Selecting a GC Column

Capillary column selection can be a challenging task for many chromatographers. Several simple principles can be kept in mind to simplify the selection process and result in the optimum column for the analytical task at hand. Keep in mind that in selecting the proper capillary column, the chromatographer is faced with many options that require optimizing analysis speed, retention or capacity, and resolution. These three analysis goals are affected by several factors or variables contained in the resolution equation:

$$R = \frac{1}{4}\sqrt{\frac{L}{h}} \times \frac{k}{k+1} \times \frac{\alpha-1}{\alpha}$$

Efficiency Capacity Selectivity

R=resolution; L=column length; h=HETP; k=capacity factor; α=selectivity

The resolution equation is divided roughly into three sections consisting of variables affecting selectivity, efficiency, and capacity or retention. Looking at how each section of the resolution equation influences the analytical separation will make column selection less difficult.

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Selectivity, α

The selectivity of the capillary column is directly related to how the analyte molecule interacts with the stationary phase being considered. If the analyte strongly interacts with the stationary phase, it can be said that strong "intermolecular" forces exist. These intermolecular forces of attraction of the analyte for the stationary phase are a function of the structure of both the analyte molecule and the stationary phase. If these two structures are similar, then these attractive forces for one another are strong. If they are weak, then analyte to stationary phase attraction is weak, and retention is less. Therefore, when selecting a stationary phase, knowledge of the structure of the analytes of interest and the stationary phase is crucial. Table II provides the chemical structure of Restek's most common stationary phases.

An example of selectivity can be shown using benzene and butanol (both have nearly the same boiling point) eluting through the 5% diphenyl/95% dimethyl polysiloxane stationary phase (Rtx®-5/Rtx®-5ms). The benzene molecule will dissolve into the stationary phase more readily than the butanol based on the concept that "likes dissolve likes". Benzene desolvating more readily with the stationary phase results in more interactions with the stationary phase as it elutes through the column. Therefore, the elution of these two compounds on the Rtx®-5/Rtx®-5ms stationary phase will be butanol eluting first and benzene second.

As methyl groups are replaced by different functionalities such as phenyl or cyanopropyl pendant groups, the selectivity of the column shifts towards compounds that will have a better solubility in the stationary phase. For example the Rtx®-200 stationary phase provides high selectivity for analytes containing lone pair electrons, such as halogens, nitrogen, or carbonyl groups. Polyethylene glycol columns, such as the Stabilwax® and Rtx®-Wax columns are highly selective towards polar compounds such as alcohols. Again using the example above, the butanol will more readily desolvate into the polyethylene glycol stationary phase; therefore, the butanol will have more interaction with the phase and elute after benzene.

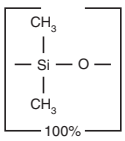
Table I lists the Kovats retention indices for the stationary phases in Table II. Assigning a retention index to each probe listed provides a basis for comparing several stationary phases and their relative retention to one another for a set of molecular probes. For example, when Kovats indices are identical on two column phases, then the resulting separations will be identical. If, however, a Kovats value of one probe varies significantly from the value on another phase for the same probe, then the resulting compound elution order will differ. Thus, the Kovats indices are useful for comparing selectivity of different types of compounds among different phases.

Table I Retention indices for Restek phases

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	1158	998	1230

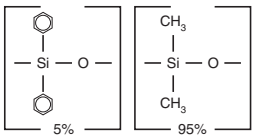
Table II Structures, polarities, properties, and uses for Restek capillary column phases, in order of increasing polarity

Rxi®-1ms, Rtx®-1, Rtx®-1ms
100% dimethyl polysiloxane



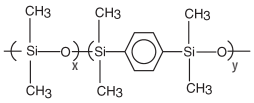
Polarity: non-polar
Uses: solvents, petroleum products, pharmaceutical samples, waxes
[G1]

Rxi®-5ms, Rtx®-5, Rtx®-5MS
5% diphenyl
95% dimethyl polysiloxane



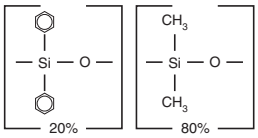
Polarity: slightly polar
Uses: flavors, environmental, aromatic hydrocarbons
[G27]

Rxi®-5Sil MS, Rtx®-5Sil MS
proprietary



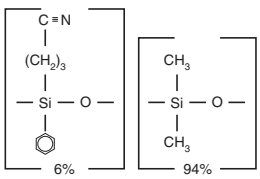
Polarity: slightly polar
Uses: flavors, environmental, pesticides, PCBs, aromatic hydrocarbons

Rtx®-20
20% diphenyl
80% dimethyl polysiloxane



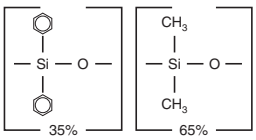
Polarity: slightly polar
Uses: volatile compounds, alcohols
[G32]

Rtx®-1301, Rtx®-624, Rtx®-G43
6% cyanopropylphenyl
94% dimethyl polysiloxane



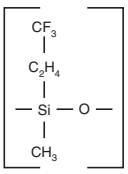
Polarity: slightly polar
Uses: volatile compounds, insecticides, residue solvents in pharmaceutical products
[G43]

Rtx®-35
35% diphenyl
65% dimethyl polysiloxane



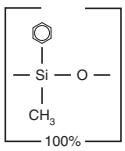
Polarity: intermediately polar
Uses: pesticides, Aroclor® PCBs, amines, nitrogen-containing herbicides
[G42]

Rtx®-200
trifluoropropylmethyl polysiloxane



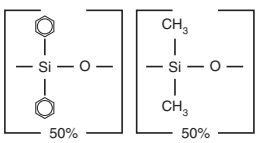
Polarity: selective for lone pair electrons
Uses: environmental, solvents, Freon® gases, drugs, ketones, alcohols
[G6]

Rtx®-50
100% methylphenyl polysiloxane



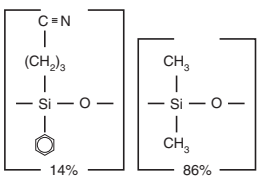
Polarity: intermediately polar
Uses: FAMEs, carbohydrates
[G3]

Rxi®-17
50% diphenyl
50% dimethyl polysiloxane



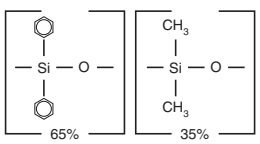
Polarity: intermediately polar
Uses: triglycerides, phthalate esters, steroids, phenols
[G3]

Rtx®-1701
14% cyanopropylphenyl
86% dimethyl polysiloxane



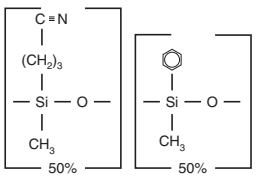
Polarity: intermediately polar
Uses: pesticides, Aroclor® PCBs, alcohols, oxygenates
[G46]

Rtx®-65TG
65% diphenyl
35% dimethyl polysiloxane



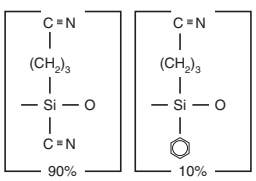
Polarity: intermediately polar
Uses: triglycerides, rosin acids, free fatty acids

Rtx®-225
50% cyanopropylmethyl
50% phenylmethyl polysiloxane



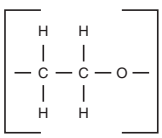
Polarity: polar
Uses: FAMEs, carbohydrates
[G7]

Rt™-2330
90% biscyanopropyl
10% cyanopropylphenyl polysiloxane



Polarity: very polar
Uses: cis/trans FAMEs, dioxin isomers, rosin acids
[G48]

Stabilwax®, Rtx®-Wax
Carbowax® PEG



Polarity: polar
Uses: FAMEs, flavors, acids, amines, solvents, xylene isomers
[G16]

ordering **note**

Designations in [brackets] are USP codes. We recommend this phase when your application calls for this code. See **page 125**.

Selecting a GC Column

Gary Stidsen
GC Columns
Product Manager
11+ years of service!

did you **know**?

Restek On-The-Road training seminars are full-day courses presented in an engaging multimedia format. They are equally valuable to beginning chromatographers, those who have moderate experience and want a better understanding of the subject matter, and those interested in the "best practices" and latest technologies. **No sales pitch is presented**, just the facts on how to make your chromatography results better. The bulk of each course is lecture, but numerous demonstrations and problem-solving exercises facilitate and reinforce the understanding of important principles. See **page 11** for more information.

Capacity, k

The capacity of the column relates to how much material a column can chromatograph without adversely affecting peak shape. If the amount of a compound (mass) exceeds the capacity of a wall coated open tubular column (WCOT), the peak will front, i.e., the column will exhibit peak symmetry of less than 1, a characteristic "shark fin" shaped peak. The goal is to select a column with sufficient capacity that peak shape will not suffer.

There are two primary column related dimensions that affect capacity, assuming we have selected the proper column phase: column internal diameter (ID), and the phase film thickness (μ).

When selecting column ID, consideration should include the type of injection, the detector being used, and the concentration of sample (amount on-column). The injection technique is an important consideration because the ID of the column may need to be selected based on whether a split, splitless, cool on-column injection, or other sample transfer to the column is being used. The second consideration is how much flow the detector can optimally work under. For example, some MS detectors can only handle column flow up to 1.5mL/min.; therefore, a 0.53mm ID column, which requires higher flows for proper chromatography, is not an option for this detector. The third consideration is sample capacity. If the concentration of the sample exceeds the column capacity, loss of resolution, poor reproducibility, and peak distortion will result. Table III shows several typical column characteristics.

Film thickness (μ) has a direct affect 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, allowing them to separate. High molecular weight compounds must be analyzed on thinner film columns. This reduces the length of time the analytes stay in the column, and minimizes bleed at required higher elution temperatures. Film thickness also affects the amount of material that can be injected onto the column without overloading. A thicker film column can be used for higher concentration samples, versus a thinner film.

Table III Typical column characteristics

	Column ID				
Characteristic	**0.10mm**	**0.18mm**	**0.25mm**	**0.32mm**	**0.53mm**
Helium Flow (@ 20cm/sec.)	0.05mL/min.	0.3mL/min.	0.7mL/min.	1.2mL/min.	2.6mL/min.
Hydrogen Flow (@ 40cm/sec.)	0.09mL/min.	0.6mL/min.	1.4mL/min.	2.4mL/min.	5.2mL/min.
Sample Capacity (max load per component)	<10ng	<50ng	50–100ng	400–500ng	1000–2000ng
Theoretical Plates/Meter	8000	3700	2700	2100	1300

Efficiency, N

Column efficiency (N) is the column length divided by the height equivalent of a theoretical plate (HETP). The effective theoretical plates are affected by how well the phase has been coated onto the column walls and is measured by how narrow the peaks are when they are eluted at the end of the column. Therefore, the higher the column efficiency (N), the better resolution power the column will have.

Capillary columns are made in various lengths, typically in standard lengths of 10, 15, 30, 60, and 105 meters. Longer columns provide more resolving power, but increase analysis time. Doubling the column length increases resolution by approximately 41% (note: the column length is under the square root function). However, under isothermal conditions, it will double analysis time. In temperature-programmed analyses, retention times are more dependent on temperature than column length, with a marginal increase (approx. 10-20%) in analysis time upon doubling the column length.

reference **pages**

Choosing a Volatiles GC column
see page 563

Table of Contents for Applications
see pages 518-519

Applications by Phase Index, GC
see pages 561-562

Applications by Compound Class Index
see pages 734-735

ordering **note**

Prefer a different column cage?
5-inch column cage/Agilent 6850: add the suffix "6850" to your column catalog number. No additional cost.

Uncaged: add the suffix "051" to your column catalog number. No additional cost.

4-inch column cage (not available for 0.53mm ID columns): add the suffix "280" to your column catalog number. Additional cost

In your cage: add the suffix "031" to your column catalog number. Additional cost

What Are the Operating Temperatures for My Column?

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.

Rtx®-VMS (fused silica)

ID	df (µm)	temp. limits
0.25mm	1.40	-40 to 240/260°C
0.32mm	1.80	-40 to 240/260°C
0.45mm	2.55	-40 to 240/260°C
0.53mm	3.00	-40 to 240/260°C

Many phases list 2 maximum operating temperatures. The first temperature is the maximum isothermal operating temperature, the temperature to which the columns are guaranteed to meet the minimum bleed specification (i.e., lowest bleed level). 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). The maximum isothermal operating temperature usually is 10–20°C lower than the temperature-programmed temperature. 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 will occur.

Selection of Capillary Column Summary

Selecting the proper column for an analysis can be done by utilizing the resources available. This includes the following steps:

1) Choose proper phase

a. Review the application section of this catalog or www.restek.com for similar compound list.
b. Call Restek's experienced technical support team (800-356-1688, ext. 4) or e-mail us at:
   i. support@restek.com (in the USA)
   ii. intltechsupp@restek.com (international)
   iii. or contact your Restek representative.

2) Select column ID, film thickness, and length

a. Base consideration on:
   i. Injection technique (split, splitless, cool on-column, etc.)
   ii. Detector type (is higher flow required?)
   iii. Amount of analyte being injected onto column (sample capacity)

3) Set optimum parameters for your analysis

a. Optimize column flow (mL/min.)
b. Choose appropriate carrier gas (hydrogen, helium, or nitrogen)
c. Optimize oven temperature program