

Organic Volatile Impurities (OVI) Analysis



Rick Morehead
Marketing Specialist
17+ years of service!

Rtx®-G27 (Crossbond® 5% diphenyl/95% dimethyl polysiloxane)
with Integra-Guard™ Guard Column

Rtx®-G43 (Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)
with Integra-Guard™ Guard Column

- 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®-G27 stable to 290°C; 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.

please **note**

Analytical Reference Materials for USP <467> are available on **page 510**.

Rtx®-G27 Column (fused silica with 5-meter Integra-Guard™ guard column)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

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

Rtx®-G43 Column (fused silica with 5-meter Integra-Guard™ guard column)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

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

free literature

A Technical Guide for Static Headspace Analysis Using GC

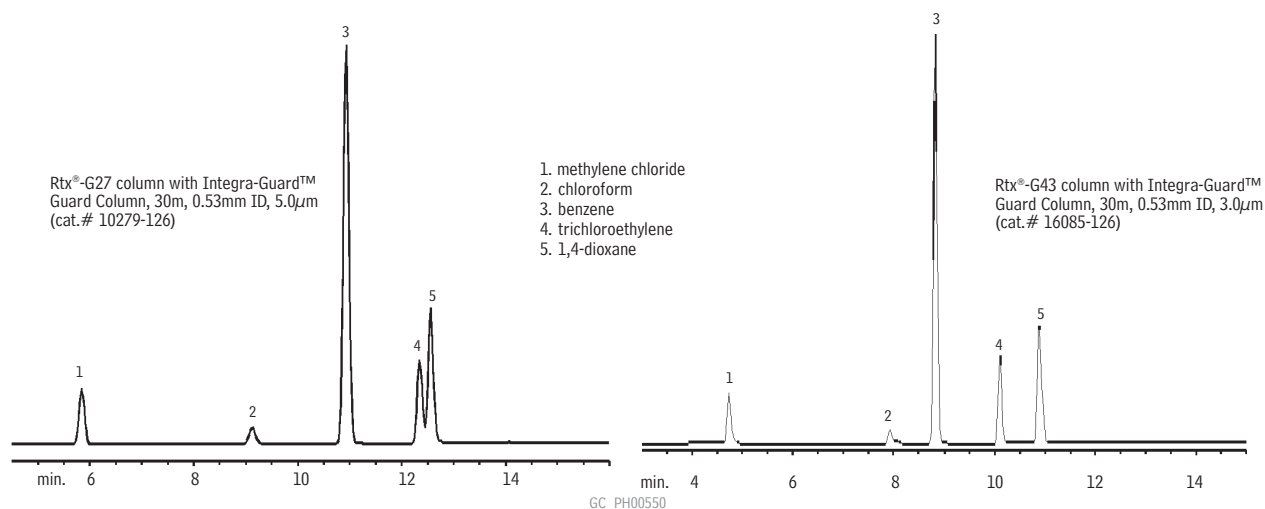
Download your free copy from www.restek.com.

Technical Guide
lit. cat.# 59895A

Searching for a chromatogram?

www.restek.com

Specially-designed Rtx®-G27 and Rtx®-G43 columns resolve residual solvents in USP <467>.



Inj.: 1.0µL direct injection of USP <467> Mix #1 (cat.# 36004)
Oven temp.: 35°C (hold 5 min.) to 175°C @ 8°C/min. to 260°C @ 35°C/min.
Inj./det. temp.: 200°C/240°C
Carrier gas: helium
Linear velocity: 34cm/sec. set @ 35°C
FID sensitivity: 1 x 10⁻¹¹ AFS
Recommended liner: Uniliner® inlet liner

Organic Volatile Impurities (OVI) Analysis

Rtx®-1301/Rtx®-624 (low to midpolarity phase; Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

- General purpose columns for residual solvents, alcohols, oxygenates, and volatile organic compounds.
- Temperature range: -20°C to 240°C.
- Equivalent to USP G43 phase.

Many analysts feel the Rtx®-1301/Rtx®-624 column has the best cyanosilicone bonded stationary phase available, with no other column manufacturer providing lower bleed, longer lifetime, or better inertness. Our polymer is fully characterized to ensure long-term reproducibility, column-to-column consistency, and low bleed—even with sensitive detectors such as ECDs and MSDs.

Rtx®-1301 (G43) Columns (fused silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter	75-Meter	105-Meter
0.53mm	3.00	-20 to 240°C	16082	16085	16088	16076	16091

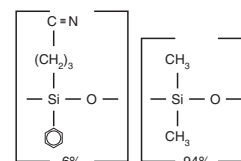
Rtx®-624 Columns (fused silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.32mm	1.80	-20 to 240°C	10970	10972

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Rtx®-1301/Rtx®-624 Structure



similar **phases**

DB-1301, DB-624, HP-1301, HP-624, SPB-1301, SPB-624, VF-1301, VF-624ms, CP-1301, CP-Select 624 CB

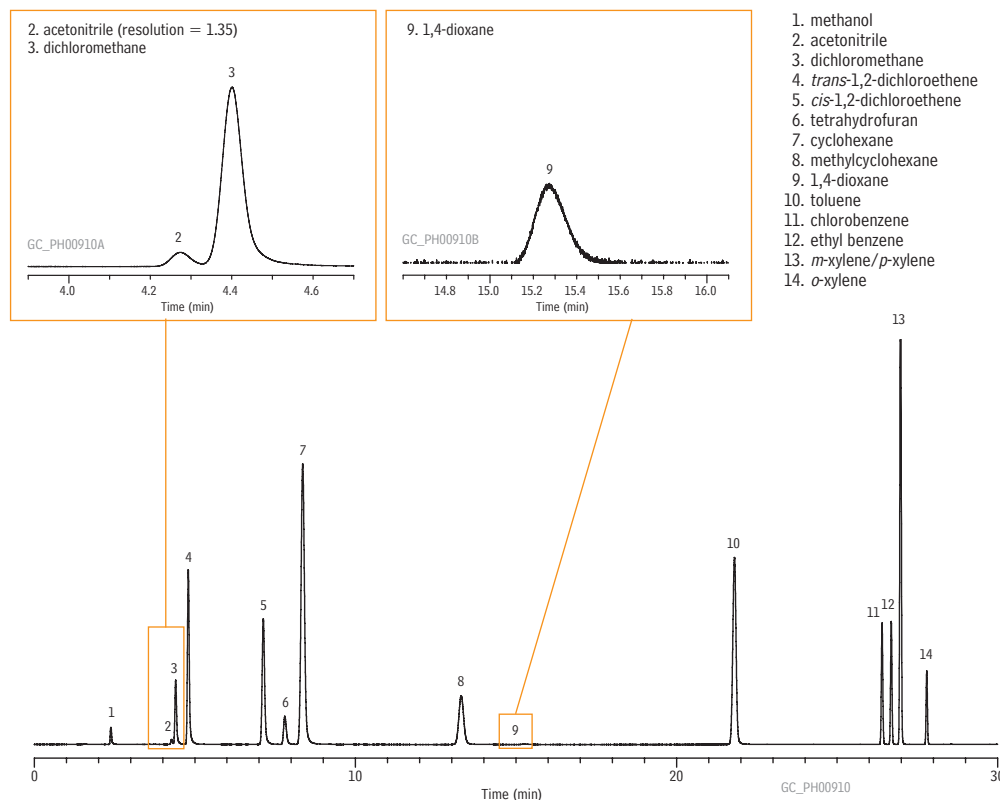
please **note**

Rtx®-1301 and Rtx®-624 columns are available with Integra-Guard™ built-in guard columns. Get the protection without the connection! See **page 30** for descriptions and ordering information.

also **available**

MXT® Columns
Rugged, flexible, Siltek® treated stainless steel tubing; inertness comparable to fused silica tubing. See **pages 105 and 107** for our MXT®-1301 and MXT®-624 columns.

USP Residual Solvent Class 2 Mixture A standard solution on an Rtx®-624 column.

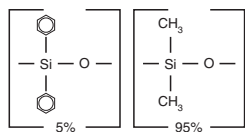


Column: Rtx®-624, 30m, 0.32 ID, 1.8µm (cat. # 10970)
Sample: USP <467> Class 2 Mixture A Standard Solution (cat.# 36271) in 20mL headspace vial
Inj.: Headspace injection (split ratio 1:5), 1mm Split liner Siltek® deactivated (cat.# 20972-214.1)
Inj. temp.: 140°C
Carrier gas: helium, constant flow
Flow rate: 2.16mL/min., 35.3cm/sec.
Oven temp.: 40°C for 20 min. to 240°C @ 10°C/min. (hold for 20 min.)
Det.: FID @ 240°C

Headspace Conditions
Instrument: Tekmar HT3
Transfer Line Temp.: 105°C
Valve Oven Temp.: 105°C
Sample Temp.: 80°C
Sample Equil. Time: 45 min.
Vial Pressure: 10psi
Pressurize Time: 0.5 min.
Loop Fill Pressure: 5psi
Loop Fill Time: 2.00 min.
Inject Time: 1.00 min.

See our OVI Retention Index on page 721.

Organic Volatile Impurities (OVI) Analysis

Rtx®-5
Structure**Rtx®-5** (low polarity phase; Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

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

The 5% diphenyl/95% 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.

similar phases

DB-5, HP-5, HP-5MS, Ultra-2, SPB-5, Equity-5, MDN-5

Rtx®-5 Columns (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.53mm	5.00	-60 to 270/290°C	10277	10279	10283

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

also available

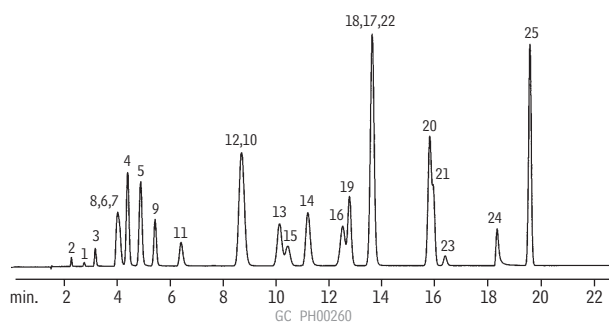
MXT® Columns

Rugged, flexible, Siltek® treated stainless steel tubing; inertness comparable to fused silica tubing. See page 102 for our MXT®-5 columns.

it's a fact

For exceptional inertness, ultra-low bleed, and unsurpassed performance, choose Rxi®-5ms columns! See pages 33-35, 37.

Organic volatile impurities on an Rtx®-5 (Rtx®-G27) column.



Rtx®-5 (Rtx®-G27) with 5m phenylmethyl Integra-Guard™ guard column, 30m, 0.53mm ID, 5.0µm (cat. # 10279-126)

Inj.: Headspace injection of common solvents for pharmaceutical processing. Prepared to equal about 500ppm in the bulk pharmaceutical. Samples shaken and heated at 90°C for 15 minutes, 1mL headspace injection.

Oven temp.: 35°C (hold 10 min.) to 100°C @ 5°C/min., to 240°C @ 25°C/min. (hold 5 min.)

Inj./det. temp.: 220°C/240°C

FID sensitivity: 1.05 x 10⁻¹¹ AFS

Carrier gas: helium, 35cm/sec. set @ 35°C

Split ratio: 2:1

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

See our OVI Retention Index on page 721.

Organic Volatile Impurities (OVI) Analysis

Stabilwax® (polar phase; Crossbond® Carbowax® polyethylene glycol)

- General purpose columns for FAMES, flavor compounds, essential oils, amines, solvents, xylene isomers, and US EPA Method 603 (acrolein/acrylonitrile).
- Resistant to oxidative damage.
- Temperature range: 40°C to 250°C.
- Equivalent to USP G14, G15, G16, G20, and G39 phases.

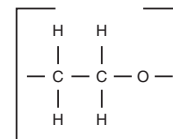
Our polar-deactivated surface tightly binds the Carbowax® polymer and increases thermal stability, relative to competitive columns. The bonding mechanisms produce a column that can be rejuvenated by solvent washing. Compared to silicone stationary phases, PEG phases are more resistant to damage from strongly acidic or basic volatile compounds, including inorganic acids and volatile inorganic bases.

Stabilwax® Columns (fused silica)

(Crossbond® Carbowax® polyethylene glycol)

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.32mm	0.25	40 to 250°C	10621	10624	10627
0.53mm	0.25	40 to 250°C	10622	10625	10628

Stabilwax® Structure



similar phases

DB-WAX, DB-WAXetr,
HP-Wax, HP-Innowax,
Supelcowax 10

ordering note

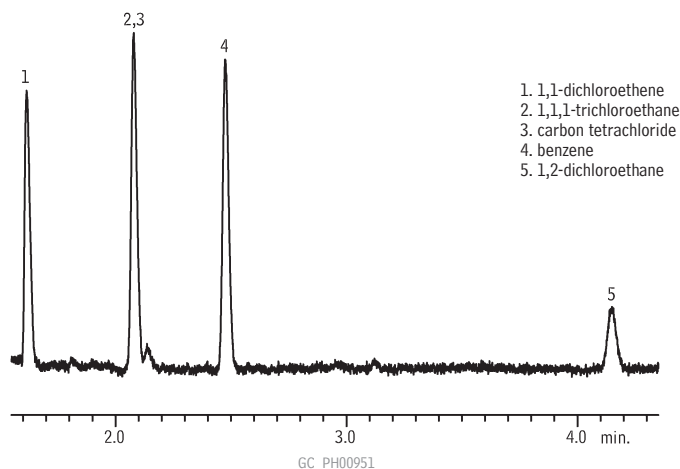
Stabilwax® columns are available with Integra-Guard™ built-in guard columns. Get the protection without the connection! See [page 30](#) for descriptions and ordering information.

also available

MXT® Columns

Rugged, flexible, Siltek® treated stainless steel tubing; inertness comparable to fused silica tubing. See [page 106](#) for our MXT®-WAX columns.

Residual solvents class 1 on a Stabilwax® (G16) column.



- 1,1-dichloroethene
- 1,1,1-trichloroethane
- carbon tetrachloride
- benzene
- 1,2-dichloroethane

Column:	Stabilwax®, 30m, 0.32mm ID, 0.25µm (cat. # 10624)	Headspace Conditions
Sample:	USP Stock Mixture USP<467> Residual Solvents Class 1 Mix (cat. # 36279) in 20mL headspace vial (cat. # 24685), water diluent	Instrument: Overbrook Scientific HT200H
Inj.:	headspace injection (split ratio 1:5), 2mm splitless liner IP deactivated (cat. # 20712)	Syringe temp.: 100°C
Inj. temp.:	140°C	Sample temp.: 80°C
Carrier gas:	helium, constant flow	Sample equil. time.: 45 min.
Flow rate:	2.15mL/min., 35.2cm/sec.	Injection vol.: 1.0mL
Oven temp.:	50°C for 20 min., to 165°C @ 6°C/min. (hold for 20 min.)	Injection speed: setting 8
Det.:	FID @ 250°C	Injection dwell: 5 sec.