☐ *Stationary Phase Selectivity*

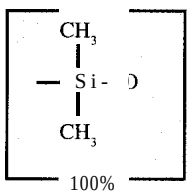
When purchasing a new column, the most important consideration is the stationary phase. There are many different interactions that occur between the analytes and the functional groups of the stationary phase. These interactions contribute more to the overall results of the analysis than any other factor in the column. That is why it is important to understand as much about your column and sample as possible.

Table I shows the chemical structure of most common stationary phases. Changes in selectivity can be observed by using a column with different functional groups as well as increasing the percentage of substitution of those functional groups. The non-polar Rtx-1 phase will preferentially retain non-polar compounds compared to polar compounds such as alcohols. As non-polar methyl units are substituted with polar functionalities such as phenyl and cyanopropyl units, the selectivity of the column shifts towards more polar compounds. In turn, non-polar compounds are retained less as there are less overall methyl units for the non-polar compounds to interact with. The Rtx-200 stationary phase contains triflouropropyl units which provide high selectivity for analytes containing lone pair electrons, such as nitro and carbonyl groups. Polyethylene glycol columns, such as Stabilwax and Rtx/MXT-WAX, are polar and are highly selctive towards polar compounds such as alcohols.

☐ *Table 2*

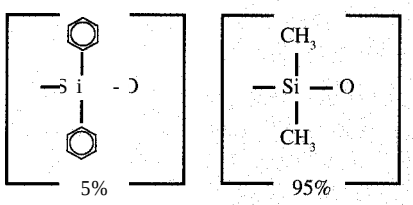
Comparison of structures, polarities, properties, and uses for each capillary column phase listed in order of increasing polarity.

Rtx/MXT-1
100% dimethyl polysiloxane



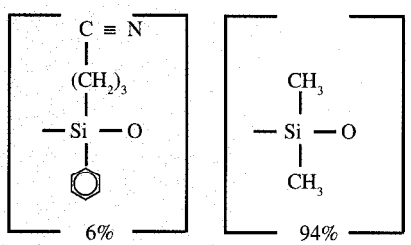
Polarity: *non-polar*
Uses: *solvents, petroleum products, pharmaceutical samples, waxes*

Rtx/MXT/XTI-5
5% diphenyl - 95% dimethyl polysiloxane



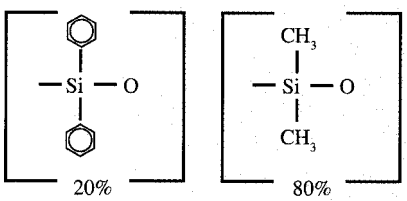
Polarity: *non-polar*
Uses: *flavors, environmental samples, aromatic hydrocarbons*

Rtx/MXT-1301, Rtx/MXT-624
6% cyanopropylphenyl
94% dimethyl polysiloxane



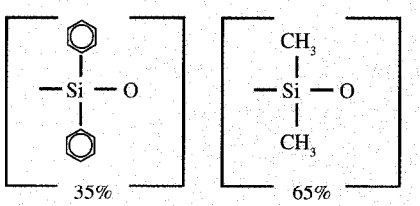
Polarity: *slightly polar*
Uses: *volatile compounds, insecticides, residue solvents in pharmaceutical products*

Rtx/MXT-20
20% diphenyl - 80% dimethyl polysiloxane



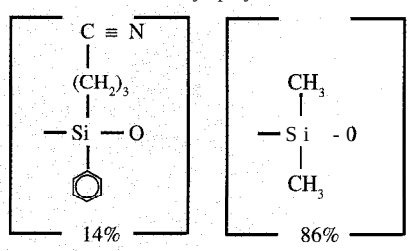
Polarity: *slightly polar*
Uses: *volatile compounds, alcohols*

Rtx/MXT-35
35% diphenyl - 65% dimethy polysiloxane



Polarity: *intermediately polar*
Uses: *pesticides, Aroclors, amines, nitrogen containing herbicides*

Rtx/MXT-1701
14% cyanopropylphenyl
86% dimethyl polysiloxane



Polarity: *intermediately polar*
Uses: *pesticides, Aroclors, alcohols oxygenates*

Table I (cont.) *Table I, listing column phase structures, is continued on page 38.*

☐ *Stationary Phase Selectivity*

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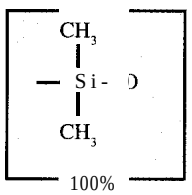
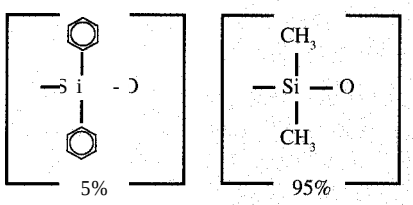
100% dimethyl polysiloxane



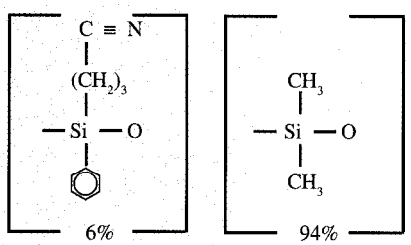
Polarity: *non-polar*
Uses: *solvents, petroleum products, pharmaceutical samples, waxes*

Rtx/MXT/XTI-5
5% diphenyl - 95% dimethyl polysiloxane



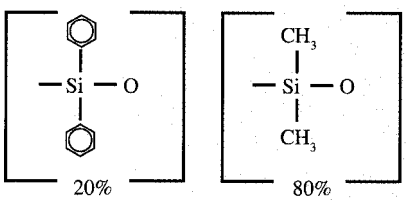
Polarity: *non-polar*
Uses: *flavors, environmental samples, aromatic hydrocarbons*

Rtx/MXT-1301, Rtx/MXT-624
6% cyanopropylphenyl
94% dimethyl polysiloxane



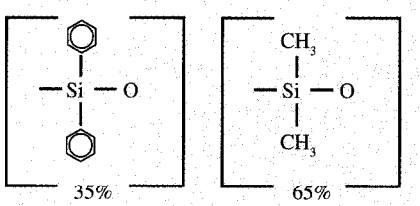
Polarity: *slightly polar*
Uses: *volatile compounds, insecticides, residue solvents in pharmaceutical products*

Rtx/MXT-20
20% diphenyl - 80% dimethyl polysiloxane



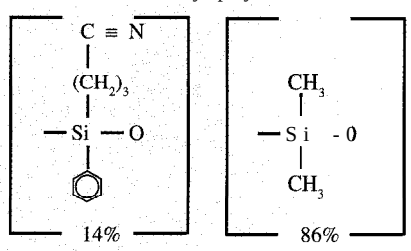
Polarity: *slightly polar*
Uses: *volatile compounds, alcohols*

Rtx/MXT-35
35% diphenyl - 65% dimethy polysiloxane



Polarity: *intermediately polar*
Uses: *pesticides, Aroclors, amines, nitrogen containing herbicides*

Rtx/MXT-1701
14% cyanopropylphenyl
86% dimethyl polysiloxane

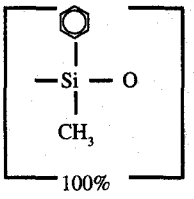


Polarity: *intermediately polar*
Uses: *pesticides, Aroclors, alcohols oxygenates*

Table I (cont.) *Table I, listing column phase structures, is continued on page 38.*

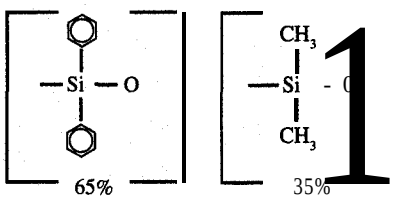
Table I (cont.) *Table I, listing column phase structures continued from page 38.*

Rtx/XT-50
50% phenyl- 50% methyl polysiloxane



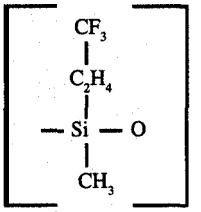
Polarity: intermediately polar
Uses: triglycerides, phthalate esters, steroids, phenols

Rtx/MXT-65TG
65% diphenyl - 35% dimthyl polysiloxane



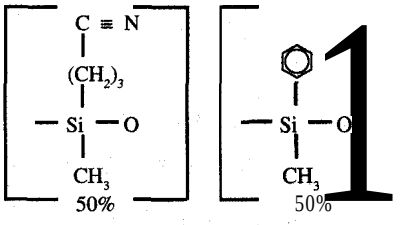
Polarity: intermediately polar
Uses: triglycerides, rosin acids, free fatty acids

RtxYMXT@-200
trifluoropropylmethyl polysiloxane



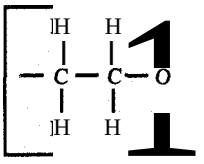
Polarity: selective for lone pair electrons
Uses: environmental samples, solvents, Freons, drugs, ketones, alcohols

Rtx®-225
50% cyanopropylphenyl
50% phenylmethyl polysiloxane



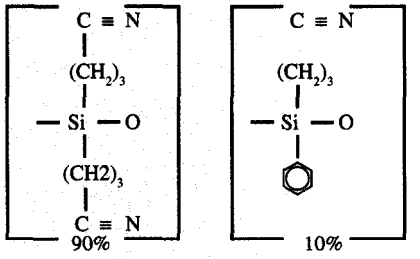
Polarity: polar
Uses: FAMEs, carbohydrates

Stabilwax®/MXT®-WAX
Carbowax® PEG



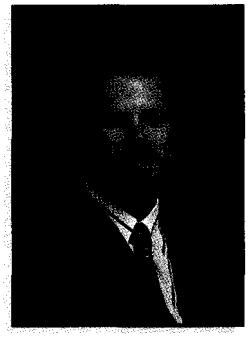
Polarity: polar
Uses: FAMEs, flavors, acids, amines, solvents, xylene isomers

Rtx®-2330
90% biscyanopropyl
10% cyanopropylphenyl polysiloxane



Polarity: very polar
Uses: FAMEs,cis/trans & dioxin isomers, rosin acids

Table II shows retention indices for the stationary phases shown in Table I. Retention indices are mathematical derivations indicating the elution point of a probe with respect to two hydrocarbons. For example, if the retention index for benzene was 650, then it would elute halfway between C6 (RI=600) and C7 (RI=700).



Rick Morehead
Fused Silica
Manufacturing Manager

Table 11

The retention indices for each phase illustrate the differences in selectivity for a variety of compounds.

Phase	Benzene	Butanol	Pentanone	Nitropropane
Rtx/MXT- 1	651	651	667	705
Rtx/MXT-5/ XTI-5/Rtx-5MS	667	667	689	743
Rtx/MXT-1301/624	689	729	739	816
Rtx/MXT-20	711	704	740	820
Rtx/MXT-35	746	733	773	867
Rtx/MXT-1701	721	778	784	881
Rtx/MXT-50	778	769	813	921
Rtx/MXT-65TG	794	779	825	938
Rtx/MXT-200	738	758	884	980
Rtx-225	847	937	958	
	963	1158	998	1230

☐ *Internal Diameter (ID)*

When selecting an internal diameter, sample concentration and instrumentation must be considered. If the concentration of the sample exceeds the column's capacity, loss of resolution, poor reproducibility, and peak distortion will result. Table III shows typical column characteristics. Note the limited capacity of narrow bore columns (0.18mm ID <50ng) versus the high capacity of 0.53mm ID columns (2000ng). Also, 0.53mm ID columns are recommended in high flow situations, such as with a purge and trap unit. Conversely, narrow bore columns can be installed directly into a MSD because of the limited flow at optimum linear velocity.

❑ *Table 111*

Typical Column Characteristics

Column ID	**0.l8mm**	**0.25mm**	**0.32mm**	**0.53mm**
Helium (flow: 20cm/sec.)	0.3cc/min.	0.7cc/min.	1.2cc/min.	2.6cc/min.
Hydrogen (flow: 40cm/sec.)	0.6cc/min.	1.4cc/min.	2.4cc/min.	2.6cc/min.
Sample Capacity	<50ng	50-100ng	400-500ng	l000-2000ng
Trenzahl Values	**40**	**30**	**25**	**15**
Theoretical Plates/Meter	**5300**	**3300**	**2700**	**1600**
Effective Plates/Meter	**3900**	**2500**	**2100**	**1200**

❑ *Film Thickness*

Film thickness has a direct effect on the retention and elution temperature for each sample compound. Thicker films retain compounds longer by maximizing the amount of time the compounds spend in the stationary phase. Thinner films retain compounds less by minimizing the amount of time the compounds spend in the stationary phase. Therefore, very volatile compounds should be analyzed on thick filmed columns to increase the time the compounds spend in the column and allow them to separate. High molecular weight compounds such as triglycerides must be analyzed on a thin film column. This minimizes the amount of time the analytes stay in the column and provide low bleed at elevated temperatures which are required when analyzing high molecular weight compounds.

Kristi
Assistant Supervisor,
Fused Silica Manufacturing

Film thickness directly effects phase ratio (beta) which is an important consideration when changing internal diameter. When internal diameter increases, film thickness (df) must increase in order to provide the similar resolution and retention. Table IV shows beta values for common dimensions of columns. Similar values indicate similar elution for different IDS.

- Taleb IV

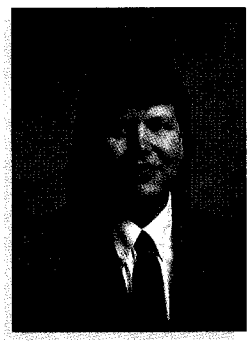
Common beta Values

Column ID	**0.10um**	**0.25um**	**0.50um**	**1.00um**	**1.50um**	**3.00um**	**5.00um**
0.18mm	**450**	**180**	**90**	**45**	**30**	**15**	**9**
0.25mm	**625**	**250**	**125**	**63**	**42**	**21**	**13**
0.32mm	**so0**	**320**		**80**	**53**	**27**	**16**
0.53mm	**1325**	**530**	**265**	**128**	**88**	**43**	**27**

The following chromatograms show a sample containing low boiling compounds analyzed on a 0.25, 1.0, and 5.0µm column with all other variables held constant. Notice that the 0.25µm column does not resolve butanol from benzene (peaks 1 & 2). The 1.0µm column provides about 80% resolution of this pair. Note that the retention times of the compounds eluting on the 0.25µm column more than double on the 1.0µm column. Now, compare the 5.0µm to the 0.25 and 1.0µm columns. The resolution between butanol and benzene (peaks 1 & 2) is not any better than the 1.0µmcolumn, and the retention times have increased six times over the 0.25µm. For this particular sample, the 1.0µm column is best. The resolution is better than the 0.25µm column and the 5.0µm column does not offer any additional improvements. If our true interest was in resolving the compounds prior to butanol (peak 1), then the 5.0µm column would be the preferred film thickness.

Would you like to know more about **WHICH COLUMN** to use for your analyses?

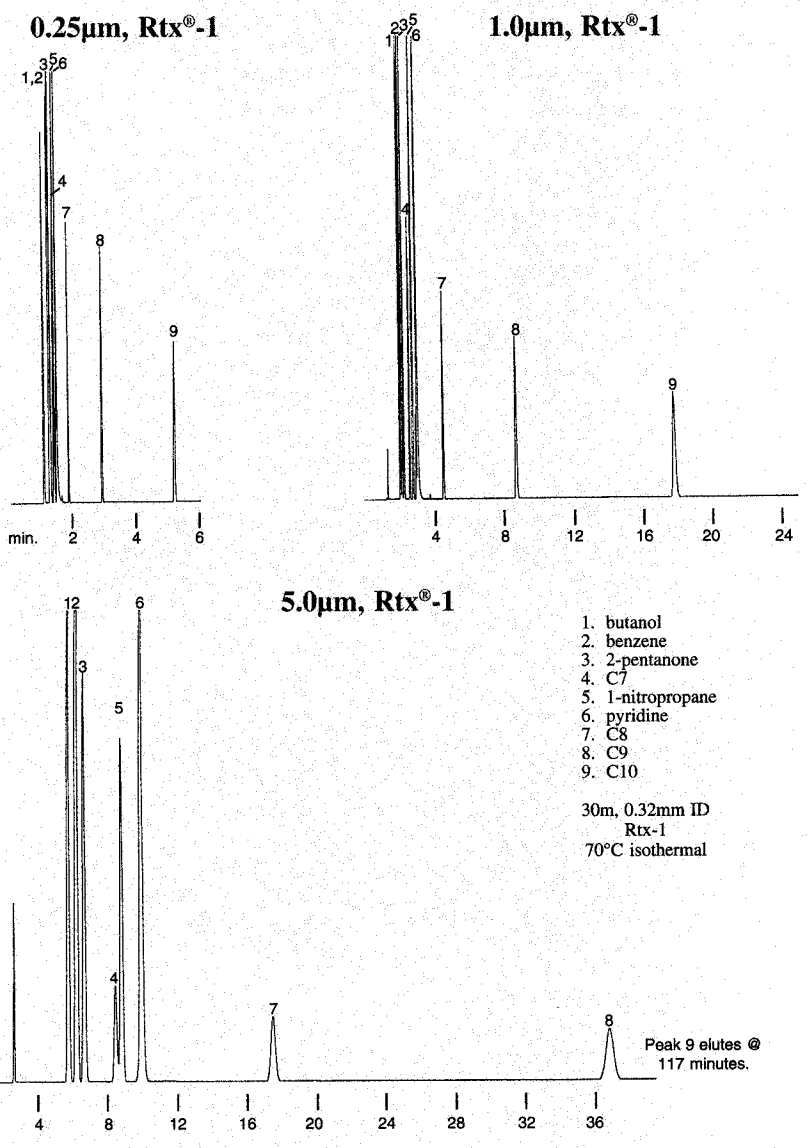
Have one of Restek's chromatography seminars presented in-house. Call your local distributor for information.



Rick Crago
Product Marketing Manager, Fused Silica

Film Thickness Effects

A sample containing low boiling components shows the differences in resolution between 0.25, 1.0, and 5.0µm columns. The 1.0µm offers better resolution than the 0.25µm and the 5.0µm does not offer any further improvements for compounds eluting after C6.



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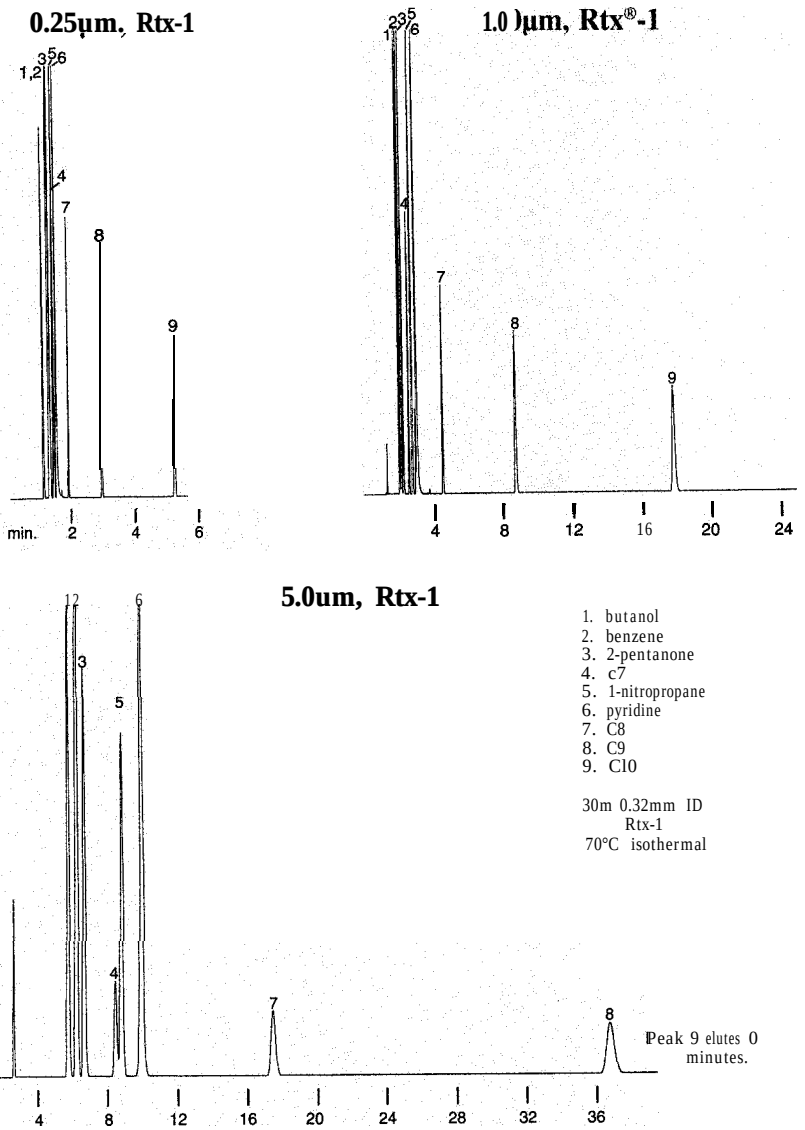


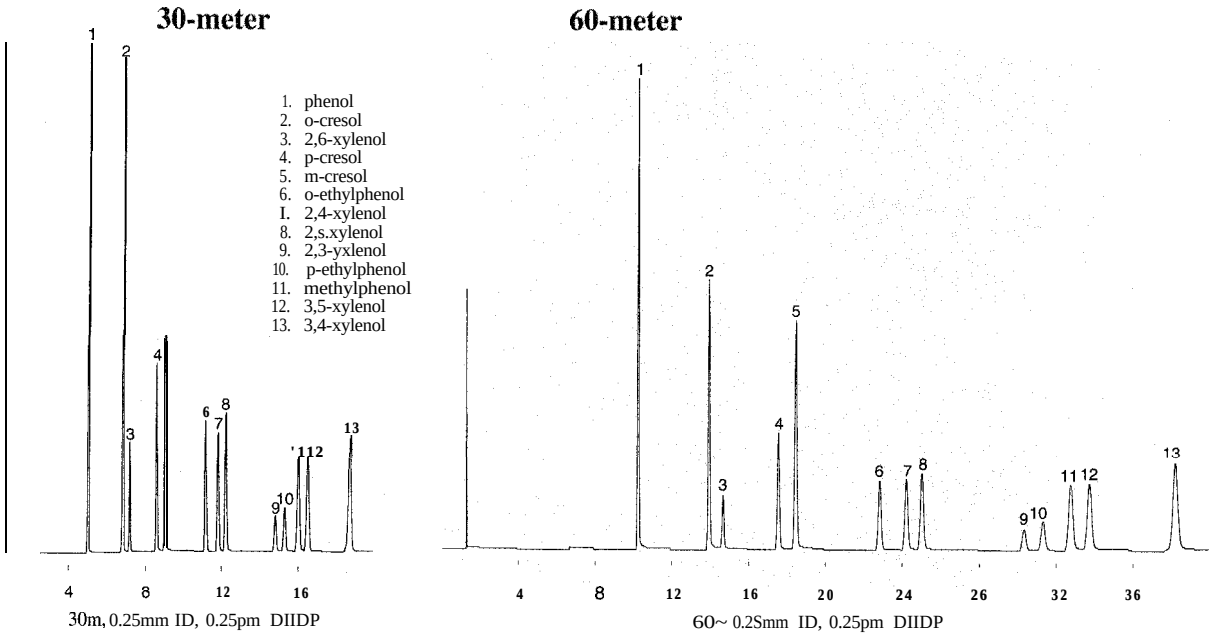
- *Length*

Longer columns provide more resolving power, increase analysis times, and cost more. Often an analyst must determine whether the amount of resolution increase is worth the extra time and expense. The benefits of using longer columns differ depending on whether isothermal or temperature programmed analyses are being performed.

For an isothermal analysis, retention time is dependent on length of the column. If the column length is doubled, the analysis time will double as well. However, the increase in resolution is only approximately 40%, since resolution is calculated using the square root of the length.

isothermal Analysis

When using a 60-meter column in an isothermal analysis, the resolution increases but the artalysis time is approximately double that of the 30-meter column.



30m, 0.25mm ID, 0.25pm DIIDP

60~ 0.2Smm ID, 0.25pm DIIDP

LENGTH EFFECTS

Length affects resolution and speed of analysis.

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

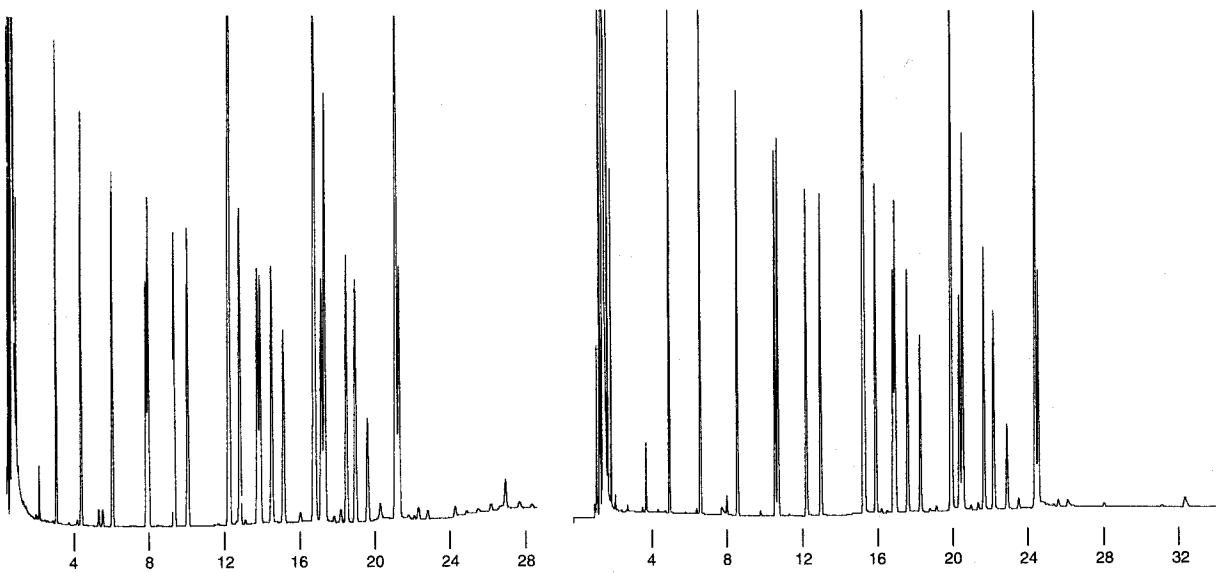
L = length
h = HETP
k = capacity factor
a = selectivity

In the case of temperature programmed analyses, retention times are more dependent on temperature than column length. The increase in resolution is the same as an isothermal run, but there is only a marginal increase in analysis time.

Temperature Programmed Analysis

When using temperature programming, 60-meter columns provide better resolution than 30-meter columns without a significant increase in analysis time.

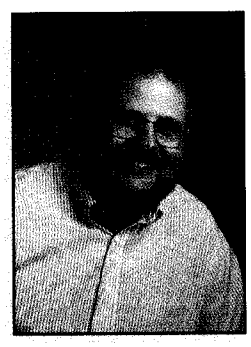
30-meter **60-meter**



30m, 0.53mm ID, 0.50µm Stabilwax®

60m, 0.53mm ID, 0.50µm Stabilwax®

30 vs. 60m column
Bacterial Acid Methyl Esters
130°C (hold 2 min.) to 250°C @ 4°C/min.



Marty Stern
Fused Silica
Manufacturing Chemist

Bob Paloskey
QA Assistant
Supervisor

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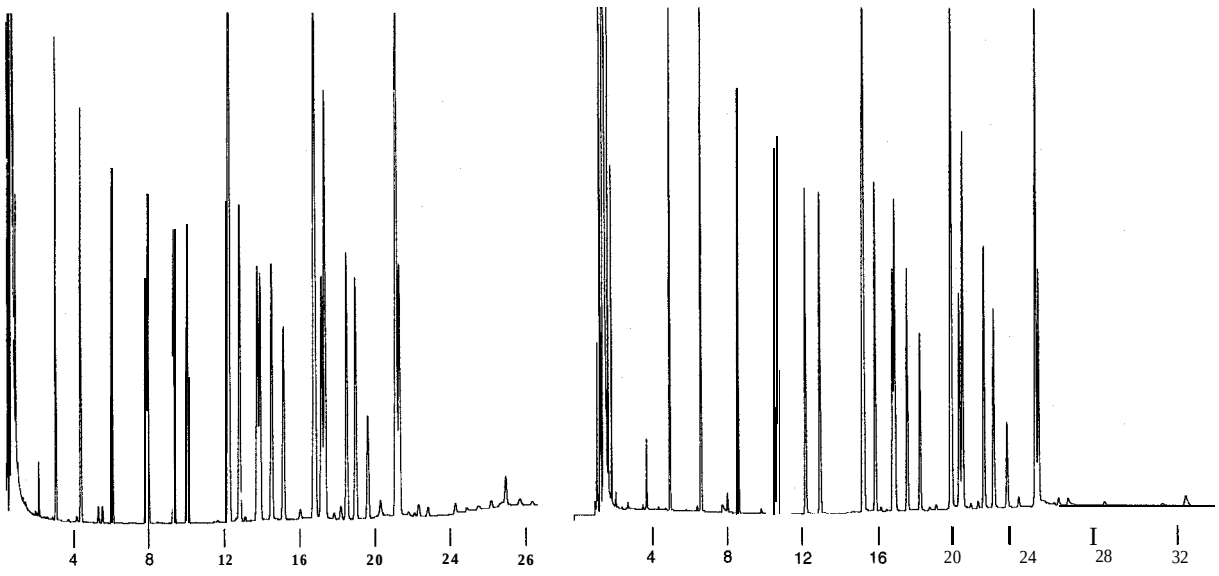
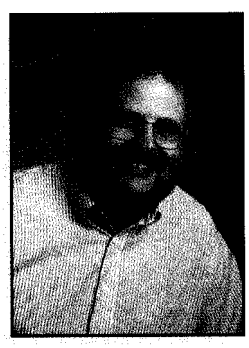
30-meter **60-meter**



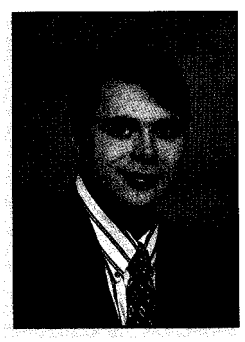
30m, 0.53mm ID, **0.50um** Stabilwax

60m 0.53mm ID, 0.50um Stabilwae

30vs.60m column
Bacterial Acid Methyl Esters
130°C (hold 2 min.) to 250°C @ 4Wmin.



Marty
Fused Silica
Manufacturing Chemist



Bob
QA Assistant
Supervisor

❑ *Custom Phases*

We have over 35 custom stationary phases in stock. Over the years, we have developed proprietary phases for specific analyst needs as well as phases written into methods. If you don't see it in the catalog, call us. Put our experienced synthesis group to work for you.

❑ *Custom Column Dimensions*

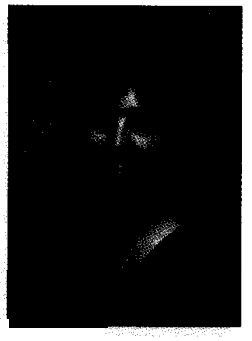
Need a specific film thickness? Locked into a method that uses a 42 meter column? If you need it, we can make it. Restek has shipped lengths from 3 to 150 meters, and film thickness' from .lpm to 10pm. If it is physically possible, we'll try it.

❑ *Special Testing*

Set the specifications for your own capillary column. Restek's knowledgeable chemists will prepare a special test mix to evaluate your column and ensure that it meets your specifications. We have provided specially tested columns to environmental testing labs, petrochemical companies, and other chromatographers using test mixtures that address their specific analytical requirements.

Special Tests	suffix
>90% Coating Efficiency	-001
Benzidines and Phenols	-002
Volatile organics	-004
Hexamethylenediamine impurities	-005
Chlorinated pesticides	-006
Morpholine	-007
Free Fatty Acids	-008
Endrin Breakdown	-009
MEA	-010
DEA	-011
Endrin/DDT Breakdown	-012
Methylamines	-013
Ethanolamines	-014
DETA	-015
Mixed Amines	-016
Certified gas concentration	-017
Soybean oil	-018

Chromatographers often say the "common" phases for capillary GC are 100% dimethyl polysiloxane, 5% diphenyl 95% dimethyl polysiloxane, and polyethylene glycol (Waxes). At Restek, we offer four different types of Rtx-1 phases, four different types of Rtx-5 phases, and four different types of wax phases-all tailored to meet the specific needs of the customer. If we don't have what you need, let us know-chances are we will offer it soon.



Cincly Ross
Customer Rkspons Team
Group Leader

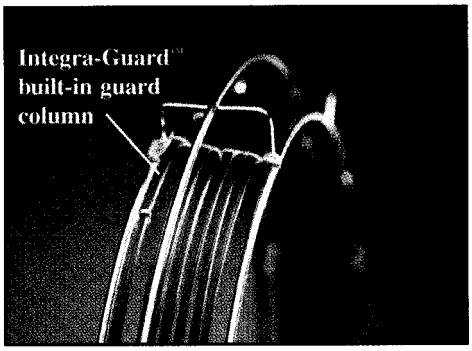
Custom Phases
(currently in stock)

Apolane 87
Apiezon' L
Butane 1.4 diol Succinate
Carbowax 300 (G20)
Carbowax 600
Carbowax 1000 (G14)
Carbowax 8000
Carbowax 20M
DEGS (G4)
DC-550 (G28)
DIIDP (G24)
Igepal CO-880@
OS-124
OS-138
OV"-202 (G6)
OV-275
OV-351 (G35)
/3p' oxydiproprionitrile
Rt-2330 (G8)
Rt-2340
SE@-30 (Gl,G2)
SE@-54 (G36)
Silar@ 5CP
Silar@ 9CP (G8)
SilaraY 1OCP
SP-1000
SP-2401 (G6)
Squalane
Squalene
Superox 20M
TCEP
Tricresyl phosphate
UCON SOHB
XE-60
W-1150

•1 Innovative Integra-Guard Columns

Guard columns WITHOUT press-tight connections-

protecting your analytical column has never been this easy!



How many times has it been said about press-fit connectors, "Some people swear by them.. .others swear at them"? For many analysts, the art of attaching a guard column to an analytical column is **a** mystery. Restek's chemists have discovered the solution to this mystery-the most reliable connection is no connection at all! No guard column system is more permanent than one continuous length of tubing containing both the guard column and the analytical column.

Restek now offers a wide variety of Integra-Guard'" capillary columns with a guaranteed leak-free connection! The column is tied using high temperature string with the guard column set apart from the analytical column. The transition area between the columns is the point at which the guard column ends and the analytical column begins. The entire setup is suspended in our unique "crush-free" cage that prevents the column from coming in contact with anything that could damage it.

The Integra-Guard column is so economical that we challenge you to beat our price with that of a conventional connection, even if you assemble it yourself. If you are currently using a guard column or considering one for the future, call your local distributor today and ask about our Integra-Guard columns.

Ordering is easy. Just add the appropriate suffix ***number and*** *price to the analytical column's catalog number and price. For example, a 30m, 0.25mm ID, 0.25um Rt@-5 with a 5-meter Integra-Guard column is cat.# 10223-124.*

mm ID	Length	Suffer #
0.25	5m	-124
	10m	-127
0.32	5m	-125
	10m	-128
0.53	5m	-126
	10m	-129

Restek-bringing You breakthroughs in column protection!

Don McCandless,
Marketing Director and 5th degree black belt in Isshinryu karate.