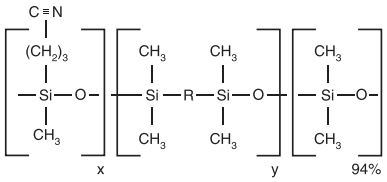
Organic Volatile Impurities (OVI) Analysis

G43 phase

Rxi®-624Sil MS Structure



Similar to: (6%-cyanopropylphenyl)-methylpolysiloxane

similar phases

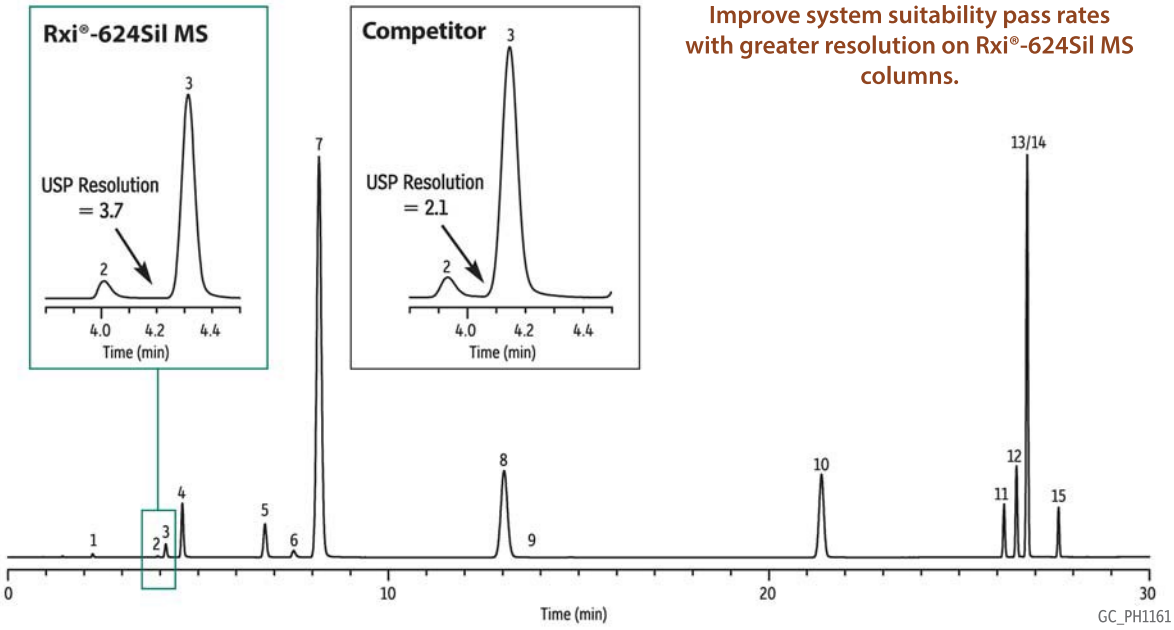
DB-624, VF-624ms, CP-Select 624 CB

Rxi®-624Sil MS Columns (fused silica)
(midpolarity Crossbond® phase)

- Low-bleed, high-thermal stability column—maximum temperatures up to 320 °C.
- Inert—excellent peak shape for a wide range of compounds.
- Selective—G43 phase highly selective for volatile organics and residual solvents, great choice for USP<467>.
- Manufactured for column-to-column reproducibility—well-suited for validated methods.

ID	df	temp. limits	20-Meter cat.#	30-Meter cat.#	60-Meter cat.#	75-Meter cat.#	105-Meter cat.#
0.18 mm	1.00 µm	-20 to 300/320 °C	13865				
0.25 mm	1.40 µm	-20 to 300/320 °C		13868	13869		
0.32 mm	1.80 µm	-20 to 300/320 °C		13870	13872		
0.53 mm	3.00 µm	-20 to 280/300 °C		13871	13873	13874	13875

Competitor Comparison: Class 2 - Mix A Residual Solvents for USP <467> Water-Soluble Articles



Column	Rxi®-624Sil MS, 30 m, 0.32 mm ID, 1.80 µm (cat.# 13870)
Sample	Residual solvents class 2 - mix A (cat.# 36271)
Diluent:	Water
Injection	Headspace-loop split (split ratio 5:1)
Liner:	1 mm split (cat.# 20972)
Headspace-Loop	
Inj. Port Temp.:	140 °C
Instrument:	Tekmar HT3
Inj. Time:	1 min
Transfer Line Temp.:	110 °C
Valve Oven Temp.:	110 °C
Sample Temp.:	80 °C
Sample Equil. Time:	60 min
Vial Pressure:	10 psi
Pressurize Time:	0.5 min
Pressure Equilibration Time:	0.05 min
Loop Pressure:	5 psi
Loop Fill Time:	0.1 min
Oven	
Oven Temp.:	40 °C (hold 20 min) to 240 °C at 10 °C/min (hold 20 min)
Carrier Gas	He, constant flow
Linear Velocity:	35 cm/sec
Dead Time:	1.45 min @ 40 °C
Detector	FID @ 250 °C
Data Rate:	5 Hz
Instrument	Agilent/HP6890 GC
Acknowledgement	Teledyne Tekmar

Peaks	tR (min)	Conc. (µg/mL)
1. Methanol	2.281	25.00
2. Acetonitrile	4.009	3.42
3. Dichloromethane	4.313	5.00
4. *trans*-1,2-Dichloroethene	4.798	7.83
5. *cis*-1,2-Dichloroethene	7.028	7.83
6. Tetrahydrofuran	7.706	5.75
7. Cyclohexane	8.708	32.33
8. Methylcyclohexane	14.099	9.83
9. 1,4-Dioxane	15.054	3.17
10. Toluene	22.018	7.42
11. Chlorobenzene	26.570	3.00
12. Ethylbenzene	26.837	3.07
13. *m*-Xylene	27.147	10.85
14. *p*-Xylene	27.147	2.53
15. *o*-Xylene	27.927	1.63

Organic Volatile Impurities (OVI) Analysis

Stabilwax® Columns (fused silica)
(polar phase; Crossbond® polyethylene glycol)

- Most stable polyethylene glycol (PEG) column available.
- Rugged enough to withstand repeated water injections.
- Lowest-bleed PEG column on the market; long column lifetimes.
- Temperature range: 40 °C to 260 °C.
- Equivalent to USP G14, G15, G16, G20, and G39 phases.

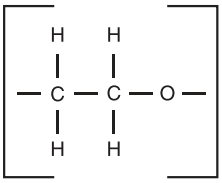
Restek's polar-deactivated surface tightly binds the Carbowax® polymer and increases thermal stability, relative to competitive columns. Because of the increased stability produced by the bonding process, Stabilwax® columns exhibit long column lifetimes, even when programming repeatedly up to 260 °C. The bonding mechanism of the column also produces polar compound retention times that do not shift, as is often observed on other wax-type columns. In addition, this bonding mechanism produces a column that can be rejuvenated by solvent washing.

ID	df	temp. limits	15-Meter cat.#	30-Meter cat.#	60-Meter cat.#
0.25 mm	0.10 µm	40 to 250/260 °C	10605	10608	10611
	0.25 µm	40 to 250/260 °C	10620	10623	10626
	0.50 µm	40 to 250/260 °C	10635	10638	10641
0.32 mm	0.25 µm	40 to 250/260 °C	10621	10624	10627
	0.50 µm	40 to 250/260 °C	10636	10639	10642
	1.00 µm	40 to 240/250 °C	10651	10654	10657
0.53 mm	0.25 µm	40 to 250/260 °C	10622	10625	10628
	0.50 µm	40 to 250/260 °C	10637	10640	10643
	1.00 µm	40 to 240/250 °C	10652	10655	10658
	1.50 µm	40 to 230/240 °C	10666	10669	10672
	2.00 µm	40 to 220/230 °C	10667	10670	

ID	df	temp. limits	10-Meter cat.#	20-Meter cat.#
0.15 mm	0.15 µm	40 to 250/260 °C	43830	43831
0.18 mm	0.18 µm	40 to 250 °C		40602

G16 phase

Stabilwax® Structure



similar phases

HP-INNOWax, CP-Wax 52 CB, VF-WAX MS, ZB-WAXplus

ordering note

Get the protection without the connection!
For Stabilwax® columns with built-in Integra-Guard® guard columns, see **page 23**.

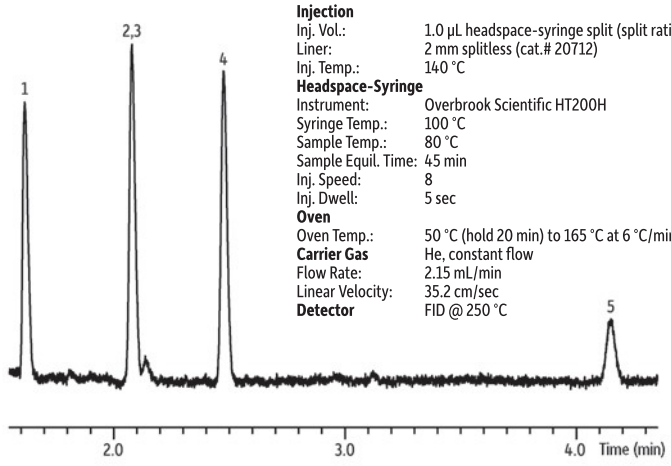
free literature

Custom Residual Solvent Mixes

Download your free copy from
www.restek.com

lit. cat.#
PHTS1212

Residual Solvents (Class 1) on Stabilwax® (G16)



Column	Stabilwax®, 30 m, 0.32 mm ID, 0.25 µm (cat.# 10624)
Sample	Residual solvents - class 1 (cat.# 36279) in 20 mL headspace vial (cat.# 24685)
Diluent:	Water
Injection	
Inj. Vol.:	1.0 µL headspace-syringe split (split ratio 5:1)
Liner:	2 mm splitless (cat.# 20712)
Inj. Temp.:	140 °C
Headspace-Syringe	
Instrument:	Overbrook Scientific HT200H
Syringe Temp.:	100 °C
Sample Temp.:	80 °C
Sample Equil. Time:	45 min
Inj. Speed:	8
Inj. Dwell:	5 sec
Oven	
Oven Temp.:	50 °C (hold 20 min) to 165 °C at 6 °C/min (hold 20 min)
Carrier Gas	He, constant flow
Flow Rate:	2.15 mL/min
Linear Velocity:	35.2 cm/sec
Detector	FID @ 250 °C

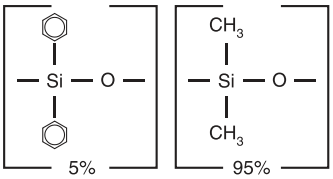
Peaks
1. 1,1-Dichloroethene
2. 1,1,1-Trichloroethane
3. Carbon tetrachloride
4. Benzene
5. 1,2-Dichloroethane

System suitability criteria met

GC_PH00951

G27 phase

Rtx®-5 Structure



Similar to: (5%-phenyl)-methylpolysiloxane

similar phases

HP-5, DB-5, CP-Sil 8 CB, ZB-5

NOTE: DB-5MS is a silarylene-based polymer, similar to Rxi-5Sil MS.

USP
Pharmaceutical Standards
See **pages 595–596**.

Organic Volatile Impurities (OVI) Analysis

Rtx®-5 (G27) Columns (fused silica)
(low-polarity phase; Crossbond® diphenyl dimethyl polysiloxane)

- General-purpose columns for drugs, solvent impurities, pesticides, hydrocarbons, PCB congeners (e.g., Aroclor mixes), essential oils, semivolatiles.
- Temperature range: -60 °C to 350 °C.
- Equivalent to USP G27 and G36 phases.

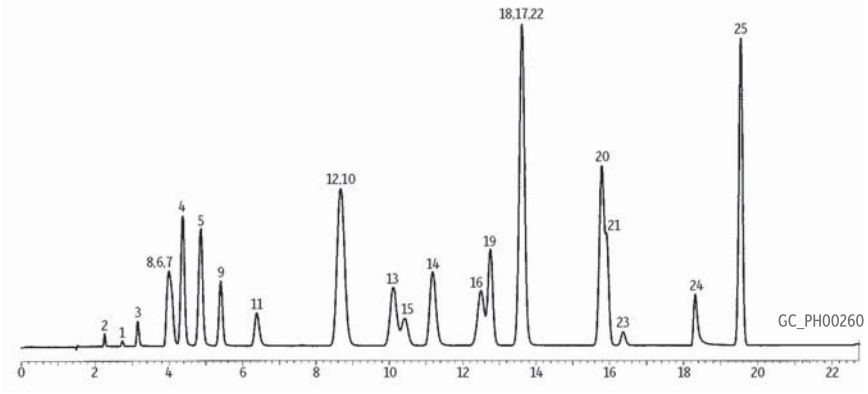
The diphenyl dimethyl polysiloxane stationary phase is the most popular GC stationary phase and is used in a wide variety of applications. All residual catalysts and low molecular weight fragments are removed from the Rtx®-5 polymer, providing a tight mono-modal distribution and extremely low bleed.

ID	df	temp. limits*	15-Meter cat.#	30-Meter cat.#	60-Meter cat.#	105-Meter cat.#
0.25 mm	0.10 µm	-60 to 330/350 °C	10205	10208	10211	
	0.25 µm	-60 to 330/350 °C	10220	10223	10226	10229
	0.50 µm	-60 to 330/350 °C	10235	10238	10241	10244
	1.00 µm	-60 to 325/340 °C	10250	10253	10256	10259
0.32 mm	0.10 µm	-60 to 330/350 °C	10206	10209		
	0.25 µm	-60 to 330/350 °C	10221	10224	10227	
	0.50 µm	-60 to 330/350 °C	10236	10239	10242	
	1.00 µm	-60 to 325/340 °C	10251	10254	10257	10260
	1.50 µm	-60 to 310/330 °C	10266	10269	10272	10275
	3.00 µm	-60 to 280/300 °C	10281	10284	10287	10290
0.53 mm	0.10 µm	-60 to 320/340 °C	10207	10210		
	0.25 µm	-60 to 320/340 °C	10222	10225	10228	
	0.50 µm	-60 to 320/330 °C	10237	10240	10243	
	1.00 µm	-60 to 320/330 °C	10252	10255	10258	
	1.50 µm	-60 to 310/330 °C	10267	10270	10273	
	3.00 µm	-60 to 270/290 °C	10282	10285	10288	
	5.00 µm	-60 to 270/290 °C	10277	10279	10283	

ID	df	temp. limits	10-Meter cat.#	20-Meter cat.#	40-Meter cat.#
0.18 mm	0.20 µm	-60 to 325/340 °C	40201	40202	40203
	0.40 µm	-60 to 315/330 °C	40210	40211	40212

*Maximum temperatures listed are for shorter length columns. Longer columns may have a different maximum temperature.

Organic Volatile Impurities on Rtx®-5 (Rtx®-G27)



Peaks
1. Ethylene oxide
2. Methanol
3. Ethanol
4. Diethyl ether
5. 1,1-Dichloroethene
6. Acetone
7. Isopropanol
8. Acetonitrile
9. Methylene chloride
10. *n*-Hexane
11. *n*-Propanol
12. Methyl ethyl ketone
13. Ethyl acetate
14. Tetrahydrofuran
15. Chloroform
16. 1,1,1-Trichloroethane
17. Carbon tetrachloride
18. Benzene
19. 1,2-Dichloroethane
20. Heptane
21. Trichloroethylene
22. *n*-Butanol
23. 1,4-Dioxane
24. Pyridine
25. Toluene

Column Rtx®-5 w/5m Integra-Guard® Column (Rtx®-G27), 30 m, 0.53 mm ID, 5.00 µm (cat.# 10279-126)
Sample Headspace injection of common solvents for pharmaceutical processing. Prepared to equal about 500 ppm in the bulk pharmaceutical. Samples shaken and heated at 90 °C for 15 minutes, 1 mL headspace injection.
Injection
Inj. Vol.: 1,000 µL headspace-syringe split (split ratio 2:1)
Inj. Temp.: 220 °C
Oven
Oven Temp.: 35 °C (hold 10 min) to 100 °C at 5 °C/min to 240 °C at 25 °C/min (hold 5 min)
Carrier Gas He, constant pressure
Linear Velocity: 35 cm/sec @ 35 °C
Detector FID @ 240 °C
Notes FID sensitivity: 1.05 x 10^{-11} AFS

Organic Volatile Impurities (OVI) Analysis

Rtx®-G27 Column (fused silica with 5-meter Integra-Guard® guard column)
(Crossbond® diphenyl dimethyl polysiloxane)

- Application-specific columns for residual solvents in pharmaceutical products.
- Analytical column with Integra-Guard® guard column eliminates connecting problems and leaks.
- Rtx®-G27 stable to 290 °C.

Some methods require the use of a guard column. Our Integra-Guard® integrated guard column system makes it easy to comply.

			30-Meter with 5-Meter, 0.53mm ID Integra-Guard Guard Column
ID	**df**	**temp. limits**	**cat.#**
0.53 mm	5.00 µm	-60 to 270/290 °C	10279-126

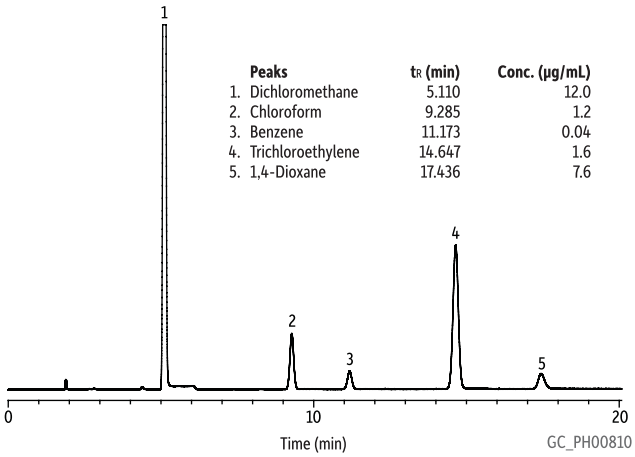
Rtx®-G43 Column (fused silica with 5-meter Integra-Guard® guard column)
(Crossbond® cyanopropylmethyl phenylmethyl polysiloxane)

- Application-specific columns for residual solvents in pharmaceutical products. Meet all requirements of USP <467>.
- Analytical column with Integra-Guard® guard column eliminates connecting problems and leaks.
- Rtx®-G43 stable to 240 °C.

Some USP <467> methods require the use of a guard column. Our Integra-Guard® integrated guard column system makes it easy to comply.

			30-Meter with 5-Meter, 0.53mm ID Integra-Guard Guard Column
ID	**df**	**temp. limits**	**cat.#**
0.53 mm	3.00 µm	-20 to 240 °C	16085-126

USP <467> Residual Solvents on Rtx®-1301 (G43) by Static Headspace



Peaks	tR (min)	Conc. (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

Column	Rtx®-1301 w/5 m Integra-Guard®, 30 m, 0.53 mm ID, 3.00 µm (cat.# 16085-126)
Sample	USP <467> calibration mixture #5 (cat.# 36007)
Diluent:	DMSO
Conc.:	To each 22 mL headspace vial 5ml water, ~ 1.0 g of sodium sulfate and 100 µL of stock standard were added.
Injection	headspace-loop split (split ratio 2:1)
Headspace-Loop	
Inj. Port Temp.:	180 °C
Instrument:	Teledyne Tekmar HT3
Inj. Time:	1.0 min
Transfer Line Temp.:	150 °C
Valve Oven Temp.:	150 °C
Standby flow rate:	10 mL/min
Sample Temp.:	80 °C
Platen temp equil. time:	2.0 min
Sample Equil. Time:	15.0 min
Mixer time:	2.0 min
Mixing level:	5
Mixer stabilize time:	0.5 min
Vial Pressure:	15 psi
Pressurize Time:	2.0 min
Pressure Equilibration Time:	0.5 min
Loop Pressure:	5 psi
Loop Fill Time:	2.0 min
Loop fill equil. time:	0.5 min
Oven	
Oven Temp.:	40 °C (hold 20 min) to 240 °C at 25 °C/min (hold 10 min)
Carrier Gas	He, constant flow
Flow Rate:	5 mL/min
Detector	FID @ 250 °C
Make-up Gas Flow Rate:	45 mL/min
Notes	**FID conditions:** hydrogen flow: 40 mL/min air flow: 450 mL/min