<b>7</b> _/		lumns

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Foods, Flavors, & Fragrances

**Environmental** 

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At Restek, we are dedicated to your success.

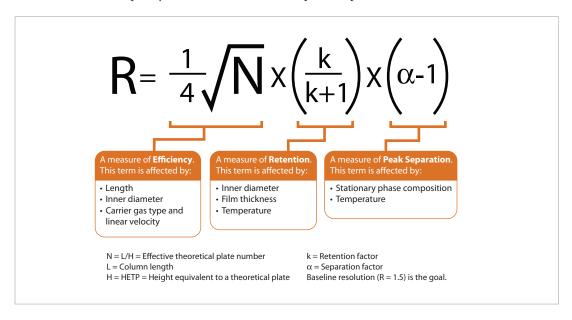
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### Selecting a GC Column

Strategic column choices can improve lab productivity by assuring that speed and resolution are optimized. While the number of choices available can be daunting, consideration of the resolution equation variables—separation factor, retention (capacity) factor, and efficiency—simplifies the decision. Separation factor determines which stationary phase is most appropriate. Once the phase has been chosen, physical dimensions (inner diameter, film thickness, length) can be selected based on retention factor and efficiency. Understanding how separation factor, retention factor, and efficiency influence separations allows analysts to make effective, informed choices and quickly select the best column for specific separations.







#### Separation Factor (α)

Choosing the right stationary phase is the first step toward optimizing your GC separation. It is the most important decision you will make because separation factor  $(\alpha)$  has the greatest impact on resolution, and it is strongly affected by stationary phase polarity and selectivity.

Stationary phase polarity is determined by the type and amount of functional groups in the stationary phase. Structures for Restek stationary phases are presented in order of polarity on page 15. When choosing a column, consider the polarity of both the stationary phase and your target analytes. If the stationary phase and analyte polarities are similar, then the attractive forces are strong and more retention will result. Greater retention often results in increased resolution. Stationary phase polarity strongly influences column selectivity and separation factor, making it a useful consideration when selecting a column.

Stationary phase selectivity is defined by IUPAC as the extent to which other substances interfere with the determination of a given substance. Selectivity is directly related to stationary phase composition and how it interacts with target compounds through intermolecular forces (e.g., hydrogen bonding, dispersion, dipole-dipole interactions, and shape selectivity). As methyl groups in the stationary phase are replaced by different functionalities, such as phenyl or cyanopropyl pendant groups, compounds that are more soluble with those functional groups (e.g., aromatics or polar compounds, respectively) will interact more and be retained longer, often leading to better resolution and increased selectivity. In another example of the effect of stationary phase-analyte interactions, an Rtx\*-200 stationary phase is highly selective for analytes containing lone pair electrons, such as halogen, nitrogen, or carbonyl groups, due to interactions with the fluorine pendant group in this phase. Selectivity can be approximated using existing applications or retention indices (Table I), making these useful tools for comparing phases and deciding which is most appropriate for a specific analysis.

Phase	Benzene	Butanol	Pentanone	Nitropropane
Rtx-1	651	651	667	705
Rtx-5/Rtx-5MS	667	667	689	743
Rtx-20	711	704	740	820
Rtx-1301/Rtx-624	689	729	739	816
Rtx-35	746	733	773	867
Rtx-200	738	758	884	980
Rtx-50	778	769	813	921
Rtx-1701	721	778	784	881
Rtx-65TG	794	779	825	938
Rtx-225	847	937	958	958
Stabilwax	963	1.158	998	1.230

Stationary phase polarity and selectivity also affect how much sample loading capacity the column will have for a particular analyte; the more soluble an analyte is in the stationary phase, the greater the sample loading capacity will be for that analyte. For example, a nonpolar stationary phase will have higher sample loading capacity for a nonpolar compound (e.g., pentane) than for a polar compound (e.g., ethanol).

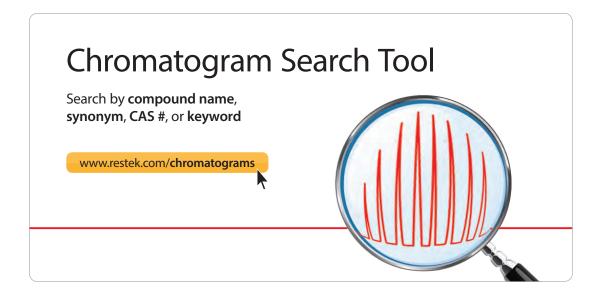


The relationship between polarity, selectivity, and sample loading capacity can be illustrated using an example. Consider the analysis of benzene and butanol, which have nearly the same boiling point, on an Rtx\*-20 column (diphenyl dimethyl polysiloxane stationary phase). Since the benzene molecule is structurally more similar to the diphenyl phase than butanol is, benzene will solvate into the stationary phase more readily than butanol based on the concept that "like dissolves like." Since benzene solvates more readily with the stationary phase, it has more interaction with the stationary phase as it elutes through the column and will be retained longer. Since butanol solvates less with the stationary phase, it has fewer interactions with the stationary phase and less will be retained. Therefore, the elution order of these two compounds on an Rtx\*-20 column will be butanol first and benzene second. In addition, since benzene is more soluble in the diphenyl phase, the column has more capacity for benzene. This results in a more symmetrical peak shape for benzene than for butanol. A more polar column, such as a polyethylene glycol (PEG) column, will provide retention and better peak shape for butanol compared to benzene.

Due to their influence on separation factor, polarity and selectivity are primary considerations when selecting a column. However, temperature limits must also be considered. In general, highly polar stationary phases have lower maximum operating temperatures, so choosing a column with the appropriate maximum operating temperature, as well as optimal polarity and selectivity for the type of compounds being analyzed, is crucial.

#### **Retention Factor (k)**

The retention factor (k) of a column is based on the time an analyte spends in the stationary phase relative to the time it spends in the carrier gas. It is influenced primarily by column inner diameter (ID), phase film thickness, and temperature. Retention factor is sometimes referred to as capacity factor, which should not be confused with sample loading capacity. As a general rule, the thicker the film and the smaller the inner diameter, the more an analyte will be retained. Note that as temperature increases, k decreases; therefore, at higher temperatures analytes stay in the carrier gas longer and are less retained.





When selecting column ID, consider the type of injection, the detector being used, and the concentration of sample (amount on-column). The injection technique is important because the column ID may need to be selected based on whether a split, splitless, direct, cool on-column injection, or other sample transfer method is being used. For example, 0.53 mm ID columns are ideal for cool on-column injections since the syringe needle (26 gauge) will fit into the large column ID. In addition to column ID, the detector and its flow requirements must be considered. For example, some MS detectors can only operate under column flow rates of up to 1.5 mL/min; therefore, a 0.53 mm ID column, which requires higher flows for proper chromatography, is not an option for MS work. Table II shows typical column characteristics for columns of various inner diameters.

Table II:	General	Column	Characte	rictics	Rased o	n ID
I a vie II.	General	COMMITTEE	CHALACTE	115116.5	Daseu	11 11 1

	Column Inner Diameter (mm)							
Characteristic	0.10	0.15	0.18	0.25	0.32	0.53		
Nitrogen flow (mL/min)	0.2	0.3	0.3	0.4	0.6	0.9		
Helium flow (mL/min)	0.6	0.8	1.0	1.4	1.8	3.0		
Hydrogen flow (mL/min)	0.7	1.1	1.3	1.8	2.3	3.7		
Sample loading capacity (ng)	2.5	10	20	50	125	500		
Theoretical plates/meter	11,000	7,000	6,000	4,000	3,000	2,000		

Note: Flows listed are for maximum efficiency. Sample loading capacities are estimates only. Actual sample loading capacity varies with film thickness and analyte.

Film thickness has a direct effect on the retention and elution temperature for each sample component. Extremely volatile compounds should be analyzed on thick film columns to increase the time the compounds spend in the stationary phase, which allows them to better separate. High molecular weight compounds must be analyzed on thinner film columns. This reduces the length of time that the analytes stay in the column and minimizes phase bleed at higher elution temperatures. Film thickness also affects the amount of material that can be injected onto the column without overloading it. A thicker film column can be used for higher concentration samples.

## **Technical Service**

For quick answers to commonly asked questions any time of the day, visit **www.restek.com/answers** or contact us directly:

#### In the U.S.

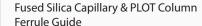
Phone: 1-800-356-1688, ext. 4 e-mail: support@restek.com

**Hours of operation (Eastern Time):** Monday - Thursday, 8:00 a.m. to 6:00 p.m.

Friday, 8:00 a.m. to 5:00 p.m.

#### Outside the U.S.

Contact International Technical Service at intltechsupp@restek.com or find a local distributor at www.restek.com/distributor



GC Column ID	Ferrule ID
0.15 mm	0.4
0.18 mm	0.4
0.25 mm	0.4
0.32 mm	0.5
0.53 mm	0.8





Checking for leaks, using a Restek® electronic leak detector, is an easy way to protect your instrument and column from damage.

Film thickness also directly affects phase ratio  $(\beta)$ , which must be accounted for when changing to a column with a different inner diameter. When inner diameter increases, film thickness  $(d_f)$  must also increase in order to provide comparable resolution and retention. Table III shows  $\beta$  values for common column dimensions; similar values indicate similar separations on different ID columns.

**Table III:** Phase Ratio (β) Values for Common Column Dimensions\*

	Film Thickness (dr) / β Value								
Column ID	0.10 µm	0.25 μm	0.50 µm	1.0 µm	1.5 µm	3.0 µm	5.0 µm		
0.18 mm	450	180	90	45	30	15	9		
0.25 mm	625	250	125	63	42	21	13		
0.32 mm	800	320	160	80	53	27	16		
0.53 mm	1325	530	265	128	88	43	27		

#### Efficiency (N)

Column efficiency (N) is the column length divided by the height equivalent of a theoretical plate (HETP). The effective number of theoretical plates is affected by how well the phase has been coated onto the column walls, and it is measured by how narrow the peaks are when they elute out of the column. Higher column efficiency (N) results in greater resolution between peaks. Inner diameter also influences efficiency; a simple rule of thumb is the smaller the column ID, the more efficient the column.

Capillary columns are made in various lengths, typically 10, 15, 30, 60, and 105 meters. Longer columns provide more resolving power, but will also increase analysis time and cost more. When column length is doubled, analysis time will increase by as much as a factor of two. However, doubling the column length increases resolution by only approximately 40% since the column length term is under the square root function in the resolution equation. When selecting column length, the increase in resolution obtained in a longer column must be weighed against the increase in cost and analysis time.

#### Conclusion

A basic understanding of the resolution equation allows analysts to make more effective column choices. Phase choice should be influenced primarily by separation factor, which can be approximated by considering the structures of both the phase and the analyte, as well as by referencing retention indices or existing applications. Retention factor and efficiency also affect peak separations and should be considered when choosing column inner diameter, film thickness, and length. By better understanding these factors, analysts can simplify the column selection process, optimize separations, and increase lab productivity.

#### What Do the Temperature Limits Mean?

All Restek columns have published minimum and maximum operating temperatures that establish the working range for the stationary phase. Note that these ranges vary with the thickness of the coating.

Rxi®-5Sil MS Columns (fused silica)								
ID	df (µm)	temp. limits						
0.25 mm	0.25	-60 to 320/350 °C						
0.32 mm	0.50	-60 to 320/350 °C						
0.53 mm	1.50	-60 to 320/330 °C						

The second temperature is the maximum temperature-programmed operating temperature, the temperature to which the column can be heated for short periods of time (i.e., during a temperature-programmed analysis). If only one temperature is listed, it is both the isothermal and the maximum temperature.

The minimum operating temperature defines the lowest usable temperature before the stationary phase solidifies. Operating the column below the minimum temperature will not harm the phase, but poor peak shape and other chromatography problems may occur.

Many phases list two maximum operating temperatures. The first temperature is the maximum isothermal operating temperature. This is the temperature to which the columns are guaranteed to meet the minimum bleed specification (i.e., lowest bleed level).



#### Structures, Polarities, Properties, and Uses for Restek® Capillary Column Phases, in Order of Increasing Polarity

#### Rxi®-1ms, Rxi®-1HT, Rtx®-1

Dimethyl polysiloxane



Similar to: (100%-methyl)-polysiloxane

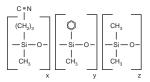
Polarity: nonpolar

Uses: solvents, petroleum products. pharmaceutical samples, waxes

[G2] [G38]

#### Rtx®-1301, Rtx®-624, Rtx®-G43

Cyanopropylmethyl phenylmethyl polysiloxane



Similar to: (6%-cyanopropylphenyl)methylpolysiloxane

Polarity: intermediately polar

[G431

volatile compounds, insecticides

#### Rtx®-200

Trifluoropropylmethyl polysiloxane

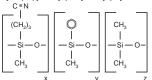


Similar to: (trifluoropropyl)-methylpolysiloxane Polarity: selective for lone pair electrons

environmental, solvents, Freon® gases, drugs, ketones, alcohols [G6]

#### Rtx®-1701

Cyanopropylmethyl phenylmethyl polysiloxane



Similar to: (14%-cyanopropylphenyl)-methylpolysiloxane Polarity: intermediately polar

pesticides, PCBs, alcohols, oxygenates [G46]

## Stabilwax®, Rtx®-Wax,

Stabilwax®-MS Polyethylene glycol



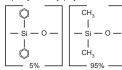
Polarity:

polar FAMEs, flavors, acids, amines, solvents, xylene isomers

[G14] [G15] [G16] [G20] [G39]

#### Rxi®-5ms, Rxi®-5HT, Rtx®-5, Rtx®-5MS

Diphenyl dimethyl polysiloxane

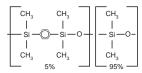


Similar to: (5%-phenyl)-methylpolysiloxane

Polarity: slightly polar Uses: flavors, environmental, [G27] [G36] aromatic hydrocarbons

#### Rxi®-5Sil MS

1,4-bis(dimethylsiloxy)phenylene dimethyl polysiloxane



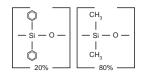
Similar to: (5%-phenyl)-methylpolysiloxane

Polarity: slightly polar

flavors, environmental, pesticides, PCBs, aromatic hydrocarbons

#### Rtx®-20

Diphenyl dimethyl polysiloxane

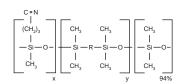


Similar to: (20%-phenyl)-methylpolysiloxane Polarity: slightly polar

volatile compounds, alcohols

[G28] [G32]

#### Rxi®-624Sil MS



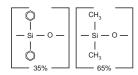
Similar to: (6%-cyanopropylphenyl)-methylpolysiloxane

Polarity: intermediately polar

volatile compounds, insecticides, residue solvents in pharmaceutical products

#### Rtx®-35

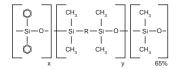
Diphenyl dimethyl polysiloxane



Similar to: (35%-phenyl)-methylpolysiloxane Polarity: intermediately polar Polarity:

pesticides, PCBs, amines [G42] nitrogen-containing herbicides

#### Rxi®-35Sil MS



Similar to: (35%-phenyl)-methylpolysiloxane Polarity: intermediately polar

pesticides, PCBs, amines nitrogen-containing herbicides

#### Rtx®-50

Phenyl methyl polysiloxane



Similar to: (50%-phenyl)-methylpolysiloxane Polarity: intermediately polar

FAMEs, carbohydrates

Rtx®-65, Rtx®-65TG

Diphenyl dimethyl polysiloxane

0

Polarity:

[G17]

CH<sub>3</sub>

Si -0

CH<sub>2</sub>

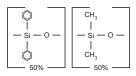
triglycerides, rosin acids, free fatty acids

Similar to: (65%-phenyl)-methylpolysiloxane

intermediately polar

#### Rxi®-17

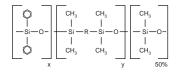
Diphenyl dimethyl polysiloxane



Similar to: (50%-phenyl)-methylpolysiloxane Polarity: intermediately polar

triglycerides, phthalate esters, steroids, phenols

#### Rxi®-17Sil MS

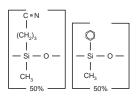


Similar to: (50%-phenyl)-methylpolysiloxane Polarity: intermediately polar

triglycerides, phthalate esters, steroids, phenols

#### Rtx®-225

Cyanopropylmethyl phenylmethyl polysiloxane



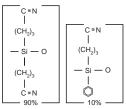
Similar to: (50%-cvanopropylmethyl)methylphenylpolysiloxane

polar FAMEs, carbohydrates Polarity:

[G7] [G19]

#### Rtx®-2330

Biscyanopropyl cyanopropylphenyl polysiloxane



Similar to: (95%-cyanopropyl)-phenyl polysiloxane

Uses: cis/trans FAMEs, dioxin isomers, rosin acids [G8] [G48]

#### note

Structures, polarities, and properties also apply to metal MXT® stationary phases.



## **Columns by Phase**

		USP				Macherey-			
Restek	Phase Description	Nomenclature*		SGE	Phenomenex	Nagel	Supelco	Alltech	Quadrex
Rtx-1 (p. 41) MXT-1 (p. 107)	dimethyl polysiloxane	G1, G2, G38	HP-1, DB-1, CP-Sil 5 CB	BP1	ZB-1	OPTIMA 1	SPB-1	AT-1, EC-1	007-1
Rxi-1HT (p. 39)	dimethyl polysiloxane		DB-1ht		ZB-1HTinferno			AT-1ht	
Rxi-1ms (p. 29)	dimethyl polysiloxane (low bleed)	G1, G2, G38	HP-1ms, HP-1msUI, DB-1ms, DB-1msUI, VF-1ms, Ultra 1	BP1	ZB-1, ZB-1ms	OPTIMA 1 MS, OPTIMA 1 MS Accent	SPB-1, Equity-1	AT-1ms	007-1
Rtx-5MS (p. 44) Rtx-5 (p. 42-43, 96) MXT-5 (p. 108)	diphenyl dimethyl polysiloxane	G27, G36	HP-5, DB-5, CP-Sil 8 CB	BP5	ZB-5	OPTIMA 5	SPB-5	EC-5, AT-5	007-5
Rxi-5HT (p. 39)	diphenyl dimethyl polysiloxane		DB-5ht, VF-5ht	HT5	ZB-5HTinferno	OPTIMA 5HT			
Rxi-5ms (p. 30)	diphenyl dimethyl polysiloxane (low bleed)	G27, G36	HP-5msSV, HP-5ms, HP-5msUI, DB-5, Ultra-2, CP-Sil 8 CB	BP5ms	ZB-5, ZB-5msi	OPTIMA 5, OPTIMA 5 MS	SPB-5, Equity-5	AT-5ms	007-5
Rxi-5Sil MS (p. 32, 67, 75, 77, 85)	1,4-bis(dimethylsiloxy)phenylene dimethyl polysiloxane		DB-5ms, DB-5msUI, VF-5ms	BPX5	ZB-5ms, ZB- SemiVolatiles	OPTIMA 5MS Accent	SLB-5ms		007-5MS
Rxi-XLB (p. 31, 71)	proprietary phase		DB-XLB, VF-Xms		MR1, ZB-XLB	OPTIMA XLB			
Rtx-20 (p. 45)	diphenyl dimethyl polysiloxane	G28, G32					SPB-20	EC-20, AT-20	007-20
Rtx-35 (p. 46)	diphenyl dimethyl polysiloxane	G42	HP-35, DB-35	BPX35, BPX608	ZB-35		SPB-35, SPB-608	AT-35, AT-35ms	007-35
Rxi-35Sil MS (p. 34, 82)	proprietary phase		DB-35ms, DB35msUI, VF-35ms	BPX35	MR2	OPTIMA 35 MS			
Rtx-50 (p. 47) MXT-50 (p. 109)	phenyl methyl polysiloxane	G3	HP-50+, CP-Sil 24 CB				SPB-50	AT-50	007-17
Rxi-17 (p. 35)	diphenyl dimethyl polysiloxane	G3	HP-17, DB-17, DB-17ht, DB-608		ZB-50	OPTIMA 17	SPB-17		
Rxi-17Sil MS (p. 36, 76)	proprietary phase	G3	DB-17ms, VF-17ms	BPX50		OPTIMA 17 MS			
Rtx-65 (p. 47)	diphenyl dimethyl polysiloxane								007-65HT
Rxi-624Sil MS (p. 38, 81, 87, 94)	proprietary phase	G43	DB-624, VF-624ms, CP-Select 624 CB	BP624		OPTIMA 624 LB			
Rtx-1301 (p. 51) Rtx-624 (p. 52) MXT-1301 (p. 109)	cyanopropylmethyl phenylmethyl polysiloxane	G43	DB-1301, DB-624, DB-624UI, VF-1301ms, VF-624ms, CP-1301	BP624	ZB-624	OPTIMA 1301, OPTIMA 624	SPB-624	AT-624, AT-1301	007-1301, 007-624
Rtx-1701 (p. 53) MXT-1701 (p. 109)	cyanopropylmethyl phenylmethyl polysiloxane	G46	DB-1701P, DB-1701, CP-Sil 19 CB, VF-1701ms, VF-1701 Pesticides	BP10	ZB-1701, ZB-1701P	OPTIMA 1701	Equity-1701	AT-1701	007-1701
Rtx-200 (p. 48) MXT-200 (p. 110)	trifluoropropylmethyl polysiloxane	G6	DB-210, DB-200, VF-200ms			OPTIMA 210		AT-210	
Rtx-200MS (p. 48–49)	trifluoropropylmethyl polysiloxane (low bleed)	G6	VF-200ms						
Rtx-225 (p. 54)	cyanopropylmethyl phenylmethyl polysiloxane	G7, G19	DB-225ms, CP-Sil 43 CB	BP225		OPTIMA 225	SPB-225	AT-225	007-225
Rtx-440 (p. 50)	proprietary phase				Restek i	nnovation	1		
Rtx-2330 (p. 55)	biscyanopropyl cyanopropylphenyl polysiloxane	G8, G48	VF-23ms	BPX70			SP-2330, SP-2331, SP-2380	AT-Silar90	007-23
Rt-2560 (p. 56, 83)	bicyanopropyl polysiloxane		HP-88, CP-Sil 88				SP-2560		
Rtx-Wax (p. 57)	polyethylene glycol	G14, G15, G16, G20, G39	DB-Wax, CP-Wax 52 CB	BP20	ZB-Wax	OPTIMA WAX		AT-WAXms, EC-WAX	007-CW
Stabilwax (p. 58, 95) Stabilwax-MS (p. 59) MXT-WAX (p. 110)	polyethylene glycol	G14, G15, G16, G20, G39	HP-INNOWax, CP-Wax 52 CB, VF-WAX MS		ZB-WAXplus	OPTIMA WAXplus	Supelcowax-10	AT-WAX	

See page 117 for Restek PLOT Column Phase Cross-Reference chart.



<sup>\*</sup>See page 147 for our USP Liquid Phase and Solid Support Cross-Reference.

## **Application-Specific Columns by Industry**

Restek	Applications	Agilent	Supelco	Macherey-Nagel	SGE	Alltech	Phenomenex
Chiral Columns						1	
Rt-βDEXm, Rt-βDEXsm,							
Rt-βDEXse, Rt-βDEXsp,	Chiral compounds						
Rt-βDEXsa, Rt-βDEXcst,							
Rt-γDEXsa (p. 103)							
Clinical, Forensic, & Toxicology							
Rtx-BAC Plus 1 (p. 65)	Blood alcohol testing	DB-ALC1					ZB-BAC1
Rtx-BAC Plus 2 (p. 65)		DB-ALC2					ZB-BAC2
Environmental							
Rxi-5Sil MS (p. 67, 75, 77)	Semivolatiles - EPA Methods 8270, 625, 525	DB-5ms, DB-5msUI, VF-5ms	SLB-5ms	OPTIMA 5MS Accent	BPX5		ZB-5ms, ZB-SemiVolatile
Rtx-VMS (p. 78)	Volatiles - EPA Methods 8260, 624, 524			<b>Restek innov</b>	ation		
Rxi-624Sil MS (p. 81)	Volatiles - EPA Method 624	DB-624, VF-624ms, CP-Select 624 CB		OPTIMA 624 LB	BP624		
Rtx-502.2 (p. 80)		DB-502.2	VOCOL			AT-502.2	
Rtx-Volatiles (p. 80)	Volatiles - EPA Methods		VOCOL				
Rtx-VRX (p. 79)	8010, 8020, 502.2, 601, 602	DB-VRX	10002				
Rtx-CLPesticides (p. 72)	Ormanashlarina nastisidas	DB-CLP1					ZB-CLP1
	Organochlorine pesticides -						
Rtx-CLPesticides2 (p. 72)	EPA Methods 8081, 8082, 608, 505, 508	DB-CLP2					ZB-CLP2
Rtx-1614 (p. 66)	Brominated flame retardants			Restek innov			
Rtx-PCB (p. 70)	Polychlorinated biphenyl -			Restek innov	ation		
Rxi-XLB (p. 71)	EPA Methods 8082, 608, PCB congeners	DB-XLB, VF-XMS					MR1, ZB-XLB
Rtx-OPPesticides (p. 74)	Organophosphorus pesticides -			Restek innov	ation		
Rtx-OPPesticides2 (p. 74)	EPA Method 8141			Restek innov			
Rtx-Dioxin2 (p. 68)	Dioxin & Furans - EPA Methods			Restek innov			
Rtx-Mineral Oil (p. 69)	DIN EN ISO 9377-2	Select Mineral Oil		nestek illilov	ation		
Rxi-17Sil MS (p. 76)	Polycyclic aromatic hydrocarbons	DB-17ms, VF-17ms		OPTIMA 17 MS	BPX50		
Foods Flours & Fragrances							
Foods, Flavors, & Fragrances	cis/trans FAMEs	HP-88	SPB-2560				
Rt-2560 (p. 83) FAMEWAX (p. 83)	Marine oils	Select FAME	Omegawax			AT-AquaWax,	
				D		AT-FAME	
Rxi-PAH (p. 84)	PAHs			Restek innov			
Rtx-65 TG (p. 89)	Triglycerides			Restek innov	ation		
Petroleum & Petrochemical							
Rt-Alumina BOND/CFC (p. 123)	Chlorinated fluorocarbons (CFCs)						
Rtx-DHA (p. 92)	Detailed hydrocarbon analysis	HP-PONA, DB-Petro, CP-Sil PONA CB	Petrocol DH		BP1PONA		
Rtx-2887 (p. 93) MXT-2887 (p. 113)	Hydrocarbons - ASTM 2887	DB-2887	Petrocol 2887, Petrocol EX2887			AT-2887	
D3606 (p. 138)	Ethanol - ASTM 3606			Restek innov	ation		
Rt-TCEP (p. 90)		CP-TCEP	TCEP				
MXT-1HT SimDist (p. 114)	Simulated distillation	DB-HT-SimDis, CP-SimDist,			BPX1	AT-3710	ZB-1XT SimDis
MANA E00 Cin. Dint / 115	-	CP-SimDst Ultimetal		Do et als !	-41		
MXT-500 SimDist (p. 115)		ni-di 101 i		Restek innov	ation		
Rtx-Biodiesel TG (p. 91) MXT-Biodiesel TG (p. 113)	Triglycerides in biodiesel	Biodiesel, Select Biodiesel		OPTIMA Biodiesel			ZB-Bioethano
Pharmaceutical						1	
Rtx-G27 w/IntegraGuard (p. 97) Rtx-G43 w/IntegraGuard (p. 97)	_		OVI-G43				
	1	DB-624, VF-624ms,		ODTINA COLLE	DD CO.		
Rxi-624Sil MS (p. 94)	Organic volatile impurities (OVI) - USP 467	CP-Select 624 CB		OPTIMA 624 LB	BP624		
Rtx-5 (G27) (p. 96)	-	HP-5, DB-5, CP-Sil 8 CB	SPB-5	OPTIMA 5	BP5	EC-5, AT-5	ZB-5
Stabilwax (G16) (p. 95)		HP-INNOWax, CP-Wax 52 CB, VF-WAX MS	Supelcowax-10	OPTIMA WAXplus		AT-WAX	ZB-WAXplus
Specially deactivated phases				1		1	
Rtx-Volatile Amine (p. 99)	Volatile amines	CP-Volamine					
Rtx-5Amine (p. 100)				OPTIMA 5 Amine			
Rtx-35Amine (p. 101)	1			Restek innov	ation		
	Amines	CAM, CP-WAX 51					
Stabilwax-DB (p. 102)		for Amines	Carbowax Amine	FS-CW 20 M-AM PERMABOND FFAP,		AT-CAM	
Stabilwax-DA (p. 98)	Free fatty acids	HP-FFAP, DB-FFAP, CP-WAX 58 FFAP CB	NUKOL	OPTIMA FFAP, OPTIMA FFAP Plus	BP-21	AT-AquaWax DA, AT-1000	ZB-FFAP



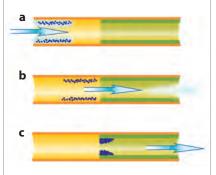
### did you know?

We test our guard columns/transfer lines with a comprehensive test mix to ensure high inertness.

### please note

Having trouble making a leak-free connection? Try our "built in" Integra-Guard® columns!
See page 23 for details.

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



- a) Sample introduction: a liquid film of solvent and sample is deposited in the first length of capillary.
- b) As oven temperature increases, the solvent evaporates and the target compounds elute unretained through the retention gap until they contact the analytical column.
- c) When target compounds come in contact with the stationary phase, they are refocused on the analytical column, resulting in a narrow initial band width.

#### **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) may take place when a piece of deactivated tubing is connected to an analytical column as in Figure 1.

#### **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 purpose 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 0.53 mm 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.

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 10-20 cm, and not allow it 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 left behind.

Using guard columns is advantageous because they prevent contamination from being introduced onto the column. Contaminants can cause active sites as well as change the conditions of the focusing zone of the analytical column. Another advantage is that the resolution of closely eluting compounds will not be affected when the column is trimmed during maintenance because the guard column does not contribute to the resolving power of the analytical column. Using guard columns is a simple, cost-effective way to extend analytical column lifetime.

In summary, the retention gap and guard column are essentially the same products, but are used for different purposes. The deactivated tubing provides an inert pathway, 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. Rxi® guard tubing 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. However, polar-deactivated guard columns are not resistant to harsh "water vaporization," which 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 solvent beading if the oven temperature is 20 °C below the solvent boiling point. Base-deactivated guard columns reduce adsorption and tailing for amines and other basic compounds.

#### How is a guard column connected to the analytical column?

To connect the guard column to the analytical column, Vu2-Union\*, Press-Tight\*, and other connectors are available. MXT\* unions, typically used for connecting metal columns together, are now available for fused silica columns. (See pages 227 to 233 for information about these connectors.)

#### it's a fact

To eliminate leaky connections and to ensure longer column lifetime, use our unique Integra-Guard® column. (See **page 23**.)

#### **Connectors for Fused Silica Columns**



Vu2 Union® Connector (See page 229.)



Press-Tight® Connectors (See pages 227–228.)



MXT® Union Connector Kit for Fused Silica (See page 231.)

