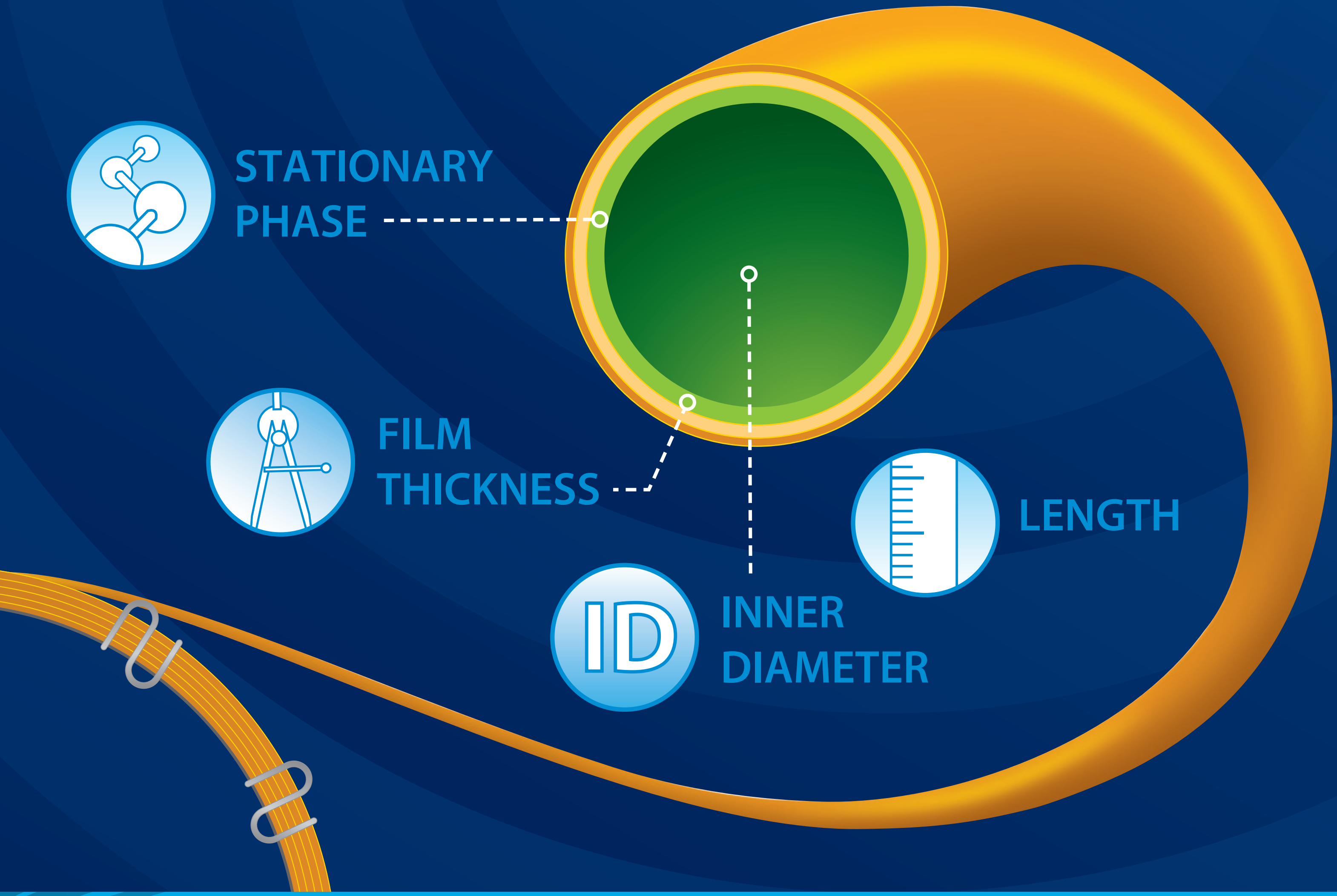
Guide to GC Column Selection and Optimizing Separations

WHICH COLUMN DO I NEED?

- Learn how to choose the right column the first time.
- Optimize separations for the best balance of resolution and speed.
- Troubleshoot quickly and effectively based on chromatographic symptoms.



STATIONARY PHASE

FILM THICKNESS

INNER DIAMETER

LENGTH

RESTEK

Pure Chromatography

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WHICH COLUMN DO I NEED?

Resolution Equation—The Key to Column Selection and Optimizing GC Separations

You can improve lab productivity by assuring that speed and resolution are optimized. One of the best ways to do this is to use the resolution equation (Figure 1) as the key to controlling your separations. This fundamental equation helps you choose the best column stationary phase, length, inner diameter (ID), and film thickness for your specific applications. Once you understand the basics of how resolution is related to column characteristics, optimizing your analysis for both separation and speed becomes easier. This GC column selection guide discusses the basics of separation and teaches you how to choose the right GC column!

Resolution is the goal of every chromatographer, but how much resolution is enough? Practically speaking, we need enough retention to get sharp symmetrical peaks that are baseline resolved from each other, but not too much retention, where retention times are too long and peaks start to broaden. To achieve this goal, we must consider the column and non-column factors that affect our "perfect separation". Only then can we work towards selecting the right column and optimizing GC separations and analysis speed. Now, let's consider separation factor (α), retention factor (k), and efficiency (N) in turn and how they can help you select the right column and optimize your separation.

Shortcut to Column Selection

1. Look for application-specific stationary phases first; these columns are optimized for specific analyses and will provide the best resolution in the shortest time (Table III).
2. If an application-specific column is not available and you need to measure low concentrations or are using a mass spectrometer (MS), then choose an Rxi® column. Rxi® technology unites outstanding inertness, low bleed, and high reproducibility, resulting in high-performance GC columns that are ideal for trace analysis and MS work (Table II).
3. For other methods, choose a general-purpose Rtx® column (Table II).

For additional help choosing a column, search our chromatograms at www.restek.com or contact Technical Service Group at support@restek.com

Figure 1: The resolution equation and factors that affect it.

$$R = \underbrace{\frac{1}{4}\sqrt{N}}\ \mathrm{x}\ \underbrace{\left(\frac{k}{k+1}\right)}\ \mathrm{x}\ \underbrace{\left(\alpha - 1\right)}$$

A measure of **Efficiency**. This term is affected by:
- Length
- Inner diameter
- Carrier gas type and linear velocity

A measure of **Retention**. This term is affected by:
- Inner diameter
- Film thickness
- Temperature

A measure of **Peak Separation**. This term is affected by:
- Stationary phase composition
- Temperature

N = L/H = Effective theoretical plate number
L = Column length
H = HETP = Height equivalent to a theoretical plate

k = Retention factor
α = Separation factor
Baseline resolution (R = 1.5) is the goal.

Use Separation Factor (α) to Choose the Best Stationary Phase

$$R = \left(\frac{1}{4}\sqrt{N}\right) \mathrm{x} \left(\frac{k}{k+1}\right) \mathrm{x} \boldsymbol{(\alpha\text{-}1)}$$

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 (α) 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. 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,

Table I: Kovat's retention indices for GC phases can be used to approximate selectivity.

Stationary Phase	Benzene	Butanol	Pentanone	Nitropropane
100% Dimethyl polysiloxane	651	651	667	705
5% Diphenyl/95% dimethyl polysiloxane	667	667	689	743
20% Diphenyl/80% dimethyl polysiloxane	711	704	740	820
6% Cyanopropylphenyl/94% dimethyl polysiloxane	689	729	739	816
35% Diphenyl/65% dimethyl polysiloxane	746	733	773	867
Trifluoropropylmethyl polysiloxane	738	758	884	980
Phenyl methyl polysiloxane	778	769	813	921
14% Cyanopropylphenyl/86% dimethyl polysiloxane	721	778	784	881
65% Diphenyl/35% dimethyl polysiloxane	794	779	825	938
50% Cyanopropylmethyl/50% phenylmethyl polysiloxane	847	937	958	958
Polyethylene glycol	963	1,158	998	1,230

tech tip

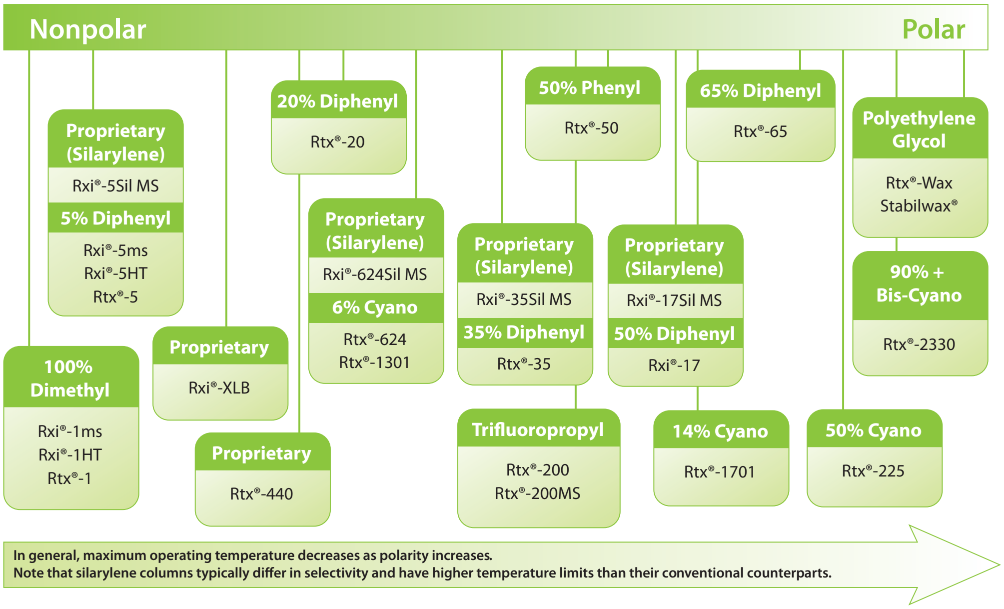
In many cases, different GC oven temperature programs can change the elution order of sample analytes on the same column. Reconfirm elution orders if changing GC oven temperature programs.

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.

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. Use Table II and Figure 2 determine which general-purpose column is most appropriate based on the selectivity, polarity, and the temperature requirements of your analysis. See Table III for a list of specialty stationary phases designed for specific applications.

STATIONARY PHASE

Figure 2: Polarity scale of common stationary phases.



In general, maximum operating temperature decreases as polarity increases.
Note that silarylene columns typically differ in selectivity and have higher temperature limits than their conventional counterparts.

tech tip

Any homologous series of compounds, that is, analytes from the same chemical class (e.g., all alcohols, all ketones, or all aldehydes, etc.) will elute in boiling point order on any stationary phase. However, when different compound classes are mixed together in one sample, intermolecular forces between the analytes and the stationary phase are the dominant separation mechanism, not boiling point.

Table II: Relative polarity and thermal stability are important considerations when selecting a GC stationary phase.

Restek	Phase Composition (USP Nomenclature)	Restek's Max Temps*	Agilent	Phenomenex
Rxi-1HT Rxi-1ms, Rtx-1	100% Dimethyl polysiloxane (G1, G2, G38)	400 °C 350 °C	HP-1/HP-1ms, DB-1/DB-1ms, VF-1ms, CP Sil 5 CB, Ultra 1, DB-1ht, HP-1ms UI, DB-1ms UI	ZB-1, ZB-1MS, ZB-1HT Inferno
Rxi-5HT Rxi-5ms, Rtx-5	5% Diphenyl/95% dimethyl polysiloxane (G27, G36)	400 °C 350 °C	HP-5/HP-5ms, DB-5, Ultra 2, DB-5ht, VF-5ht, CP-Sil 8 CB	ZB-5, ZB-5HT Inferno, ZB-5ms
Rxi-5Sil MS	5% (1,4-bis(dimethylsiloxy) phenylene/95% dimethyl polysiloxane	350 °C	DB-5ms UI, DB-5ms, VF-5ms	ZB-5msi
Rxi-XLB	Proprietary Phase	360 °C	DB-XLB, VF-Xms	MR1, ZB-XLB
Rtx-20	20% Diphenyl/80% dimethyl polysiloxane (G28, G32)	320 °C	—	—
Rtx-35	35% Diphenyl/65% dimethyl polysiloxane (G42)	320 °C	HP-35, DB-35	ZB-35
Rxi-35Sil MS	Proprietary Phase	360 °C	DB-35ms, DB-35ms UI, VF-35ms	MR2
Rtx-50	Phenyl methyl polysiloxane (G3)	320 °C	—	—
Rxi-17	50% Diphenyl/50% dimethyl polysiloxane	320 °C	DB-17ms, VF-17ms, CP Sil 24 CB	ZB-50
Rxi-17Sil MS	Proprietary Phase	360 °C	DB-17ms, VF-17ms, CP Sil 24 CB	ZB-50
Rtx-65	65% Diphenyl/35% dimethyl polysiloxane (G17)	300 °C	—	—
Rxi-624Sil MS	Proprietary Phase	320 °C	DB-624 UI, VF-624ms, CP-Select 624 CB	ZB-624
Rtx-1301, Rtx-624	6% Cyanopropylphenyl/94% dimethyl polysiloxane (G43)	280 °C 240 °C	DB-1301, DB-624, CP-1301, VF-1301ms, VF-624ms	ZB-624
Rtx-1701	14% Cyanopropylphenyl/86% dimethyl polysiloxane (G46)	280 °C	DB-1701, VF-1701ms, CP Sil 19 CB, VF-1701 Pesticides, DB-1701R	ZB-1701, ZB-1701P
Rtx-200, Rtx-200MS	Trifluoropropyl methyl polysiloxane (G6)	340 °C	DB-200, VF-200ms, DB-210	—
Rtx-225	50% Cyanopropyl methyl/50% phenylmethyl polysiloxane (G7, G19)	240 °C	DB-225ms, CP Sil 43 CB	—
Rtx-440	Proprietary Phase	340 °C	**RESTEK INNOVATION**	
Rtx-2330	90% Biscyanopropyl/10% cyanopropylphenyl polysiloxane (G48)	275 °C	VF-23ms	—
Rt-2560	Biscyanopropyl polysiloxane	250 °C	HP-88, CP Sil 88	—
Rtx-Wax	Polyethylene glycol (G14, G15, G16, G20, G39)	250 °C	DB-Wax, Wax 52 CB	ZB-WAX
Stabilwax	Polyethylene glycol (G14, G15, G16, G20, G39)	260 °C	HP-INNOWax, VF-WaxMS	ZB-WAXPlus

* Maximum operating temperatures may vary with column film thickness.

Table III: Application-specific phases designed for particular analyses.

Restek	Applications	Agilent	Supelco	Macherey-Nagel	SGE	Phenomenex
Rtx-Volatile Amine	Volatile amines	CP-VolAmine	—	—	—	—
Rtx-5Amine	Amines	CP-Sil 8 CB	—	OPTIMA 5 Amine	—	—
Rtx-35Amine	Amines	—	—	**RESTEK INNOVATION**		—
Stabilwax-DB	Amines	CAM, CP WAX 51	Carbowax Amine	FS-CW 20 M-AM	—	—
Stabilwax-DA	Free fatty acids	HP-FFAP, DB-FFAP, VF-DA, CP WAX58 CB, CP-FFAP CB	Nukol	PERMABOND FFAP,OPTIMA FFAP, OPTIMA FFAP Plus	BP-21	ZB-FFAP
Chiral Columns						
Rt-βDEXm, Rt-βDEXsm, Rt-βDEXse, Rt-βDEXsp, Rt-βDEXsa, Rt-βDEXcst, Rt-γDEXsa	Chiral compounds	—	—	—	—	—
Foods, Flavors, & Fragrances						
Rt-2560	*cis/trans* FAMEs	HP-88	SPB-2560	—	—	—
FAMEWAX	Marine oils	Select FAME	Omegawax	—	—	—
Rtx-65 TG	Triglycerides	—	—	—	—	—
Rxi-PAH	Polycyclic aromatic hydrocarbons (PAHs)	Agilent Select PAH	—	—	—	—

Table III: Application-specific phases designed for particular analyses (cont.)

Restek	Applications	Agilent	Supelco	Macherey-Nagel	SGE	Phenomenex
Petroleum & Petrochemical						
Rt-Alumina BOND/CFC	Chlorinated fluorocarbons (CFCs)	—	—	**RESTEK INNOVATION**		—
Rtx-DHA	Detailed hydrocarbon analysis	HP-PONA, DB-Petro, CP Sil PONA CB	Petrocol DH	—	BP1PONA	—
Rtx-2887	Hydrocarbons (ASTM D2887)	DB-2887	Petrocol 2887, Petrocol EX2887	—	—	—
MXT-2887	Hydrocarbons (ASTM D2887)	DB-2887	Petrocol 2887, Petrocol EX2887	—	—	—
D3606	Ethanol (ASTM D3606)	—	—	**RESTEK INNOVATION**		—
Rt-TCEP	Aromatics and oxygenates in gasoline	CP-TCEP	TCEP	—	—	—
MXT-1HT SimDist	Simulated distillation	DB-HT-SimDis,CP-SimDist, CP-SimDist Ultimetal	—	—	BPX1	ZB-1XT SimDist
MXT-1 SimDist	Simulated distillation	DB-HT-SimDis,CP-SimDist, CP-SimDist Ultimetal	—	—	—	—
MXT-500 SimDist	Simulated distillation	—	—	**RESTEK INNOVATION**		—
Rtx-Biodiesel TG, MXT-Biodiesel TG	Triglycerides in biodiesel	Biodiesel, Select Biodiesel	—	OPTIMA Biodiesel	—	ZB-Bioethanol
Clinical/Forensic						
Rtx-BAC Plus 1	Blood alcohol testing	DB-ALC1	—	—	—	ZB-BAC1
Rtx-BAC Plus 2	Blood alcohol testing	DB-ALC2	—	—	—	ZB-BAC2
Pharmaceutical						
Rtx-G27 w/IntegraGuard	Organic volatile impurities (USP <467>)	—	—	—	—	—
Rtx-G43 w/IntegraGuard	Organic volatile impurities (USP <467>)	—	—	—	—	—
Rxi-624Sil MS	Organic volatile impurities (USP <467>)	DB-624,VF-624ms, CP-Select 624 CB	—	OPTIMA 624 LB	BP624	ZB-624
Rtx-5 (G27)	Organic volatile impurities (USP <467>)	HP-5, DB-5,CP Sil 8 CB	SPB-5	OPTIMA 5	BP5	ZB-5
Stabilwax (G16)	Organic volatile impurities (USP <467>)	HP-INNOWax,CP Wax 52 CB,VF-WAX MS	Supelcowax-10	OPTIMA WAXplus	—	ZB-WAXplus
Environmental						
Rxi-5Sil MS	Semivolatiles	DB-5ms,DB-5msUI, VF-5ms,CP-Sil 8 CB	SLB-5ms	OPTIMA 5MS Accent	BPX5	ZB-5msi
Rtx-VMS	Volatiles (EPA Methods 8260, 624, 524)	—	—	**RESTEK INNOVATION**		—
Rxi-624Sil MS	Volatiles (EPA Methods 624)	DB-624,VF-624ms, CP-Select 624 CB	—	OPTIMA 624 LB	BP624	ZB-624
Rtx-502.2	Volatiles (EPA Methods 8010, 8020, 502.2, 601, 602)	DB-502.2	VOCOL	—	—	—
Rtx-Volatiles	Volatiles (EPA Methods 8010, 8020, 502.2, 601, 602)	—	VOCOL	—	—	—
Rtx-VRX	Volatiles (EPA Methods 8010, 8020, 502.2, 601, 602)	DB-VRX	—	—	—	—
Rtx-CLPesticides	Organochlorine pesticides	—	—	**RESTEK INNOVATION**		—
Rtx-CLPesticides2	Organochlorine pesticides	—	—	**RESTEK INNOVATION**		—
Rtx-1614	Brominated flame retardants	—	—	**RESTEK INNOVATION**		—
Rtx-PCB	Polychlorinated biphenyl (PCB) congeners	—	—	**RESTEK INNOVATION**		—
Rxi-XLB	Polychlorinated biphenyl (PCB) congeners	DB-XLB,VF-XMS	—	—	—	MR1, ZB-XLB
Rtx-OPPesticides	Organophosphorus pesticides	—	—	**RESTEK INNOVATION**		—
Rtx-OPPesticides2	Organophosphorus pesticides	—	—	**RESTEK INNOVATION**		—
Rtx-Dioxin2	Dioxins and furans	—	—	**RESTEK INNOVATION**		—
Rxi-17Sil MS	Polycyclic aromatic hydrocarbons (PAHs)	DB-17ms,VF-17ms, CP-Sil 24 CB	—	OPTIMA 17 MS	BPX50	ZB-50
Rtx-Mineral Oil	DIN EN ISO 9377-2	Select Mineral Oil	—	—	—	—

Select Column Film Thickness and Column ID Based on Retention Factor

Once you have chosen the stationary phase, you need to determine which column film thickness and inner diameter combination will give the retention factor (k) needed for optimal resolution and speed. Retention factor is sometimes referred to as capacity factor, which should not be confused with sample loading capacity.

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. 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, so at higher temperatures analytes stay in the carrier gas longer and are less retained.

In practice, if the value of k is too large, the peak will broaden, which can reduce resolution by causing peaks to overlap or coelute. Narrow, symmetrical peaks are important to maximizing resolution, so the goal is to select a column with a sufficient retention factor, such that resolution occurs and peak shape does not suffer. Once the proper stationary phase is selected, column film thickness, column inner diameter, and elution temperature should be optimized to produce an acceptable retention factor.

tech tip

The sample loading capacity of the column also must be considered; if the mass of the target analyte exceeds the sample loading capacity of the column, loss of resolution, poor reproducibility, and fronting peaks will result. A larger ID column with thicker film is recommended for higher concentration samples, such as purity analysis, to minimize sample overload.

Film Thickness

Film thickness (µm) has a direct effect on both the retention of each sample component and the maximum operating temperature of the column. When analyzing extremely volatile compounds, a thick film column should be used to increase retention; more separation is achieved because the compounds spend more time in the stationary phase. If analyzing high molecular weight compounds, a thinner film column should be used, as this reduces the length of time that the analytes stay in the column and minimizes phase bleed at higher elution temperatures. Use Figure 3 to select the best film thickness for your application. Note that as a general rule, the thicker the film, the lower the maximum temperature; exceeding the maximum temperature can result in column bleed and should be avoided.

FILM THICKNESS

Figure 3: Characteristics and recommended applications based on film thickness.

Thin Film **Thick Film**

0.10 µm–0.50 µm

Characteristics
- Shorter retention times
- Lower bleed
- Higher maximum temperatures
- Lower sample loading capacity
- High resolution for high molecular weight compounds

Applications

Medium and high molecular weight compounds

1.0 µm–10.0 µm

Characteristics
- Longer retention times
- Higher bleed
- Lower maximum temperatures
- Higher sample loading capacity
- High resolution for volatiles and low molecular weight compounds

Applications
- Volatile, low molecular weight compounds
- High concentration samples (e.g., purity testing)

As film thickness increases, retention, sample loading capacity, and column bleed increase; whereas, maximum temperature decreases.

tech tip

Remember, when changing either film thickness and/or the temperature program, you must reconfirm peak identifications as elution order changes can occur.

Inner Diameter (ID)

Column ID does not have as great an effect on retention factor as film thickness does. However, when selecting column ID with retention factor (k) in mind, a general rule of thumb applies; smaller ID columns produce higher retention factors compared to larger ID columns. This is due to less available mobile phase (carrier gas) volume in the column. Because smaller ID columns produce higher k values, they are more suited towards complex sample analysis where a range of low to high molecular weight compounds may exist in the sample (Figure 4). Keep in mind that both ID and film thickness should be optimized together to produce the best resolution and peak shape.

INNER DIAMETER

Figure 4: Characteristics and recommended applications based on column inner diameter.

Small ID — **Large ID**

0.15 mm–0.18 mm

Characteristics
- Highest efficiency
- Shorter analysis time
- Lower sample loading capacity

Applications
- Highly complex samples
- Fast GC
- GC-MS
- Split injection

0.25 mm–0.32 mm

Characteristics
- High efficiency
- Good performance for analysis time and sample loading capacity

Applications
- Complex samples
- Wide concentration range
- Split , splitless, direct, headspace, and on-column injection

0.53 mm

Characteristics
- Good efficiency
- Longer analysis time
- Higher sample loading capacity
- May require higher flow rates than MS detectors can tolerate

Applications
- Packed column replacement
- Purity analysis
- Split, splitless, direct, headspace, and on-column injection

As inner diameter increases, efficiency decreases, sample loading capacity increases, optimal flow rate increases, and analysis time increases.

tech tip

When choosing column ID, the injection technique is also 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, the detector and its optimal flow rate must be considered. 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.

Phase Ratio (β)

The relationship between column inner diameter and stationary phase film thickness is expressed as phase ratio (β). If a good separation has been achieved on a larger diameter column and a faster analysis is desired, this can often be accomplished by reducing the inner diameter of the column without sacrificing, and sometimes even improving, separation efficiency. To maintain a similar compound elution pattern when narrowing column inner diameter, film thickness must also be changed. By choosing a column with a similar phase ratio, it will be easier to translate your application to the new column. Phase ratios for common column dimensions and the equation for β are given in Table IV. As shown here, an analyst wanting to decrease analysis time could switch from a 0.32 mm x 0.50 µm column (β = 160) to a 0.25 mm x 0.25 µm column (β = 250) and obtain a very similar separation upon proper method translation. Importantly, column inner diameter and stationary phase film thickness show a combined effect when it comes to sample loading capacity, which is decreased as column inner diameter and film thickness are reduced. It may be necessary to inject a lower sample amount in this case.

Table IV: Phase ratio (β)* values for common column dimensions. To maintain similar separations, choose columns with similar phase ratios when changing to a column with a different inner diameter or film thickness.

	Film Thickness (d_f)						
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	1,325	530	265	128	88	43	27

*Phase ratio (β) = radius/2d_f (Note: Convert variables to the same units prior to calculation.)

$$R=\left(\frac{1}{4}\sqrt{N}\right)x\left(\frac{k}{k+1}\right)x\left(\alpha\text{-}1\right)$$

Consider Efficiency when Choosing Column Length, Column ID, and Carrier Gas

Column Length

Capillary GC columns are made in various lengths, typically 10, 15, 30, 60, and 105 meters, depending on the inner diameter. Longer columns provide more resolving power than shorter columns of the same inner diameter, but they also increase analysis time and should be used only for applications demanding the utmost in separation power. Column length should only be considered once the stationary phase has been determined. This is because separation factor has the greatest effect on resolution, and it is maximized through proper stationary phase choice for the compounds of interest. Doubling the column length (e.g., 30 m to 60 m) increases resolution by approximately 40%, while analysis time can be twice as long. In addition, longer columns cost more. Conversely, if a separation can be performed on a shorter column (e.g., 15 m versus 30 m), then both analysis time and column cost will be less. Figure 5 summarizes the characteristics and general application parameters for a range of typical column lengths.

LENGTH

Figure 5: Characteristics and recommended applications based on column length.

Short Length → **Long Length**

5 m–15 m	20 m–30 m	50 m–150 m
Characteristics • Good efficiency • Short analysis times	**Characteristics** • Better efficiency • Moderate analysis times	**Characteristics** • Best efficiency • Longer analysis times
Applications Samples with few compounds	**Applications** More complex samples	**Applications** Very complex samples

Longer Columns Can Increase Resolution...
Doubling the column length only increases resolution by approximately 40% because the column length is under the square root function in the efficiency term of the resolution equation.

But, Longer Columns Increase Cost and Analysis Time
On longer columns, analysis time is increased by as much as a factor of two. Longer columns are also more expensive.

Inner Diameter (ID)

Compared to larger ID columns, smaller ID columns generate more plates per meter and sharper peaks, leading to better separation efficiencies. When more complex samples need to be analyzed, smaller ID columns can produce better separation of closely eluting peaks than larger ID columns. However, sample loading capacities are lower for smaller ID columns. Smaller ID columns, especially those at 0.18 mm and less, demand highly efficient injection techniques so that the column efficiency is not lost at the point of sample introduction. Column characteristics based on ID are presented in Table V.

Generally speaking, a 0.25 mm column will produce the most efficient sample analysis while simultaneously considering analysis time and sample loading capacity. For these reasons, in combination with its relatively low outlet flow, it is also the best column choice for GC-MS work.

Table V: General column characteristics based on ID.

	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.

Carrier Gas Type and Linear Velocity

Carrier gas choice and linear velocity significantly affect column separation efficiency, which is best illustrated using van Deemter plots (Figure 6). The optimum linear velocity for each gas is at the lowest point on the curve, where plate height (H) is minimized, and efficiency is maximized. As seen in Figure 6, the optimum linear velocities differ among common carrier gases.

tech tip

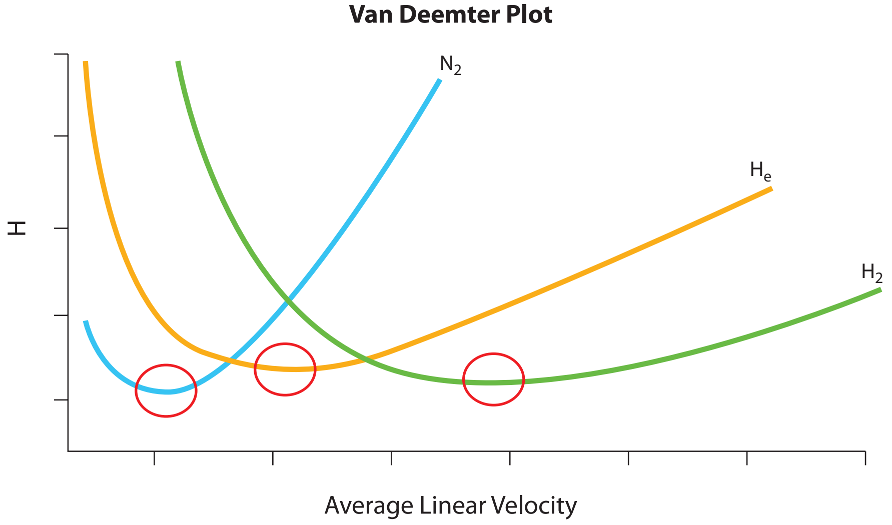
When changing carrier gas flow rates you must reconfirm peak identifications as elution order changes can occur.

Nitrogen provides the best efficiency; however, the steepness of its van Deemter plot on each side of optimum means that small changes in linear velocity can result in large negative changes in efficiency. Compared to nitrogen, helium has a wider range for optimal linear velocity, but offers slightly less efficiency. In addition, because of its optimum velocity being faster, analysis times with helium are about half those when using nitrogen, and there is only a small sacrifice in efficiency when velocity changes slightly. Of the three common carrier gases, hydrogen has the flattest van Deemter curve, which results in the shortest analysis times and the widest range of average linear velocity over which high efficiency is obtained.

Regardless of the type of gas used, the carrier gas head pressure is constant during column temperature programming, whereas the average linear velocity decreases during the run. For constant pressure work then, the optimal linear velocity should be set for the most critical separations. More commonly today, electronic pneumatic control (EPC) of carrier gas allows for constant flow or even constant linear velocity, which helps maintain high efficiency throughout a temperature programmed run.

Another consideration for carrier gas type that is important, even if not directly related to column efficiency, is whether a mass spectrometer (MS) is used as a vacuum-outlet detector for GC. In almost all cases, helium is the carrier gas of choice, not only for its chromatographic efficiency, but also because it is easier to pump than hydrogen. Hydrogen can be reactive in MS sources, leading to undesirable spectrum changes for some compounds. Nitrogen is typically not a carrier gas option for GC-MS, as it severely reduces sensitivity.

Figure 6: Operating carrier gas at the optimum linear velocity will maximize efficiency at a given temperature. Red circles indicate optimum linear velocities for each carrier gas.



GC TROUBLESHOOTING TIPS

Basic Steps

Follow these basic troubleshooting steps to isolate problems related to the sample, injector, detector, and column. Check the obvious explanations first and change only one thing at a time until you identify and resolve the problem.

Check the Obvious:

- Power supply
- Electrical connections
- Signal connections
- Gas purity
- Gas flows
- Temperature settings
- Syringe condition
- Sample preparation
- Analytical conditions

Identify the Cause:

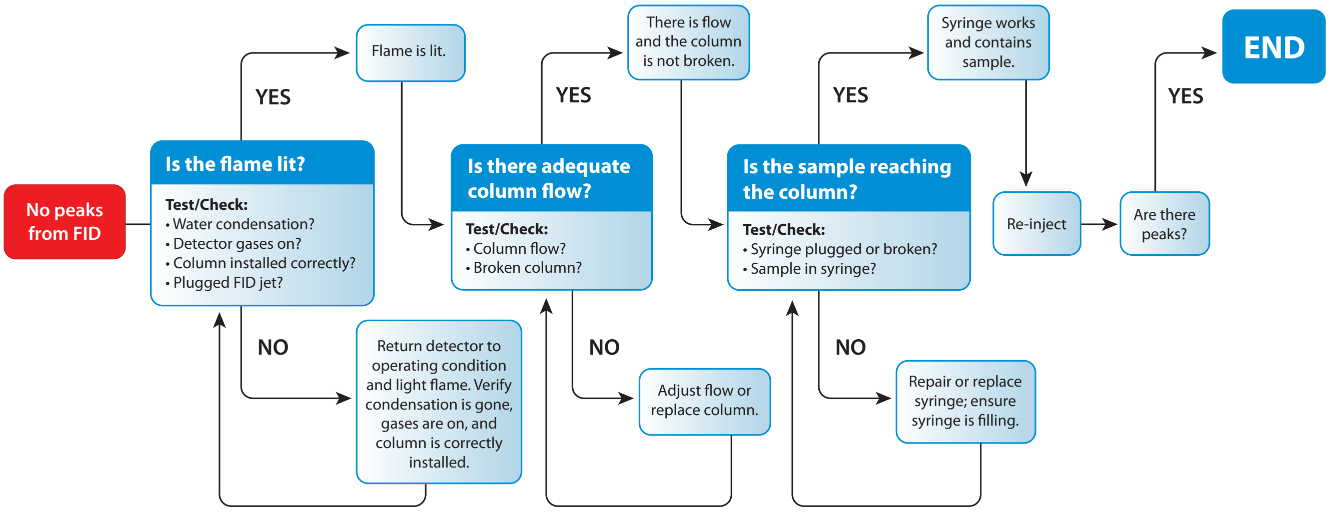
- Define the problem clearly; for example, "Over the last 4 days, only the phenols in my sample have been tailing."
- Review sample and maintenance records to identify trends in the data or problem indicators, such as area counts decreasing over time or injector maintenance not being performed as scheduled.
- Use a logical sequence of steps to isolate possible causes.

Document Work and Verify System Performance:

- Document all troubleshooting steps and results; this may help you identify and solve the next problem faster.
- Always inject a test mix and compare to previous data to ensure restored performance.

Example Troubleshooting Sequence

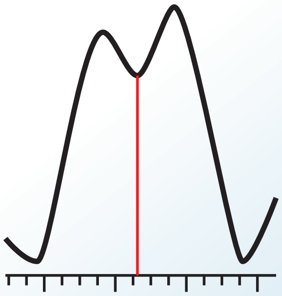
An analyst observed that no peaks appeared during a GC-FID analysis. The flowchart below shows a logical progression of steps that can be used to identify the cause and correct the problem.



Symptoms and Solutions

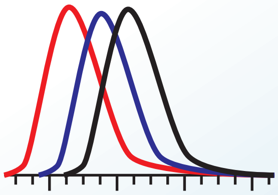
Good chromatography is critical to obtaining accurate, reproducible results. Coelutions, asymmetric peaks, baseline noise, and other issues are common challenges in the GC laboratory. These analytical problems and others can be overcome by troubleshooting your separations using the tips below.

Poor Resolution



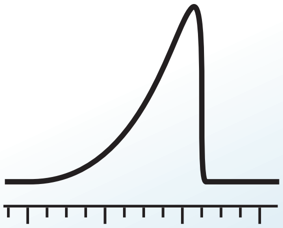
Causes	Solutions
Non-selective stationary phase	• Choose appropriate stationary phase and column dimensions.
Poor efficiency	• Optimize carrier gas linear velocity and GC oven temperature program.
Sample overload	• Adjust sample concentration or amount on column.
Incorrect analytical conditions used	• Verify temperature program, flow rates, and column parameters.

Poor Retention Time Reproducibility



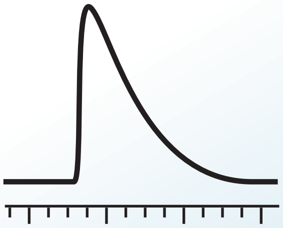
Causes	Solutions
Leaks	• Leak check injector and press-fit connections. • Replace critical seals (i.e., septa, O-rings, inlet disc, etc.)
Analyte adsorption	• Maintain inlet liner and GC column. • Use properly deactivated liners, seals, and columns.
Resolution/integration issues	• Avoid sample overload.
Incorrect column/oven temperature program	• Verify column temperature and oven temperature program.
Incorrect or variable carrier gas flow rate/linear velocity	• Verify the carrier gas flow and linear velocity. Repair or replace parts if neccessary.
Poor control of oven temperature programming	• Confirm GC oven program falls within instrument manufacturer's recommendation.
Incorrect oven equilibration time	• Extend GC oven equilibration time.
If manual injection, delay between pushing start and actual injection	• Use autosampler or standardize manual injection procedure.

Fronting Peaks



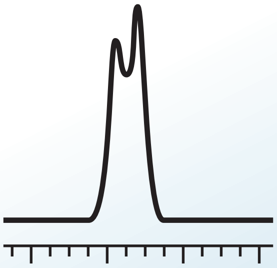
Causes	Solutions
Incompatible stationary phase	• Choose appropriate stationary phase.
Column overloading	• Reduce amount injected, dilute sample. • Increase column inner diameter and/or film thickness.

Tailing Peaks



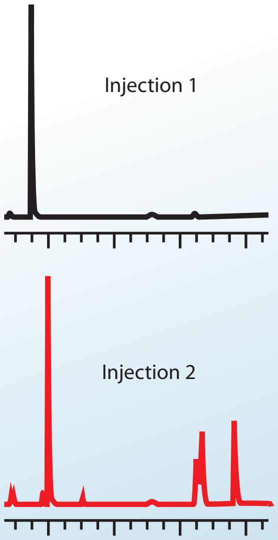
Causes	Solutions
Adsorption due to surface activity or contamination	• Use properly cleaned and deactivated liner, seal, and column. • Trim inlet end of column. • Replace column if damaged.
Adsorption due to chemical composition of compound	• Derivatize compound.
Leak in system	• Check for leaks at all connections, replace critical seals if needed.
Installation issues	• Minimize dead volume. • Verify that the column is cut properly (square). • Verify correct installation distances.

Split Peaks



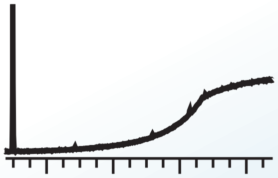
Causes	Solutions
Mismatched solvent/stationary phase polarity	• Adjust solvent or stationary phase to allow wetting.
Incomplete vaporization	• Add surface area, such as wool, to the inlet liner to enhance vaporization. • Use proper injector temperature.
Sample loading capacity exceeded	• Inject less sample (dilute, use split injection, reduce injection volume).
Fast autosampler injection into open liner	• Use wool or slow injection speed.

Carryover/Ghost Peaks



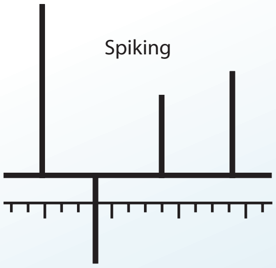
Causes	Solutions
Contaminated syringe or rinse solvent	• Replace rinse solvent. • Rinse or replace syringe.
Backflash (sample volume exceeds liner volume)	• Inject a smaller amount. • Use a liner with a large internal diameter. • Increase head pressure (i.e., flowrate) to contain the vapor cloud. • Use slower injection rate. • Lower inlet temperature. • Increase split flow. • Use liner with packing. • Use pressure-pulse injection.
Last analysis ended too soon	• Extend analysis time to allow all components and/or matrix interferences to elute.

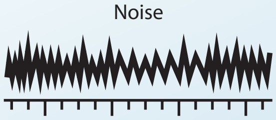
High Bleed

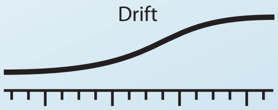


Causes	Solutions
Improper column conditioning	• Increase conditioning time and/or temperature.
Contamination	• Trim column and/or heat to maximum temperature to remove contaminants. • Replace carrier gas and/or detector gas filters. • Clean injector and detector.
Leak in system and oxidation of stationary phase	• Check for oxygen leaks across the entire system and replace seals and/or filters. • Replace column.

Unstable Baseline (Spiking, Noise, Drift)

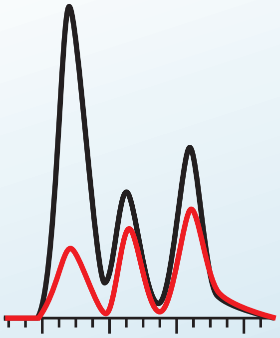






Causes	**Solutions**
Carrier gas leak or contamination	• Leak check connections and replace seals if needed. • Replace carrier gas and/or detector gas filters.
Injector or detector contamination	• Clean system and perform regular maintenance.
Column contamination or stationary phase bleed	• Condition, trim, and rinse column.
Septum coring/bleed	• Replace septum. • Inspect inlet liner for septa particles and replace liner if needed.
Loose cable or circuit board connections	• Clean and repair electrical connections.
Variable carrier gas or detector gas flows	• Verify flow rates are steady and reproducible; may need to replace or repair flow controller. • Leak check system.
Detector not ready	• Allow enough time for detector temperatures and flows to equilibrate.

Response Variation

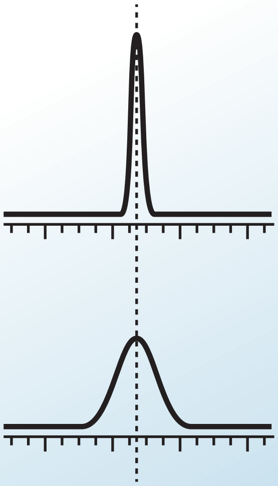


Causes	**Solutions**
Sample issues	• Check sample concentration. • Check sample preparation procedure. • Check sample decomposition/shelf life.
Syringe problems	• Replace syringe. • Check autosampler operation.
Electronics	• Verify signal settings and adjust if needed. • Repair or replace cables or boards.
Dirty or damaged detector	• Perform detector maintenance or replace parts.
Flow/temperature settings wrong or variable	• Verify steady flow rates and temperatures, then adjust settings and/or replace parts if needed.
Adsorption/reactivity	• Remove contamination and use properly deactivated liner, seal, and column.
Leaks	• Check for leaks at all connections and repair connections as needed.
Change in sample introduction/injection method	• Verify injection technique and change back to original technique. • Check that split ratio is correct. • Verify that the splitless hold time is correct.

No Peaks

Causes	Solutions
Injection problems	• Plugged syringe; clean or replace syringe. • Verify there is sample in the syringe. • Injecting into wrong inlet; reset autosampler. • Verify carrier gas is flowing.
Broken column	• Replace column.
Column installed into wrong injector or detector	• Re-install column.
Detector problems	• Signal not recorded; check detector cables and verify that detector is turned on. • Detector gas turned off or wrong flow rates used; turn detector on and/or adjust flow rates.

Broad Peaks



Causes	Solutions
High dead volume	• Minimize dead volume in the GC system; verify proper column installation, proper connectors, proper liners, etc.
Low flow rates	• Verify injector and detector flow rates and adjust if needed. • Verify make-up gas flow and adjust if needed.
Slow GC oven program	• Increase GC oven programming rate.
Poor analyte/solvent focusing	• Lower GC oven start temperature.
Column film is too thick	• Reduce retention of compounds by decreasing film thickness and length.
Sample carryover	• See Carryover/Ghost Peaks solutions.

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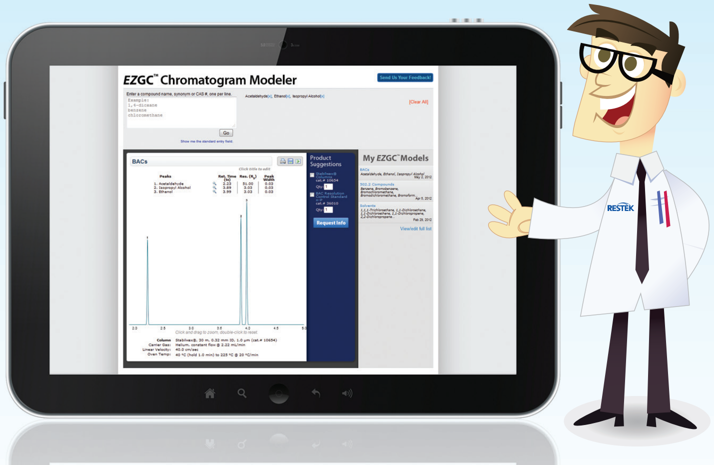

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