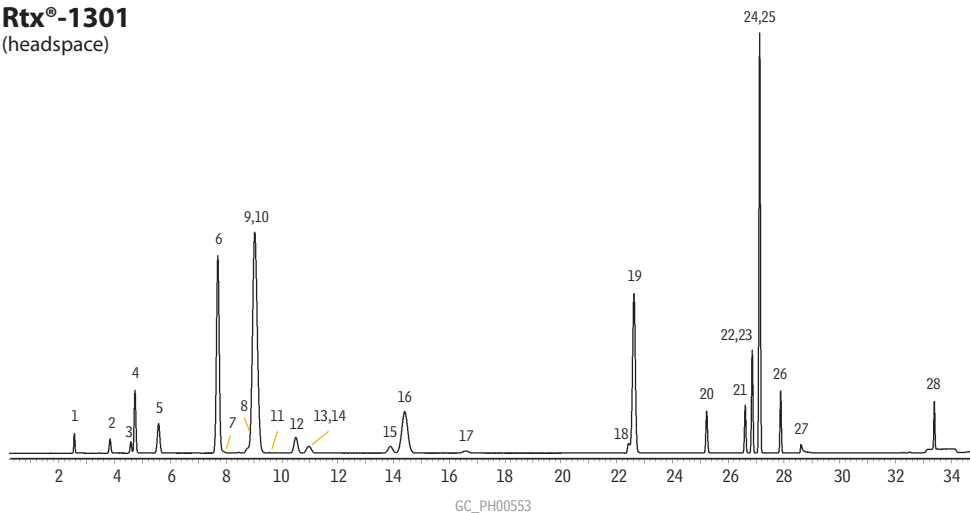


Residual Solvents

European Pharmacopoeia Class 1 and Class 2

Rtx®-1301

(headspace)



1. methanol
2. 1,1-dichloroethene
3. acetonitrile
4. methylene chloride (dichloromethane)
5. hexane (C6)
6. *cis*-1,2-dichloroethene
7. nitromethane
8. chloroform
9. cyclohexane
10. 1,1,1-trichloroethane
11. carbon tetrachloride
12. benzene
13. 1,2-dimethoxyethane
14. 1,2-dichloroethane
15. trichloroethylene (1,1,2-trichloroethene)
16. methylcyclohexane
17. 1,4-dioxane
18. pyridine
19. toluene
20. 2-hexanone
21. chlorobenzene
22. DMF
23. ethylbenzene
24. *m*-xylene
25. *p*-xylene
26. *o*-xylene
27. N,N-dimethylacetamide
28. 1,2,3,4-tetrahydronaphthalene

Column: Rtx®-1301, 30m, 0.53mm ID x 3.0µm (cat.# 16085)
 Sample: headspace injection of 28 Class 1 and 2 residual solvents for pharmaceutical processing, prepared at the regulatory limit concentration, using samples shaken and heated at 80°C for 15 min., 1mL headspace injection 40°C (hold 20 min.) to 240°C @ 10°C/min. (hold 20 min.)
 Oven temp.: 40°C (hold 20 min.) to 240°C @ 10°C/min. (hold 20 min.)
 Inj./det. temp.: 200°C/250°C
 FID sensitivity: 1.1 x 10¹¹ AFS
 Carrier gas: hydrogen @ 35cm/sec.
 Split ratio: 2:1

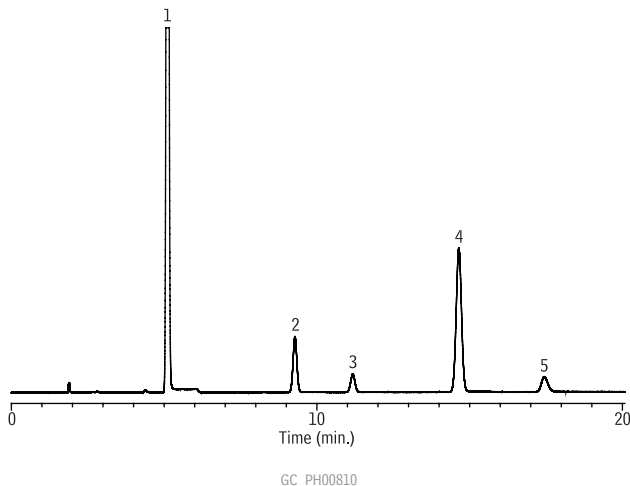


Organic Volatile Impurities

USP <467>

Rtx®-G43

(static headspace)



Column: Rtx®-G43, 30m, 0.53mm ID, 3.0µm (cat.# 16085-126)
 Sample: USP <467> Calibration Mixture #5 (cat.# 36007) in DMSO stock standard. To each 22mL headspace vial 5ml water, ~ 1.0g of sodium sulfate and 100µL of stock standard were added.
 The preparation yielded the following concentrations,

	Retention Time (min.)	Sample Concentration (µg/mL)
1. dichloromethane	5.110	12.0
2. chloroform	9.285	1.2
3. benzene	11.173	0.04
4. trichloroethylene	14.647	1.6
5. 1,4-dioxane	17.436	7.6

Inj.: static headspace injection (see static headspace conditions)
 Inj. temp.: 180°C
 Carrier gas: helium, split 2:1
 Linear velocity: 5mL/min., constant flow
 Oven temp.: 40°C for 20 min. to 240°C at 25°C/min. (hold for 10 min.)
 Det.: FID @ 250°C
 hydrogen flow: 40mL/min.
 air flow: 450mL/min.
 make-up flow: 45mL/min.

Static Headspace (Loop) Conditions:
 Instrument: Teledyne Tekmar HT3
 Valve oven temp.: 150°C
 Transfer line temp.: 150°C
 Standby flow rate: 10mL/min.
 Platen/sample temp.: 80°C
 Platen temp equil. time: 2.00 min.
 Sample equil. time: 15.00 min.
 Mixer time: 2.00 min.
 Mixing level: 5
 Mixer stabilize time: 0.50 min.
 Pressurize: 15psi
 Pressurize time: 2.00 min.
 Pressurize equil. time: 0.50 min.
 Loop fill pressure: 5psi
 Loop fill time: 2.00 min.
 Loop fill equil. time: 0.50 min.
 Inject time: 1.00 min.