

please note

For superior inertness, try our Siltek® guard columns!

See **page 28** for details.

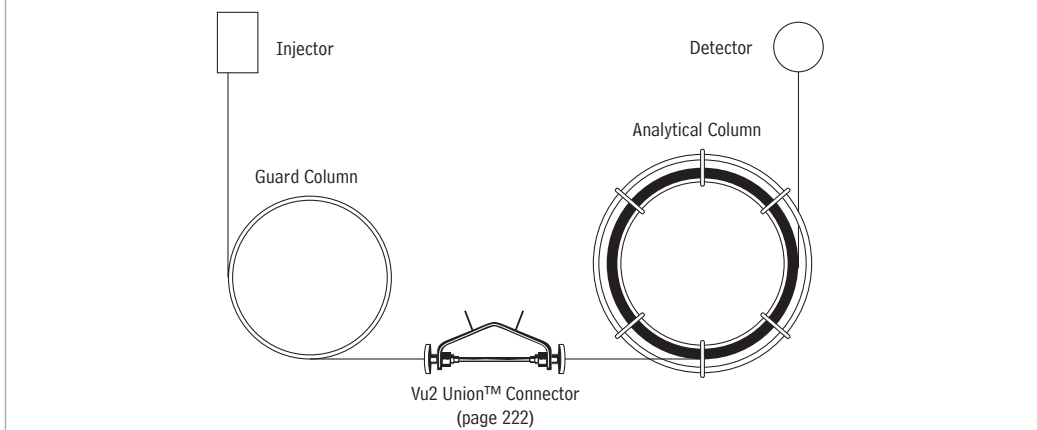
Having trouble making a leak-free connection? Try our "built in" Integra-Guard™ columns!

See **page 30** for details.

Guard Columns and Retention Gaps

Guard columns and retention gaps are widely used in gas chromatography. The concept of the guard column is to trap nonvolatile material at the head of the column, not allowing the material to reach the analytical column. The concept of the retention gap is to help focus the compounds transferred from the inlet to a small band at the head of the analytical column in order to reduce chromatographic peak broadening. Both concepts (trapping nonvolatile material and refocusing the target analytes) take place when a piece of deactivated tubing is connected to an analytical column as in Figure 1.

Figure 1 A guard column connected to an analytical column



Analyte Focusing

There are two injection techniques where the retention gap is used to help focus target analytes at the beginning of the analytical column, cool on-column injection and splitless injection.

For cool on-column injection, the use of a retention gap is to help focus the sample components when introducing a liquid sample directly into the retention gap. The cool on-column injection is performed by inserting the syringe needle into the retention gap (this can be accomplished with a 0.53mm ID retention gap and a 26s gauge syringe) and transferring the liquid sample directly into the retention gap. The injection is made with the injector and column oven set below the boiling point of the solvent. As the solvent is evaporated, the volatile target analytes migrate in the solvent towards the analytical column, and the heavier analytes will be distributed over the retention gap. As the oven temperature increases, the target analytes vaporize and move unretained down the retention gap column until the compounds reach the liquid stationary phase of the analytical column. At this juncture, the target analytes are trapped/focused by the liquid phase forming a narrow injection band.

did you know?

We test our guard columns/transfer lines with the Grob test mix to ensure high inertness.

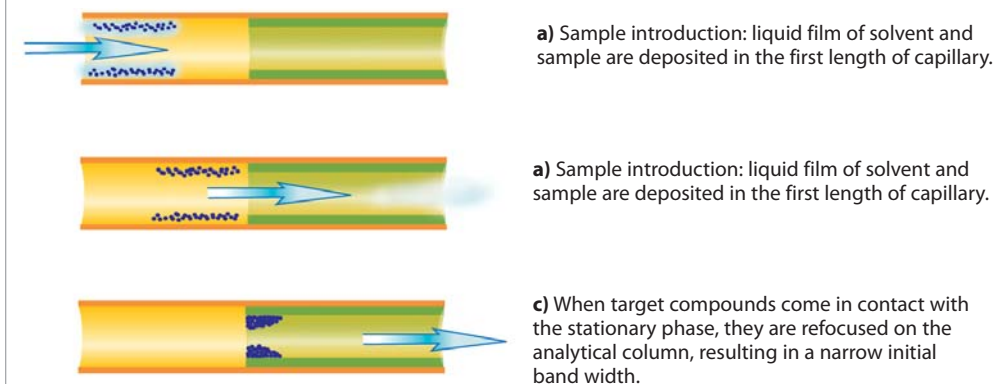
The retention gap may also be useful in hot vaporization injections when the transfer of the compounds from the inlet to the column does not form a focused band. Typical applications include water injections or injections using small ID columns, where split or tailing peaks would indicate an unfocused band. In these applications, the target analytes are trapped in a nonuniform or longitudinally diffuse band at the head of the retention gap (Figure 2a). As the oven temperature is increased, the solvent and target compounds are vaporized and move unretained through the retention gap (Figure 2b). When the target compounds come in contact with the stationary phase, they are refocused in a narrow band (Figure 2c), improving the chromatography.

Protecting the Analytical Column

The concept of a guard column is to protect the analytical column from becoming contaminated with nonvolatile compounds. The guard column is used to retain nonvolatile material, usually in the first 10-20cm, not allowing this material to elute onto the liquid phase of the analytical column. As the oven temperature increases, the more volatile target compounds vaporize, elute down the guard column, and refocus at the head of the analytical column without interference from the nonvolatile material.

Contamination can cause active sites as well as change the conditions of the focusing zone of the analytical column. Both conditions will adversely affect the chromatography. Another advantage of the guard column is when a section is trimmed for maintenance the resolution of closely eluting compounds will not be affected because the guard column is not a contributor to the resolving power of the analytical column. This allows for a longer lifetime of the analytical column, and replacing only the guard column when it becomes too short.

Figure 2 Retention gaps are used to focus components in a tight band at the beginning of the analytical column



In summary, the retention gap and guard column are essentially the same products, but are used for different purposes. The deactivated tubing helps focus target analytes at the head of the analytical column for on-column and splitless injections, and also prevents nonvolatile material from contaminating the head of the analytical column.

What type of guard column should be used?

When using a guard column, it is important to match the polarity of the solvent and the polarity of the surface deactivation. Intermediate-Polarity (IP) is good for a wide variety of applications and allows most common solvents (methylene chloride, hexane, isooctane, toluene) to easily wet and create a uniform film on the tubing surface. If more polar solvents such as methanol or water are used, a polar-deactivated guard column is recommended to allow the solvent to wet the tubing surface. Polar-deactivated guard columns are not resistant to harsh "water vaporization" that occurs when water in the liquid state is injected into the tubing and rapidly vaporizes (such as in steam cleaning). Hydroguard™ deactivation is an alternative for direct aqueous injections. However, a Hydroguard™-deactivated guard column will not allow polar solvents to wet the tubing surface, and may cause beading of the solvent if the oven temperature is 20°C below the solvent boiling point.

Siltek® deactivation creates a highly inert surface for very active compounds such as chlorinated pesticides. Base-deactivated guard columns reduce adsorption and tailing for amines and other basic compounds.

How is a guard column connected to the analytical column?

We offer Vu2-Union™, Press-Tight®, and other connectors for attaching guard columns to fused silica columns. MXT™ unions are available for connecting stainless steel MXT® columns and guard columns. See pages 224 to 227 for information about these connectors.



AJ Saclyn
Associate Product
Marketing Manager

it's a fact

To eliminate connections, use our unique Integra-Guard™ Column. See [page 30](#).

Guard/Retention Gap Columns

it's a fact

To eliminate connections, use an Integra-Guard™ Column. See page 30.

also available

MXT® Guard/Retention Gap Columns

Rugged, flexible, Siltek® treated stainless steel tubing; inertness comparable to fused silica tubing. See page 100 for our Intermediate-Polarity Deactivated MXT® Guard/Retention Gap Columns/Transfer Lines.

it's a fact

Use guard columns to:

- Reduce effects of dirty samples on column performance.
- Reduce downtime and maintenance.

Rxi® Guard/Retention Gap Columns (Fused Silica)

- Extend column lifetime.
- Excellent inertness—obtain lower detection limits for active compounds.
- Sharper chromatographic peaks by utilizing retention gap technology.
- Maximum temperature: 360°C.

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter	10-Meter/6-pk.
0.25mm	0.37 ± 0.04mm	10029	10029-600	10059	10059-600
0.32mm	0.45 ± 0.04mm	10039	10039-600	10064	10064-600
0.53mm	0.69 ± 0.05mm	10054	10054-600	10073	10073-600

Intermediate-Polarity Deactivated Guard/Retention Gap Columns/Transfer Lines (Fused Silica)

Diameters greater than 0.10mm are tested with the Grob test mix, to ensure high inertness.

- Useful for a wide range of applications.
- Use with most common solvents.
- Maximum temperature: 325°C

Nominal ID	Nominal OD	1-Meter	5-Meter	5-Meter/6-pk.
0.025mm*	0.363 ± 0.012mm	10097		
0.05mm*	0.363 ± 0.012mm	10098	10040	10040-600
0.075mm*	0.363 ± 0.012mm	10099		
0.10mm*	0.363 ± 0.012mm	10100	10041	
0.15mm	0.363 ± 0.012mm	10101	10042	
0.18mm	0.37 ± 0.04mm	10102	10046	
0.25mm	0.37 ± 0.04mm		10043	10043-600
0.28mm	0.37 ± 0.04mm		10003	10003-600
0.32mm	0.45 ± 0.04mm		10044	10044-600
0.45mm	0.69 ± 0.04mm		10005	10005-600
0.53mm	0.69 ± 0.05mm		10045	10045-600

Nominal ID	Nominal OD	10-Meter	10-Meter/6-pk.	30-Meter**	60-Meter**†
0.25mm	0.37 ± 0.04mm	10049	10049-600	10012	10013
0.32mm	0.45 ± 0.04mm	10048	10048-600	10022	10023
0.53mm	0.69 ± 0.05mm	10047		10032	10033

Siltek®-Deactivated Guard/Retention Gap Columns/Transfer Lines (Fused Silica)

Tested with the Grob test mix, to ensure high inertness.

- Revolutionary deactivation process for superior inertness.
- Minimize bleed.
- Analyze active samples accurately; ideal for chlorinated pesticide analysis (reduces endrin breakdown to less than 1%).
- Maximum temperature: 380°C.

Nominal ID	Nominal OD	5-Meter	10-Meter
0.25mm	0.37 ± 0.04mm	10026	10036
0.32mm	0.45 ± 0.04mm	10027	10037

Polar-Deactivated Guard/Retention Gap Columns (Fused Silica)

Tested with the Grob test mix, to ensure high inertness.

- Polyethylene glycol deactivation layer provides optimum wettability for polar compounds.
- Minimize peak splitting when using polar solvents such as methanol or water.
- Compatible with Stabilwax®, Rtx®-225, and Rt™-2330 capillary columns.
- Maximum temperature: 280°C.

Nominal ID	Nominal OD	5-Meter	10-Meter	30-Meter**	60-Meter**†
0.25mm	0.37 ± 0.04mm	10065	10068	10014	10015
0.32mm	0.45 ± 0.04mm	10066	10069	10024	10025
0.53mm	0.69 ± 0.05mm	10067	10070	10034	10035

*Not tested with the Grob test mix because of a large pressure drop.

**30- and 60-meter lengths are banded in 5-meter sections.

†Recommendation: Cut 60m guard columns into shorter lengths. Using full length may cause peak distortion.



did you know?

Siltek®-deactivated guard columns minimize breakdown and improve recovery of analytes!

Base-Deactivated Guard/Retention Gap Columns (Fused Silica)

- Tested with a basic amine test mix.
- Excellent inertness for basic compounds.
- Recommended for use with Rtx®-5 Amine, Rtx®-35 Amine, and Stabilwax®-DB capillary columns.
- Batch test chromatogram included.
- Maximum temperature: 315°C.

Chemists using guard columns in analyses of basic compounds frequently observe peak tailing and low recovery. This happens because conventionally deactivated tubing surfaces can be adsorptive to basic compounds. Restek offers base-deactivated guard columns for completely inert sample pathways.

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.
0.25mm	0.37 ± 0.04mm	10000	10000-600
0.32mm	0.45 ± 0.04mm	10001	10001-600
0.53mm	0.69 ± 0.05mm	10002	10002-600



Chris English
Innovations Team
Manager
11+ years of service!

Hydroguard™ Water-Resistant Guard/Retention Gap Tubing/Transfer Lines (Fused Silica)

Diameters greater than 0.10mm are tested with the Grob test mix to ensure high inertness.

- Extend analytical column lifetime by preventing degradation by harsh “steam-cleaning” water injections.
- Maximum temperature: 325°C.

When transfer lines from purge & trap systems, air monitoring equipment, or other instruments carry condensed water vapor, deactivated column tubing quickly becomes active because of the creation of free silanol groups. These silanol groups adsorb active oxygenated compounds such as alcohols and diols.

Restek chemists have addressed this concern and found a solution—the Hydroguard™ deactivation process. A unique deactivation chemistry creates a high-density surface that is not readily attacked by aggressive hydrolysis. The high-density surface coverage of the Hydroguard™ deactivation layer effectively prevents water vapor from reaching the fused silica surface beneath. Use Hydroguard™ tubing for connecting GCs to:

- Purge & trap systems.
- Headspace analyzers.
- Air analysis equipment and concentrator units.

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter	30-Meter**	60-Meter**†
0.05mm*	0.363 ± 0.012mm	10075				
0.10mm*	0.363 ± 0.012mm	10076				
0.15mm	0.363 ± 0.012mm	10077				
0.18mm	0.37 ± 0.04mm	10078				
0.25mm	0.37 ± 0.04mm	10079	10079-600	10082	10085	10088
0.32mm	0.45 ± 0.04mm	10080	10080-600	10083	10086	10089
0.53mm	0.69 ± 0.05mm	10081	10081-600	10084	10087	10090

*Not tested with the Grob test mix because of a large pressure drop.

**30- and 60-meter lengths are banded in 5-meter sections.

†Recommendation: Cut 60m guard columns into shorter lengths. Using full length may cause peak distortion.

did you know?

We test our guard columns/transfer lines with the Grob test mix to ensure high inertness.

also available

MXT® Guard Columns
Rugged, flexible, Siltek® treated stainless steel tubing; inertness comparable to fused silica tubing. See **page 100** for our Hydroguard™ MXT® Guard Tubing/Transfer Lines.

best choice

Siltek® treated tubing (cat.# 22505, page 394) is recommended for purge and trap transfer lines.

Guard Columns: Integra-Guard™ Columns

restek innovation!

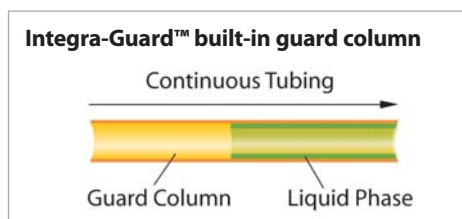
Integra-Guard™ Columns: guard columns WITHOUT connections—protecting your analytical column has never been this easy!

Innovative Integra-Guard™ Columns

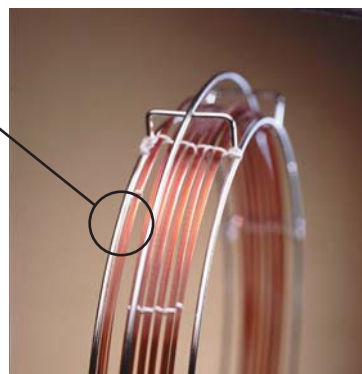
For analysts who find it inconvenient to make a leak-free connection between the guard column and the analytical column, we offer Integra-Guard™ columns. These innovative columns incorporate both guard column and analytical column in a continuous length of tubing, eliminating the connection and all connection-associated problems! The guard column section is tied separately from the analytical column, using high-temperature string.

Our wide variety of Integra-Guard™ capillary columns are listed in the figure below. The Integra-Guard™ column is so economical that we challenge you to compare our price against that of a conventional connection, even if you assemble it yourself. If you are currently using a guard column, or are considering using one, call today and ask about Integra-Guard™ columns.

Ordering is simple. Just add the appropriate suffix number and price to the analytical column's catalog number and price. For example, a 30m, 0.25mm ID, 0.25µm Rtx®-5 column with a 5-meter Integra-Guard™ column is cat.# 10223-124.



Phases currently available as Integra-Guard™ columns



Rtx®-1
Rtx®-1MS
Rtx®-5
Rtx®-5MS
Rxi®-5Sil MS
Rtx®-1301
Rtx®-624
Rtx®-1701
Rtx®-Volatiles
Rtx®-20
Rtx®-35
Rtx®-35MS
Rtx®-BAC 1 & 2
Stabilwax®

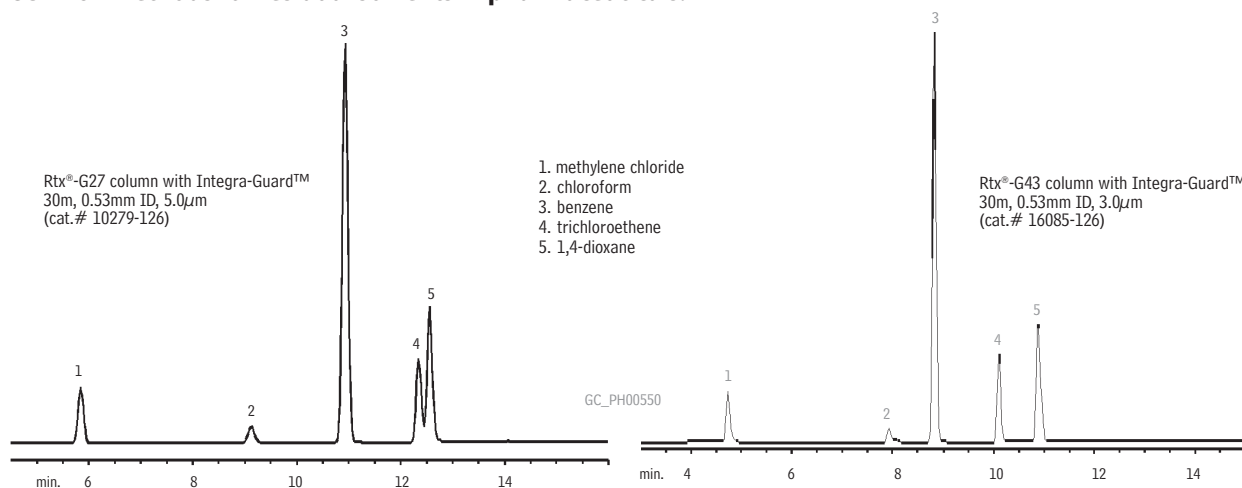
Integra-Guard™ columns are available for all phases listed, for columns with 0.25 to 0.53mm ID and lengths to 75 meters.

ID	Length	Suffix #
0.25mm	5m	-124
	10m	-127
0.28mm	5m	-243
	10m	-244
0.32mm	5m	-125
	10m	-128
0.53mm	5m	-126
	10m	-129

similar products

DuraGuard, EZ-Guard,
Guardian

An Rtx®-G27 or Rtx®-G43 column with a 5-meter Integra-Guard™ column meets the requirements of USP 467 methods for residual solvents in pharmaceuticals.



Inj. 1.0µL direct injection of USP <467> Mix #3, 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®