

# Capillary GC Column Selection and Method Development

A Primer on Column Parameters and Instrument Conditions

Updated: March 3, 2015



[sigma-aldrich.com/analytical](http://sigma-aldrich.com/analytical)

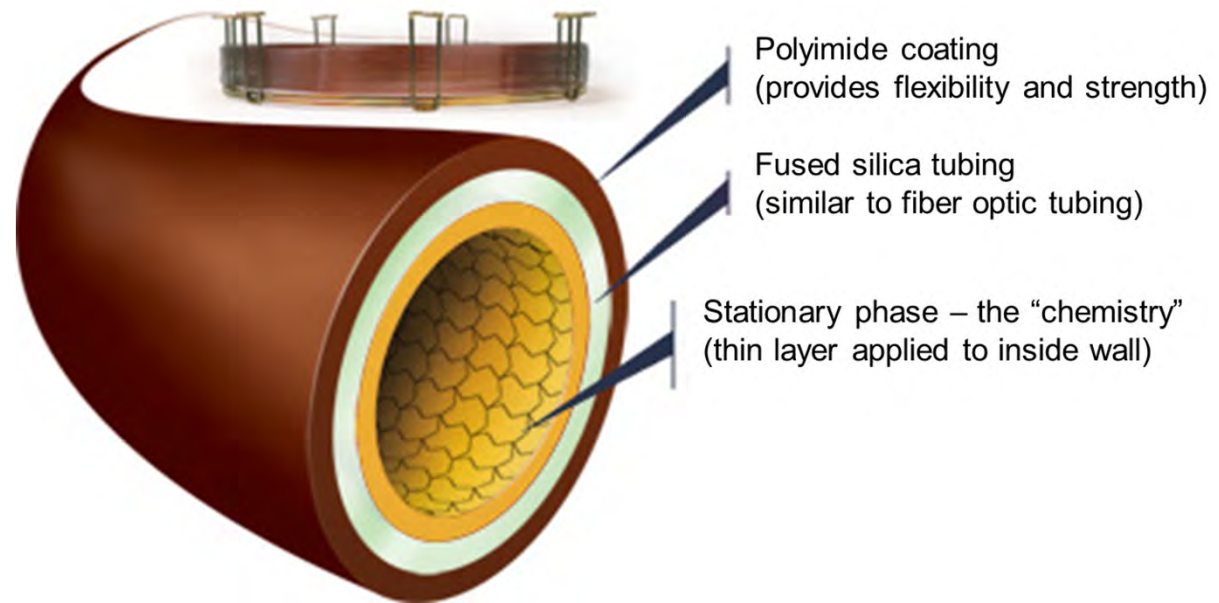
# Agenda

- **Column Selection**
  - Stationary Phase
  - Column I.D.
  - Film Thickness
  - Column Length
- **Method Development**
  - Detector Choice
  - Injection Technique
  - Carrier Gas
  - Oven Temperature
- **Chromatogram Index**
- **Summary / Resources**



# Column Selection

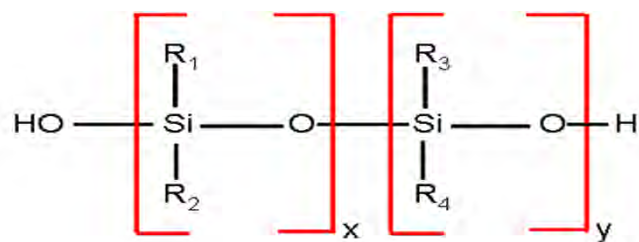
- An optimized gas chromatographic separation begins with the column
- In order, selecting the proper capillary column is based on:
  - Stationary phase
  - Column I.D.
  - Film thickness
  - Column length



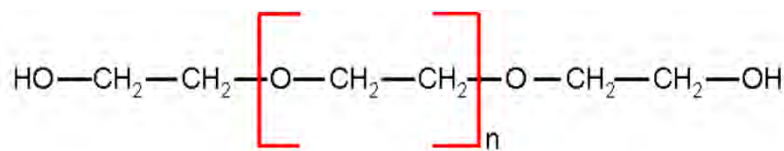
- Each parameter effects the performance of the column

Note that the information which follows is general.  
Specific situations may warrant exceptions to these guidelines.

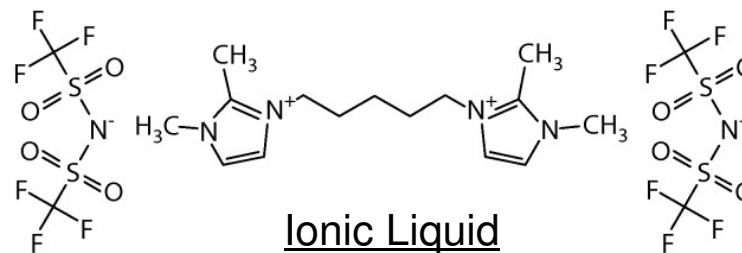
# Step 1. Stationary Phase Overview



Polysiloxane Polymer



Polyethylene Glycol



- Each stationary phase can undergo a **specific combination** of interactions (dispersive,  $\pi$ - $\pi$ , dipole-dipole, acidic, basic, hydrogen bonding, etc.) with **specific relative amounts** of each
- Differences in chemical/physical properties of analytes and their interactions with the stationary phase are the **basis of the separation process**
  - **Retention time** is a measure of all analyte-phase interactions
  - **Separation** achieved when **strength** of analyte-phase interactions **differ** for analytes

Increasing temperature will weaken all analyte-phase interactions, but at different rates.

# Step 1. Stationary Phase

## Where to Start?

- Established Applications

- Use a phase that is stated in an existing method (**copy from others**)

<OR>

- Use a phase recommended by a column manufacturer (**use what others have learned**)

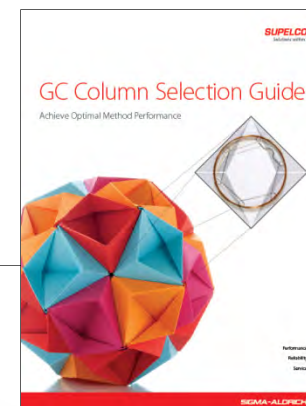
- New Applications

- No existing method to provide guidance
- Must have some knowledge of the chemistry of the analytes
- Phase selection is simply based on the general chemical principle that “likes dissolves like”

Many column manufacturers publish column selection charts.  
The next few slides highlight our column selection strategy for established applications.

# Step 1. Stationary Phase

## Established Applications (Brochure)



### Environmental Industry

	SPB®-Octyl	SLB®-5ms	Equity®-5	SPB-624	VOCOL®	Equity-1701	SPB-608	Sup-Herb™	SPB-35	SPB-50	SPB-225	SPB-1000	SLB-IL59	SLB-IL60	SLB-IL82	SP™-2331	SLB-IL111	Chiral *
Volatiles by GC-MS				●	●													
Volatiles by GC				●	●													
Semivolatiles by GC-MS		●																
Semivolatiles by GC**		●	●			●	●	●	●	●								●
Fuels (GRO, DRO, TPH)		●	●	●	●													
Dioxins by GC-HRMS		●									●						●	
PCBs by GC-HRMS	●	●											●		●		●	
PBDEs by GC-MS		●																
PAHs by GC or GC-MS		●					●		●	●			●	●				
Oil Spill Dispersants												●						
Odor Compounds (Geosmin, 2-MIB)		●																

\* See "by Application" section  
 \*\* Includes: organochlorine pesticides, PCBs as Aroclors, herbicides, organophosphorous pesticides, nitrosamines, phenols, phthalate esters, haloacetic acids, disinfection by-products and solvents, and PAHs.

28 page, 4 color brochure with charts for 13 industries.  
 GC Column Selection Guide (T407133 KCX) can be downloaded from our web site.

# Step 1. Stationary Phase

Established Applications ([sigma-aldrich.com/gc-columns](http://sigma-aldrich.com/gc-columns))

Gas Chromatography

## Capillary GC Columns

An optimized chromatographic separation begins with selection of the proper column for the application. All our capillary GC columns are organized by industry/application, by method reference, by phase polarity, and also by name. Many column types (ionic liquid, MS-grade, Fast GC, GCxGC, chiral, PLOT, and SCOT) are broken out separately. Our GC Column Selection Guide is a valuable resource, with useful instructions for column selection.

Supelco is dedicated to the development of leading edge GC column technology to meet the needs of customers. Our goal is to provide the finest products, backed by superior service. Browse through our selection of online GC chromatograms or contact us ([techservice@sial.com](mailto:techservice@sial.com)) if you require further assistance with column selection for your specific application.

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[Capillary GC Columns, by industry / application](#)

[Capillary GC Columns, by method reference](#)

[Capillary GC Columns, by phase polarity](#)

[Capillary GC Columns, by name](#)

[Ionic Liquid Capillary GC Columns](#)

[MS-Grade Capillary GC Columns](#)

[Fast GC Capillary GC Columns](#)

[GCxGC Capillary GC Columns](#)

[Chiral Capillary GC Columns](#)

[PLOT Capillary GC Columns](#)

[SCOT Capillary GC Columns](#)

[Guard Columns/Retention Gaps](#)

See also: [Packed GC Columns](#)

[How to Choose a Capillary GC Column](#)

by industry / application

# Step 1. Stationary Phase

Established Applications ([sigma-aldrich.com/gc-columns](http://sigma-aldrich.com/gc-columns))

## Capillary GC Columns, by industry / application

Gas chromatography, first established in the 1950's, is a mature analytical technique with many established applications. Therefore, it is probable that documented methods or journal articles exist stating which stationary phases have successfully been used for a given application. Today, GC is the preferred chromatographic technique in the environmental, petroleum, chemical, flavor & fragrance, and forensic industries. In the biofuel, agriculture, food & beverage, cosmetic and personal care/cleaning product, and clinical industries, its frequency of use is equal to other chromatographic techniques. It is used for specific chromatographic applications in the industrial hygiene, pharmaceutical, and life science industries.

We have identified the GC columns that are routinely used in the various industries. These are conveniently arranged by industry, and then application within that industry, to simplify the process of selecting the proper phase. First, follow the link that matches your industry. Then, locate the application within the text to identify recommended column phases.

[View ongoing GC Special Offers](#)

- Environmental Industry - (142)
- Industrial Hygiene Industry - (10)
- Petroleum Industry - (37)
- Biofuel Industry - (10)
- Chemical Industry - (155)
- Agriculture Industry - (95)
- Food & Beverage Industry - (191)
- Flavor & Fragrance Industry - (98)
- Cosmetic and Personal Care/Cleaning Product Industry - (13)
- Pharmaceutical Industry - (36)
- Clinical Industry - (52)
- Forensic Industry - (23)
- Life Science Industry - (4)

Biofuel Industry



# Step 1. Stationary Phase

Established Applications ([sigma-aldrich.com/gc-columns](http://sigma-aldrich.com/gc-columns))

## Biofuel Industry

These columns can be used to measure purity and to monitor for impurities in biofuels.

- For bioethanol (ethanol content), choose Petrocol DH 150
- For biodiesel (FAME profile), choose Omegawax, SLB-IL60, and SLB-IL111
- For biodiesel (glycerin impurity), choose MET-Biodiesel
- For biodiesel (methanol impurity), choose Equity-1

### ■ Chromatograms

- Petrocol DH 150 - (2)
- Equity-1 - (36)
- MET-Biodiesel - (2)
- Omegawax - (5)
- SLB-IL60 - (7)
- SLB-IL111 - (5)

# Step 1. Stationary Phase

## Where to Start?

- Established Applications

- Use a phase that is stated in an existing method (copy from others)

<OR>

- Use a phase recommended by a column manufacturer (use what others have learned)

- New Applications

- No existing method to provide guidance
- Must have some knowledge of the chemistry of the analytes
- Phase selection is simply based on the general chemical principle that “likes dissolves like”

The next few slides highlight our column selection strategy for new applications.

# Step 1. Stationary Phase

## New Applications: Non-Polar Analytes

Compound Polarity	Compound Examples	Recommended Phases
Non-Polar		
C and H atoms only, C-C bonds	alkanes	Petrocol, SPB-Octyl, Equity-1, SPB-1, SLB-5ms, Equity-5, SPB-5

- Analysis using a **non-polar phase**
  - Interactions primarily dispersive (van der Waals forces), but phases with phenyl functional groups can also undergo some  $\pi$ - $\pi$  interactions
  - Elution order generally follows the boiling points of the analytes

Derivatization can be used to modify analyte polarity in addition to volatility.

# Step 1. Stationary Phase

## New Applications: Polar Analytes

Compound Polarity	Compound Examples	Recommended Phases
<b>Polar</b>		
Primarily C and H atoms, also contain Br, Cl, F, N, O, P and/or S	alcohols, amines, carboxylic acids, diols, esters, ethers, ketones, thiols	SPB-624, OVI-G43, VOCOL, SPB-20, Equity-1701, SPB-35 SPB-50, SPB-225, PAG, Omegawax, SPB-1000, Nukol, SUPELCOWAX 10

- Analysis using an **intermediate polar phase** or a **polar phase**
  - Dispersive,  $\pi$ - $\pi$ , dipole-dipole, and dipole-induced dipole interactions are all strong
  - Elution order determined by the overall effects of all possible interactions

Derivatization can be used to modify analyte polarity in addition to volatility.

# Step 1. Stationary Phase

## New Applications: Polarizable Analytes

Compound Polarity	Compound Examples	Recommended Phases
Polarizable		
C and H atoms only, C=C or C≡C bonds	alkenes, alkynes, aromatic hydrocarbons	SP-2330, SP-2331, SP-2380, SP-2560, SP-2340, TCEP

- Analysis using a **highly polar phase** or an **extremely polar phase**
  - Same interactions as intermediate and polar phases (dispersive,  $\pi$ - $\pi$ , dipole-dipole, and dipole-induced dipole), plus hydrogen bonding and basic interactions
  - Elution order determined by the overall effects of all possible interactions

Derivatization can be used to modify analyte polarity in addition to volatility.

# Step 1. Stationary Phase

New Applications ([sigma-aldrich.com/gc-columns](http://sigma-aldrich.com/gc-columns))

## Gas Chromatography

### Capillary GC Columns

An optimized chromatographic separation begins with selection of the proper column for the application. All our capillary GC columns are organized by industry/application, by method reference, by phase polarity, and also by name. Many column types (ionic liquid, MS-grade, Fast GC, GCxGC, chiral, PLOT, and SCOT) are broken out separately. Our GC Column Selection Guide is a valuable resource, with useful instructions for column selection.

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[Capillary GC Columns, by industry / application](#)

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[Guard Columns/Retention Gaps](#)

See also: [Packed GC Columns](#)

[How to Choose a Capillary GC Column](#)

by phase polarity

# Step 1. Stationary Phase

New Applications ([sigma-aldrich.com/gc-columns](http://sigma-aldrich.com/gc-columns))

## Capillary GC Columns, by phase polarity

Choosing a stationary phase is the most important step in choosing a column, and should be selected based on the application to be performed. It is recommended to first consult our "Capillary GC Columns, by industry/application" section to determine if we have already identified appropriate columns. For new applications, there is often no existing reference to provide guidance. In these method development instances, one must have some knowledge of the chemistry of the compounds to be analyzed. Phase selection is based on the general chemical principle that "likes dissolves like" and relates to the specific analyte-stationary phase interactions that each group of columns can perform. Choose:

- [Non-polar GC columns](#) for non-polar compounds (such as alkanes) that contain 1) only carbon and hydrogen atoms, and 2) only single bonds between carbon atoms.
- [Intermediate polar GC columns](#) for an alternate selectivity of non-polar and/or polar compounds.
- [Polar GC columns](#) for polar compounds (such as alcohols, amines, carboxylic acids, diols, esters, ethers, ketones, and thiols) that contain 1) primarily carbon and hydrogen atoms, and 2) also some bromine, chlorine, fluorine, nitrogen, oxygen, phosphorus, and/or sulfur atoms.
- [Highly polar GC columns](#) for polarizable compounds (such as alkenes, alkynes, and aromatic hydrocarbons) that contain 1) only carbon and hydrogen atoms, and 2) some double and/or triple bonds between carbon atoms.
- [Extremely polar GC columns](#) for additional selectivity of polarizable compounds.

View ongoing GC Special Offers

- [Non-Polar Capillary GC Columns - \(246\)](#)
- [Intermediate Polar Capillary GC Columns - \(52\)](#)
- [Polar Capillary GC Columns - \(102\)](#)
- [Highly Polar Capillary GC Columns - \(37\)](#)
- [Extremely Polar Capillary GC Columns - \(22\)](#)

Polar Columns

# Step 1. Stationary Phase

New Applications ([sigma-aldrich.com/gc-columns](http://sigma-aldrich.com/gc-columns))

## Polar Capillary GC Columns

Polar GC columns are made using polar stationary phases, the most common being polyethylene glycol and modified versions. These columns are commonly used to separate polar analytes (such as alcohols, amines, carboxylic acids, diols, esters, ethers, ketones, and thiols) that contain 1) primarily carbon and hydrogen atoms, and 2) also some bromine, chlorine, fluorine, nitrogen, oxygen, phosphorus, and/or sulfur atoms. Elution order is determined by differences in the overall effects of possible interactions.

- Dispersive (van der Waals forces),  $\pi$ - $\pi$ , dipole-dipole, and dipole-induced dipole interactions are all strong with these columns.
- Moderate amounts of hydrogen bonding and basic interactions are also possible.
- SPB-1000 and Nukol columns are specially-engineered to also allow strong acidic interactions.
- Carbowax amine columns are specially-engineered to also allow strong basic interactions.

### ■ Chromatograms

- SPB-225 - (3)
- SPB-PUFA - (3)
- PAG - (4)
- SPB-1000 - (4)
- Nukol - (13)

- Carbowax Amine - (4)
- Omegawax - (5)
- SUPELCOWAX 10 - (29)
- SLB-IL59 - (3)
- SLB-IL60 - (7)
- SLB-IL61 - (3)



## Step 2. Column I.D.

### Overview

- Efficiency vs. sample capacity
- Optimizing one requires a sacrifice from the other

#### • Higher Efficiency

- Narrower, sharper peaks
- How? Decrease column I.D.
- When to optimize for it
  - Trace analysis
  - Many analytes
  - Analytes that elute closely together

#### • Increased Sample Capacity

- More mass of each analyte that can be injected
- How? Increase column I.D.
- When to optimize for it
  - Analytes at high concentrations
  - Analytes at a wide range of concentrations

Polarity of analytes and polarity of the phase will affect sample capacity (e.g. non-polar phases have higher capacities for non-polar analytes, and lower capacities for polar analytes).

## Step 2. Column I.D.

### Effects of Changing Column I.D.

Internal Diameter (mm)	Efficiency: Plates/Meter (N/m)	Efficiency: Total Plates (N)	Capacity Each Analyte (ng)
0.53	1,300	39,000	1000-2000
0.32	2,300	69,000	400-500
0.25	2,925	87,750	50-100
0.20	3,650	109,500	<50
0.18	4,050	121,500	<50
0.10	7,300	219,000	<10

Theoretical values for 30 m long columns, calculated with  $k = 6.00$  and 85% coating efficiency

High split ratios limit the mass of analytes entering small I.D. columns (still provide high efficiency).

Want to learn more? Visit [sigma-aldrich.com/fastgc](http://sigma-aldrich.com/fastgc)

## Step 3. Film Thickness

### Phase Ratio ( $\beta$ )

- Film thickness is interdependent with column I.D.
- **Phase ratio** (beta,  $\beta$ ) is the ratio of gas volume and stationary phase volume

$$\beta = \frac{\text{column radius } (\mu\text{m})}{2 \times \text{film thickness } (\mu\text{m})}$$

- As a general rule, select columns by their  $\beta$  values

$\beta$ Value	Uses
<100	Highly volatile, low molecular weight compounds
100–400	General purpose analyses Wide range of compounds
>400	High molecular weight compounds Trace analyses

## Step 3. Film Thickness

### Benefits and Drawbacks of Changing Film Thickness

- Decrease Film Thickness (**higher  $\beta$** )

- **Benefits**

- Sharper peaks (unless overloaded)
- Lower column bleed
- Increased maximum temperature

- **Drawbacks**

- Increased analyte-tubing interaction
- Decreased sample capacity

- Increase Film Thickness (**lower  $\beta$** )

- **Benefits**

- Decreased analyte-tubing interaction
- Increased sample capacity

- **Drawbacks**

- Wider peaks
- Increased column bleed
- Decreased maximum temperature

In contrast to relative terms (“thick” and “thin”),  $\beta$  values establish a distinct ranking for columns.

## Step 4. Column Length

### The Last Parameter to Consider

- Increasing Column Length

- Greater resolution, but increased back pressure and longer run
- Doubling length will NOT double resolution

- Decreasing Column Length

- Shorter run and lower back pressure, but decreased resolution
- Use when resolution is in excess (Fast GC) or not critical (screening)

Column Length (m)	Inlet Pressure (psi)	Peak 1 Retention (min)	Peak 1/2 Resolution (R)	Efficiency: Total Plates (N)
15	5.9	8.33	0.8	43,875
30	12.0	16.68	1.2	87,750
60	24.9	33.37	1.7	175,500

Theoretical values for 0.25 mm I.D. columns with 85% coating efficiency, 145 °C isothermal analyses, helium at 21 cm/sec, k (peak 1) = 6.00

If resolution between a critical pair is less than 1, doubling length will not bring it to baseline (a resolution value of at least 1.5).

# Method Development

- Optimized method = column parameters + instrument conditions
- Topics to be discussed
  - Detector choice
  - Injection technique
  - Carrier gas
  - Oven temperature



Note that the information to follow is basic.  
It is intended for GC analysts relatively new to the technique.

## Detector Choice

Detector	Common uses	Destructive?
<b>FID</b> (Flame Ionization Detector)	Organic compounds (some C and H atoms), hydrocarbons	Yes
<b>ECD</b> (Electron Capture Detector)	Chlorinated pesticides and PCBs	No
<b>MS</b> (Mass Spectrometer)	All	Yes
<b>TCD</b> (Thermal Conductivity Detector)	Permanent gases	No
<b>PID</b> (Photo Ionization Detector)	Aromatics	No
<b>FPD</b> (Flame Photometric Detector)	Sulfur-containing compounds	Yes
<b>NPD</b> (Nitrogen Phosphorus Detector)	Organophosphorus pesticides, nitrogen-containing herbicides	Yes
<b>ELCD</b> (Electrolytic Conductivity Detector)	Chlorinated hydrocarbons	Yes

# Injection Technique

## Overview

- Convert **extract or sample** to a **vapor cloud** that the GC system can handle
- Four primary injection techniques are used in GC
  - Split
  - Splitless
  - On-column
  - Direct





# Injection Technique

## Practical Considerations

- Syringe

- Large enough so not filled to capacity
- Extract/sample should occupy at least 10% of the syringe volume

- Inlet Liner

- Use the correct design for the injection technique
- Match the deactivation with the application

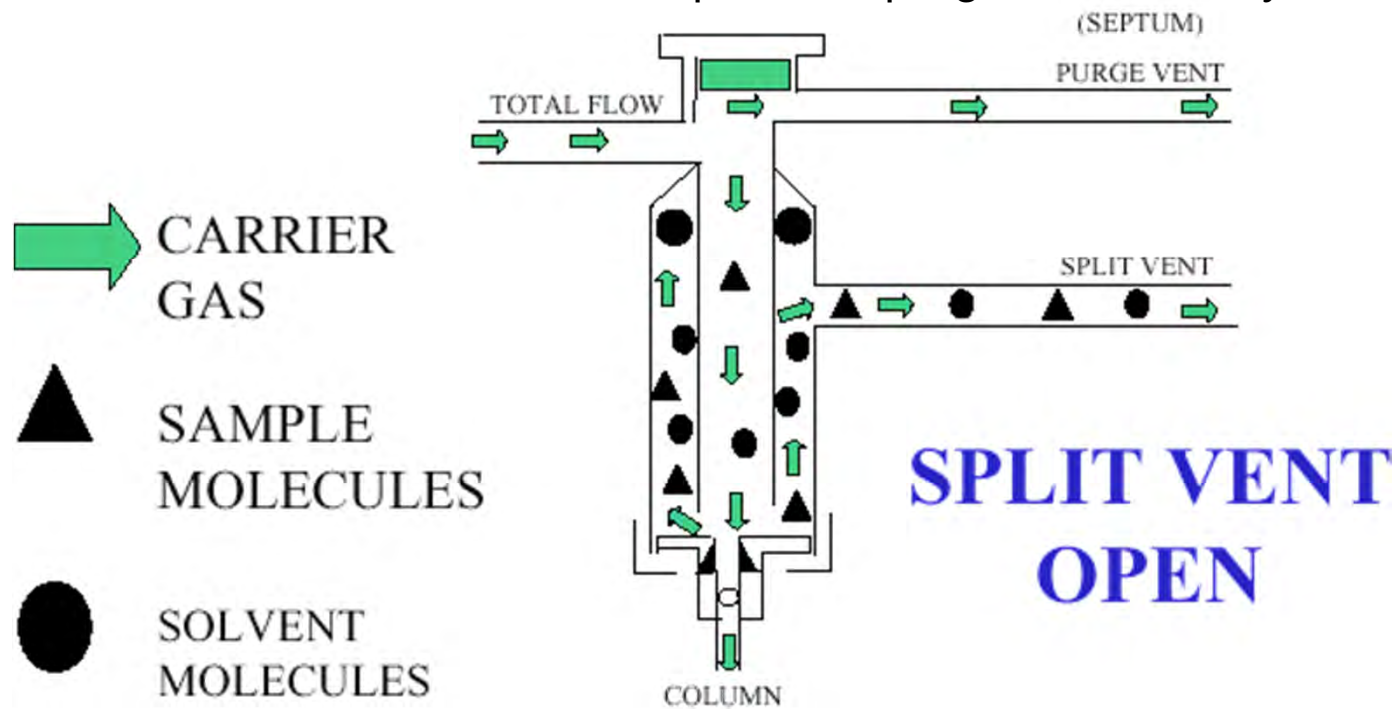
- Reproducibility

- Use an autosampler when able
- For manual injections...
  - Use a Chaney adapter
  - Be smooth and rapid

# Split Injection

Useful for High, Varied, or Unknown Concentrations

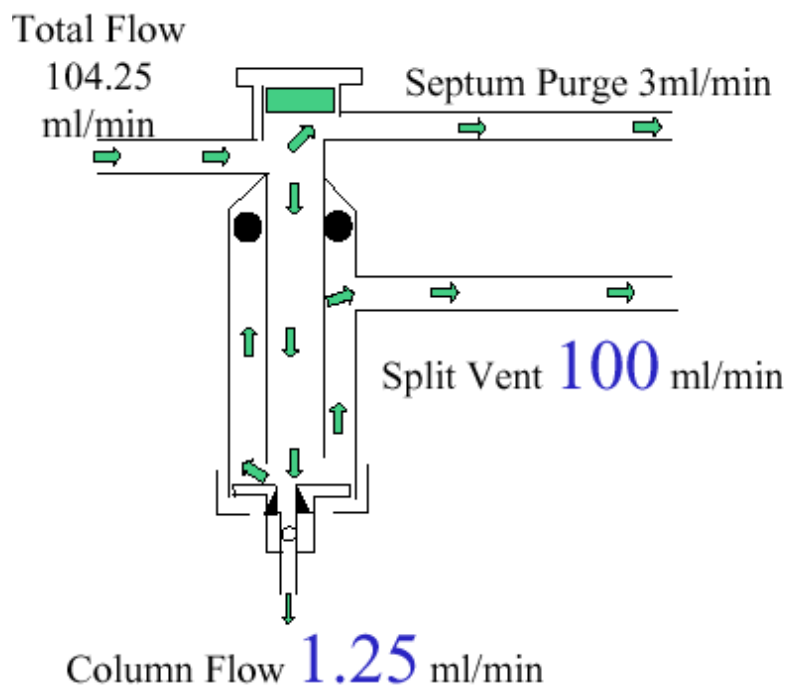
- Extract or sample is vaporized, mixed with carrier gas, then **split in the inlet**
- Limit amount of material reaches column (prevents overload + fronting peaks)
- A small portion flows to the column while the bulk is vented away
- Can be used with isothermal or temperature programmed analysis



# Split Injection

## Split Ratio

- Total flow = septum purge + column flow + split vent flow
- Split ratio = split vent flow / column flow



### Split Ratio Calculation

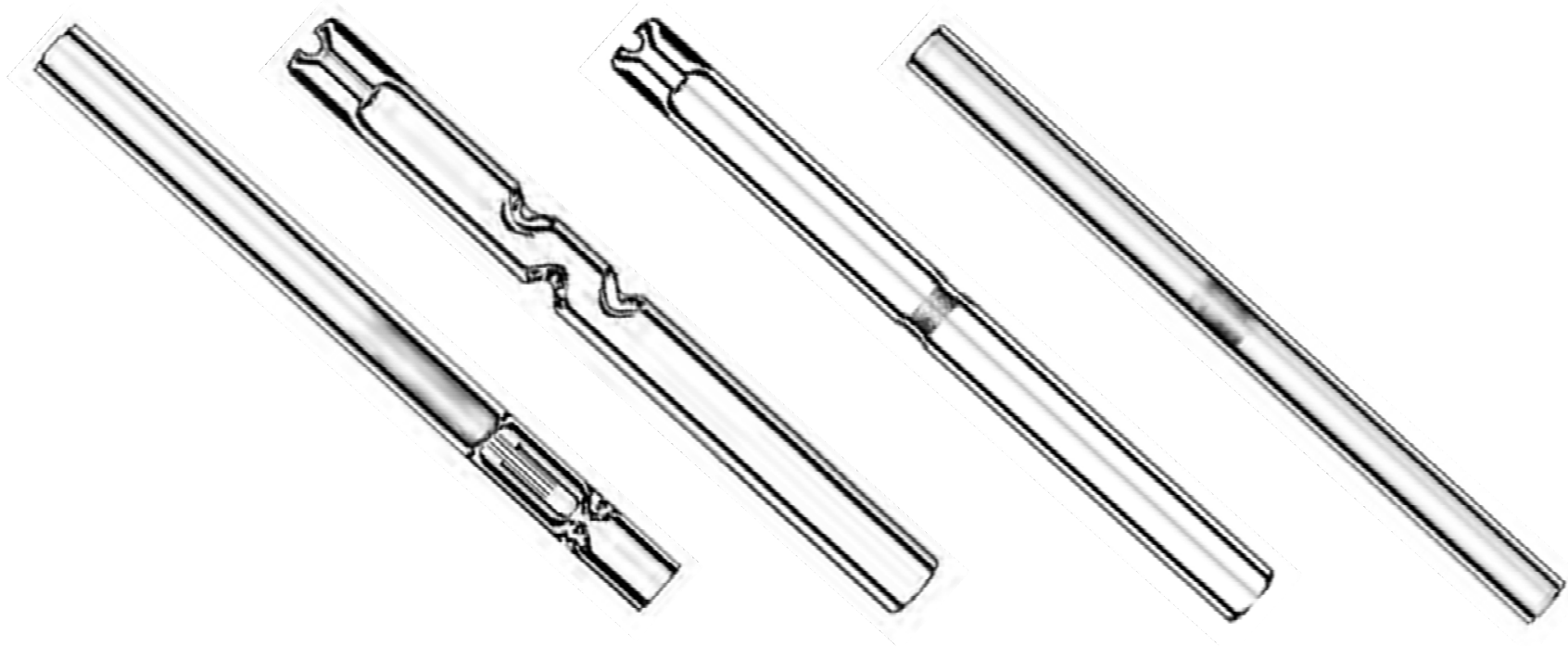
$$\begin{aligned} &= \frac{\text{Split Vent Flow}}{\text{Column Flow}} \\ &= \frac{100}{1.25} \\ &= 80 \text{ to } 1 \end{aligned}$$

Split ratios range from <5:1 (wide bore column applications) to >400:1 (Fast GC applications).  
User should experimentally determine which split ratio works best for their application.

# Split Injection

## Liner Choices

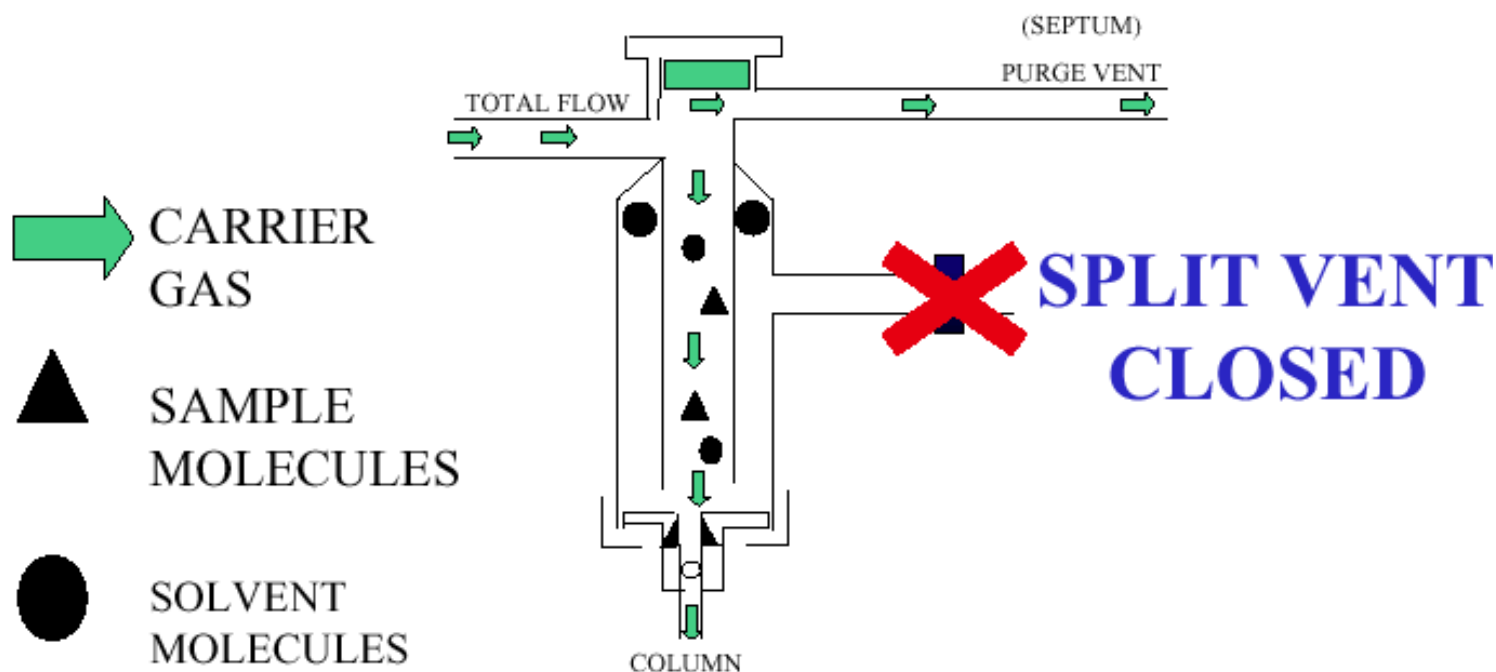
- Cups, baffles, twists, or frits establish turbulent flow rather than laminar flow
- **Facilitate sample mixing** prior to the point where the sample is split
- Wool may be used to improve vaporization
- 2-4 mm I.D. inlet liners typically used (2 mm I.D. = higher linear velocity)



# Splitless Injection

## Useful for Trace Analysis

- Extract or sample is vaporized then mixed with carrier gas in the inlet
- Carrier gas **transfers entire vapor cloud** into the column
- Split vent opens to purge inlet after 1.5-2 inlet volumes have passed through
- Can be used with isothermal or temperature programmed analysis



# Splitless Injection

## When to Open the Split Vent?

- Split vent opens to purge the injection port **after 1.5-2 inlet volumes** have passed through
- Open **too soon...**
  - Loss of response (especially for higher molecular weight analytes)
- Open **too late...**
  - Too much solvent enters column (may swamp early eluting analytes)



Typical times range from 30 seconds to 2 minutes.  
User should experimentally determine which time works best for their application.

# Splitless Injection

## Liner Choices

- Dwell time longer, so **do not need to create high turbulence**
- Tapers may help focus analytes onto column
- Wool may be used to improve vaporization
- 2-4 mm I.D. inlet liners typically used (2 mm I.D. = higher linear velocity)
- **Deactivation** of liner **very important** due to the long residence time



# Splitless Injection

## Inlet Liner Volume

- Inlet liner must contain the vapor cloud formed after all the solvent vaporizes
- If not, can cause poor reproducibility, ghost peaks, and poor peak shapes



4 mm I.D., Single taper: 850  $\mu\text{L}$



4 mm I.D., Single taper w/wool 880  $\mu\text{L}$



4 mm I.D., Straight w/wool: 985  $\mu\text{L}$

Liner  
Volume

1 $\mu\text{L}$ liquid to gas volume ( $\mu\text{L}$ )		200 $^{\circ}\text{C}$ Inlet		300 $^{\circ}\text{C}$ Inlet	
	B.P. ( $^{\circ}\text{C}$ )	10 psi	30 psi	10 psi	30 psi
Methylene chloride	40	360	200	437	241
Methanol	64.5	570	315	691	382
Water	100	1279	706	1548	855

Resulting  
vapor volume  
of solvent

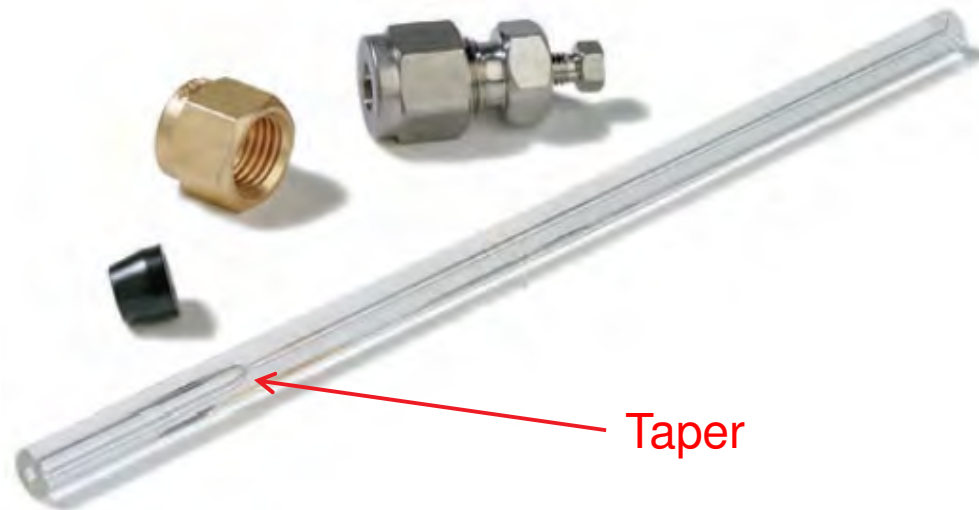
Use an inlet liner with an internal volume equal to or larger than the expansion volume of the solvent.



## On-Column Injection (Cool On-Column Injection)

Useful when Analytes have Vastly Different Boiling Points

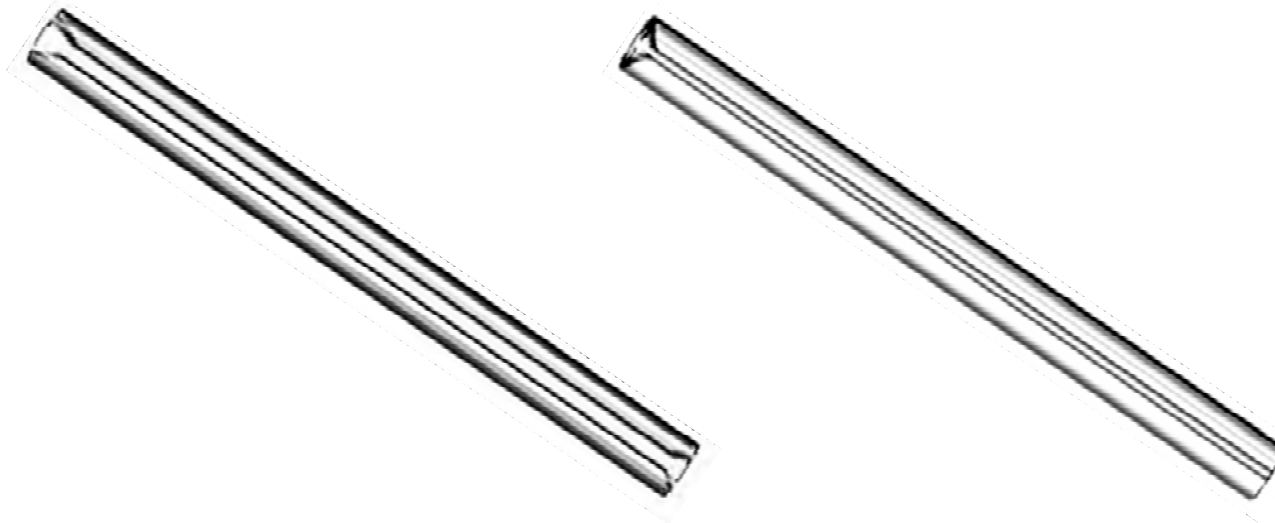
- Liquid extract or sample **deposited directly** into a 0.53 mm I.D. column
- Inlet liner with tapered region
  - Creates seal between column and liner
  - Guides the needle into the column (special syringe required)
- Must be used with temperature programmed analysis
- Eliminates splitter discrimination (inaccurate quantification)



# Direct Injection

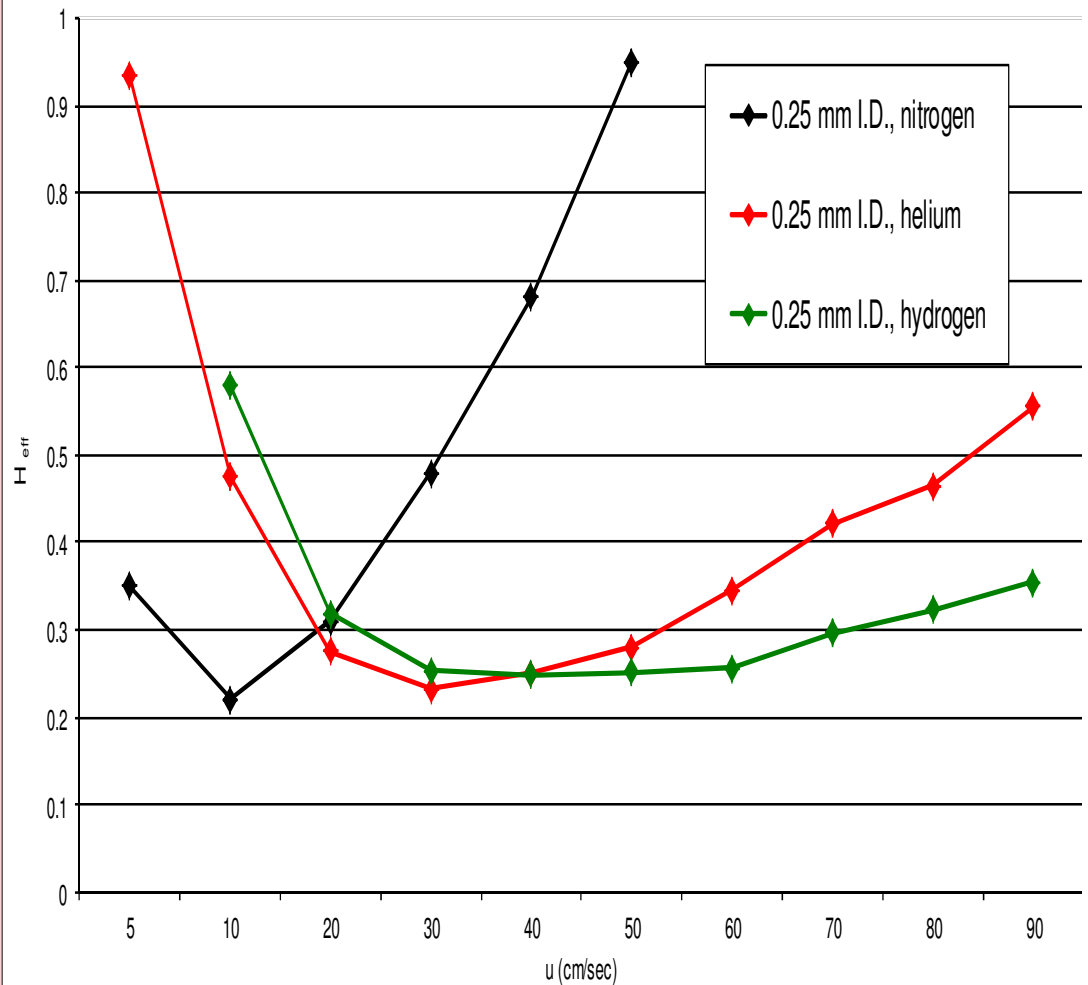
## Useful for Gas Phase Samples

- Use with headspace, purge and trap, and solid phase microextraction (SPME)
- Entire **vapor cloud transferred** to the column
- Can be used with isothermal or temperature programmed analysis
- No solvent, so little solvent expansion (large inlet liner volumes not needed)
- Narrow bore 0.5-1.5 mm I.D. inlet liners are used to maintain a high linear velocity through the injection port, minimizing band broadening



# Carrier Gas Options

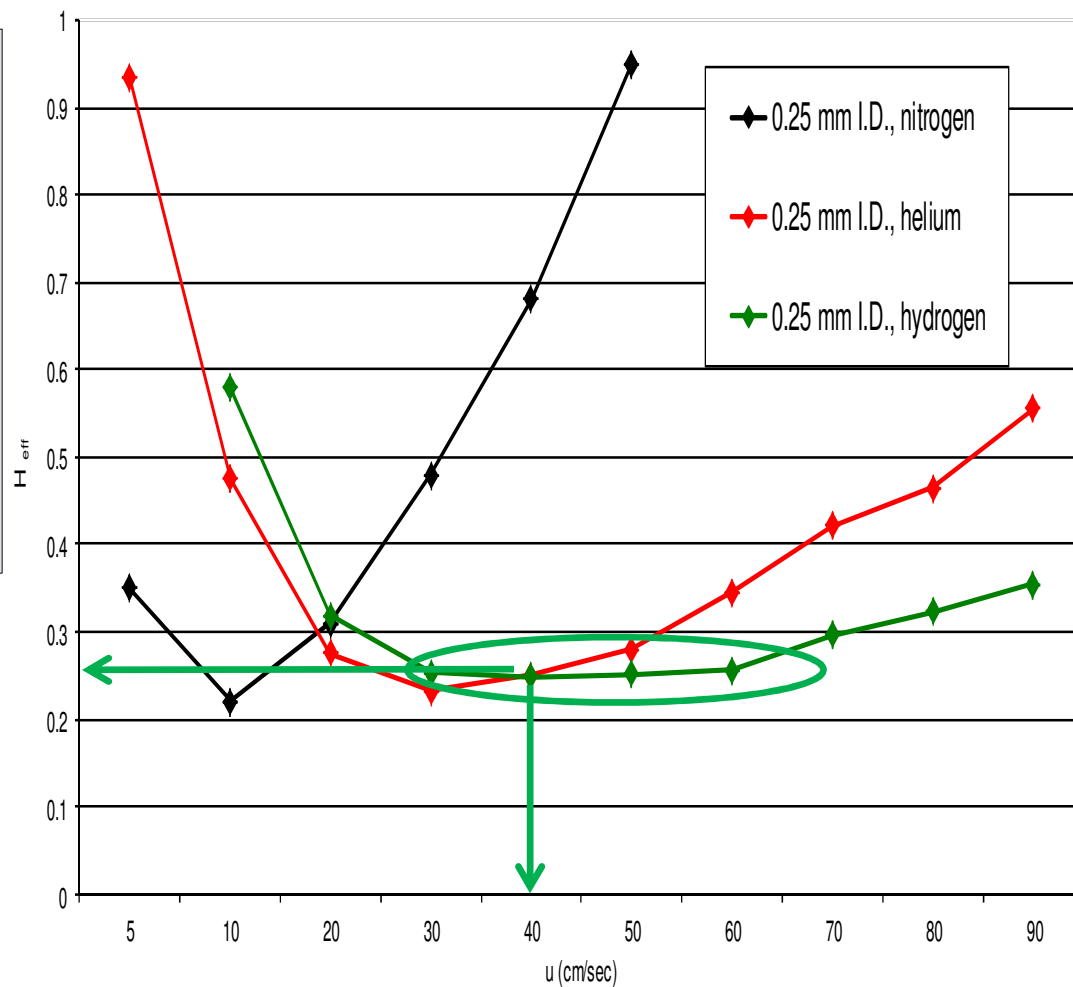
- **Golay plots** comparing carrier gas (0.25 mm I.D. column)
- Operate linear velocity ( $\mu$ )
  - Bottom of “U” if possible
  - Otherwise, on right side of “U”
- Choose
  - **Hydrogen** for most applications (temperature programmed or isothermal)
  - **Helium** for MS applications (most MS do not operate properly with hydrogen)
  - **Nitrogen** for some isothermal applications



# Carrier Gas

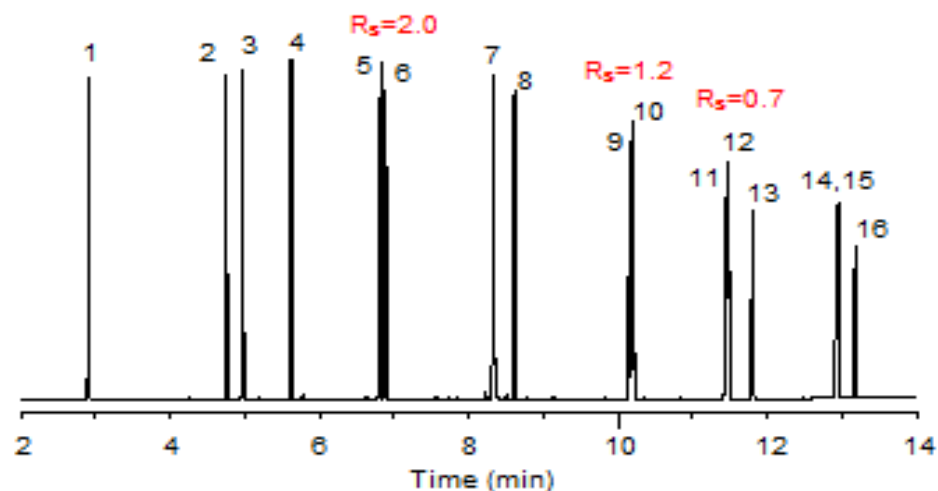
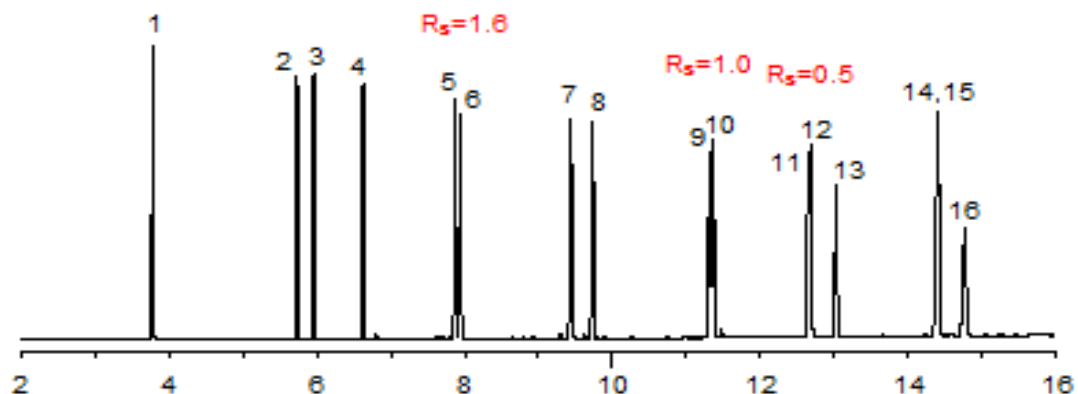
## Why Hydrogen?

- Almost as efficient as other choices
- Optimal  $\mu$  is higher
- Flattest curve (can run faster without sacrificing efficiency)
- Can generate on-site with a gas generator (save money)



# Carrier Gas

## Example: Helium vs Hydrogen



SLB-5ms

15 m x **0.25 mm**, 0.25  $\mu$ m  
70 °C (0.2 min), 20 °C/min  
to 325 °C (3 min)

**helium** at **25 cm/sec**

0.5  $\mu$ L, 10:1 split

16 PAHs

SLB-5ms

15 m x **0.25 mm**, 0.25  $\mu$ m  
70 °C (0.2 min), 20 °C/min  
to 325 °C (3 min)

**hydrogen** at **40 cm/sec**

0.5  $\mu$ L, 10:1 split

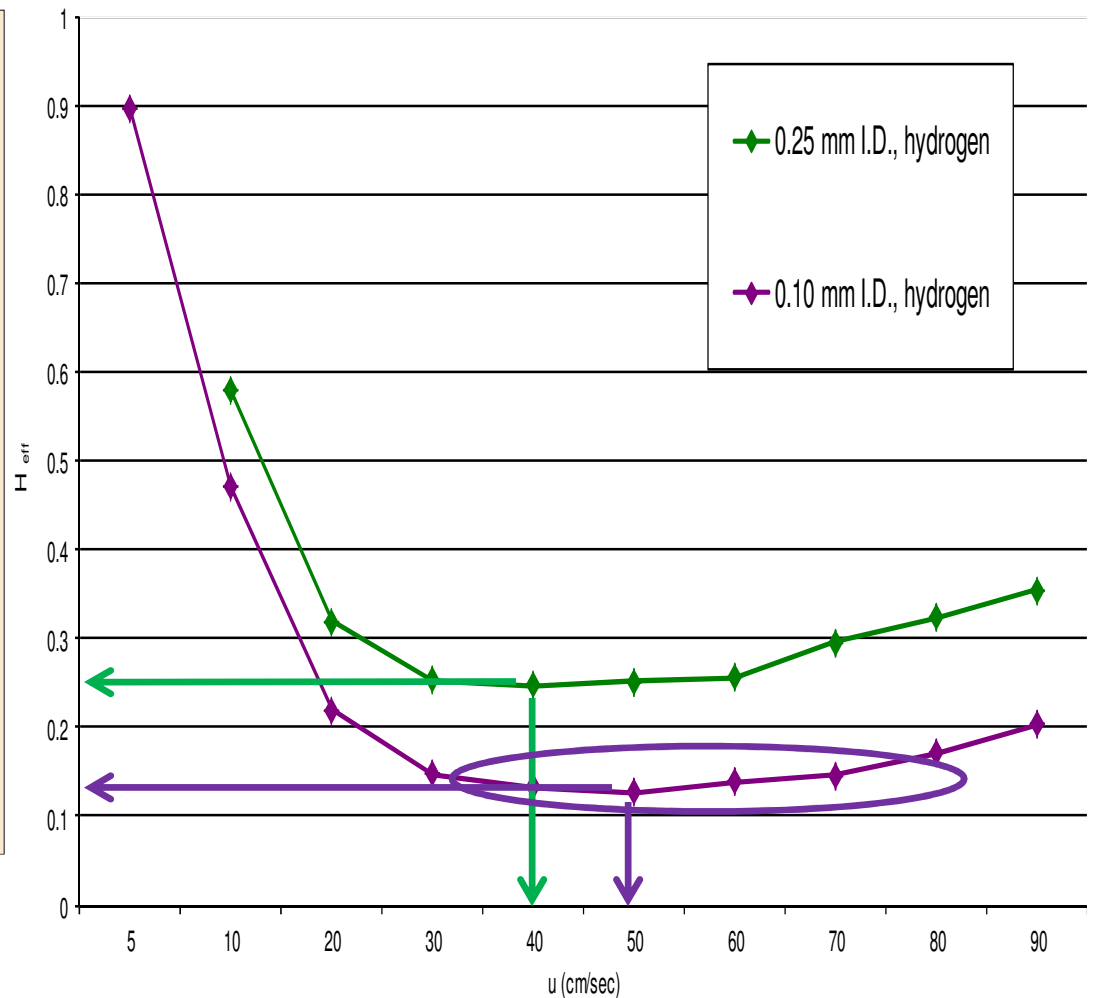
Both runs at optimal linear velocity ( $\mu$ ) for the combination of carrier gas and column I.D.

Fast analysis and better resolution with hydrogen.

# Carrier Gas

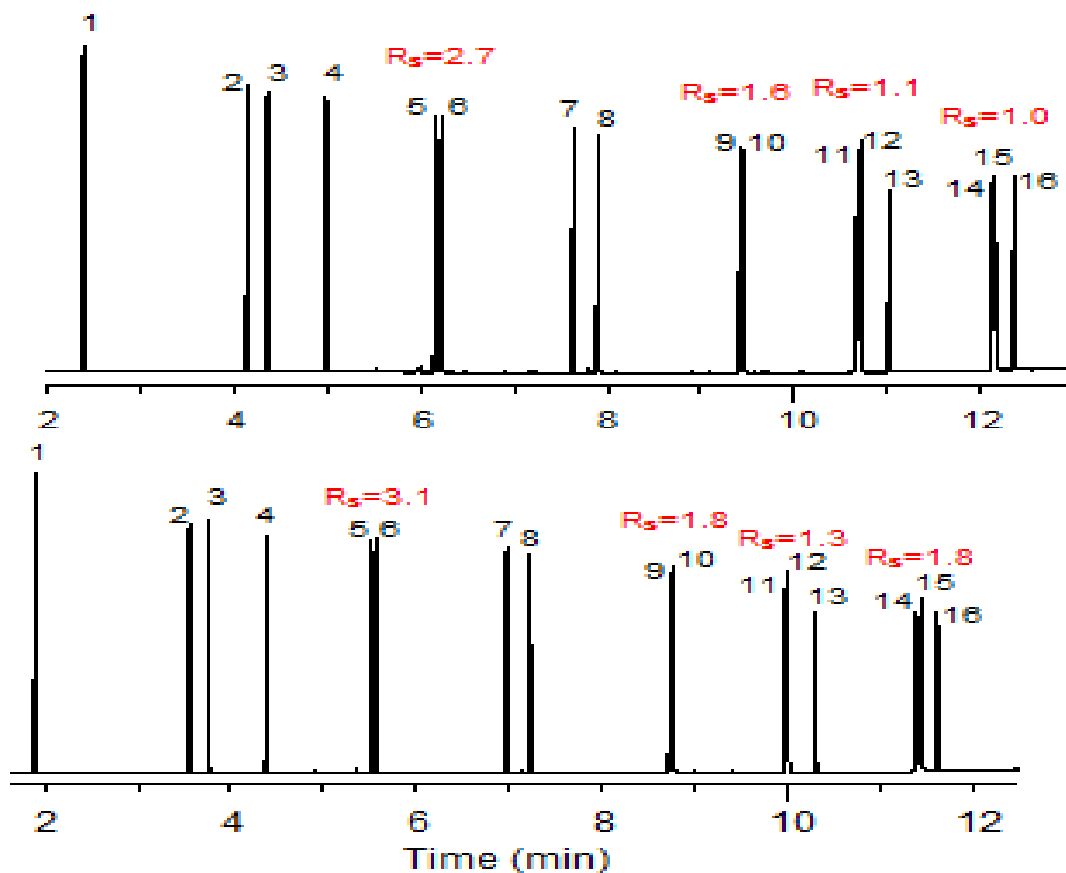
## Match Linear Velocity to Column I.D.

- **Golay plots** comparing column I.D.
- Optimal linear velocity (bottom of “U”) increases as column I.D. decreases
  - 40 cm/sec for 0.25 mm I.D.
  - 50 cm/sec for 0.10 mm I.D.
- The 0.10 mm I.D. column
  - **Better efficiency**
  - **Flatter curve** (can run faster without sacrificing efficiency)



# Carrier Gas

## Example: Below vs Above Optimal Linear Velocity



SLB-5ms

10 m x **0.10 mm**, 0.10  $\mu$ m  
70 °C (0.2 min), 20 °C/min  
to 325 °C (3 min)

**hydrogen** at **40 cm/sec**

0.5  $\mu$ L, 100:1 split

16 PAHs

SLB-5ms

10 m x **0.10 mm**, 0.10  $\mu$ m  
70 °C (0.2 min), 20 °C/min  
to 325 °C (3 min)

**hydrogen** at **60 cm/sec**

0.5  $\mu$ L, 100:1 split

40 cm/sec is optimal for 0.25 mm I.D., 50 cm/sec is optimal for 0.10 mm I.D. column.

Need to adjust linear velocity when column I.D. changes.

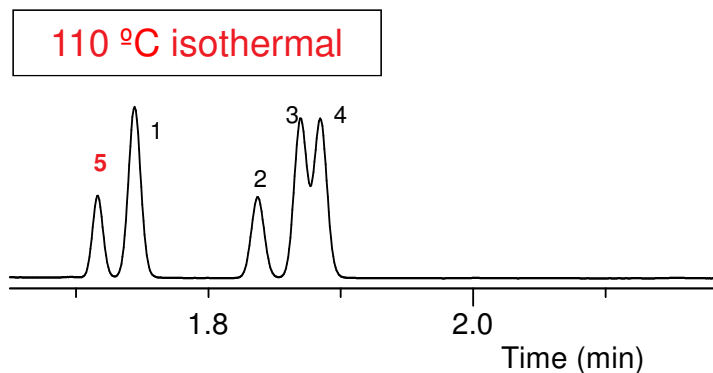
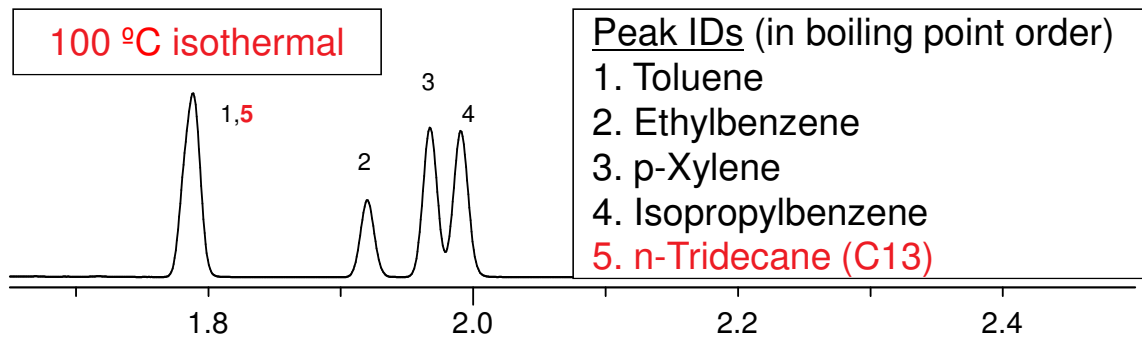
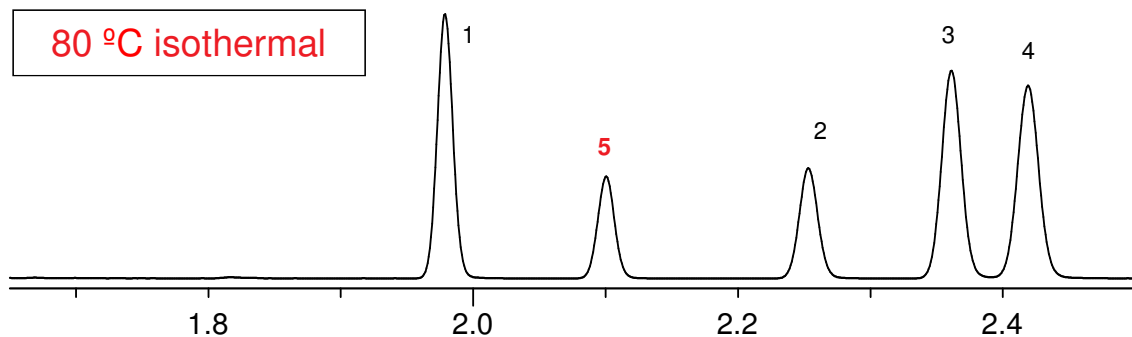
# Oven Temperature

## General Guidelines

- **Isothermal?**
  - Good option if analytes have similar chemical/physical properties
  - Eliminates oven cool-down time between runs
- **Initial temperature**
  - Common to use 10-20 °C below boiling point of injection solvent
  - Exception (water): 105 °C unless a guard column is used
- **Initial hold**
  - Common to use 30 sec to 2 min
  - Allows analytes to partition into stationary phase
- **Ramp rate**
  - Affects run time and possibly resolution
  - Resolution may be better if elution occurs on a ramp vs isothermal
  - Resolution may be better if elution occurs on a steeper ramp



# Oven Temperature can Effect Selectivity

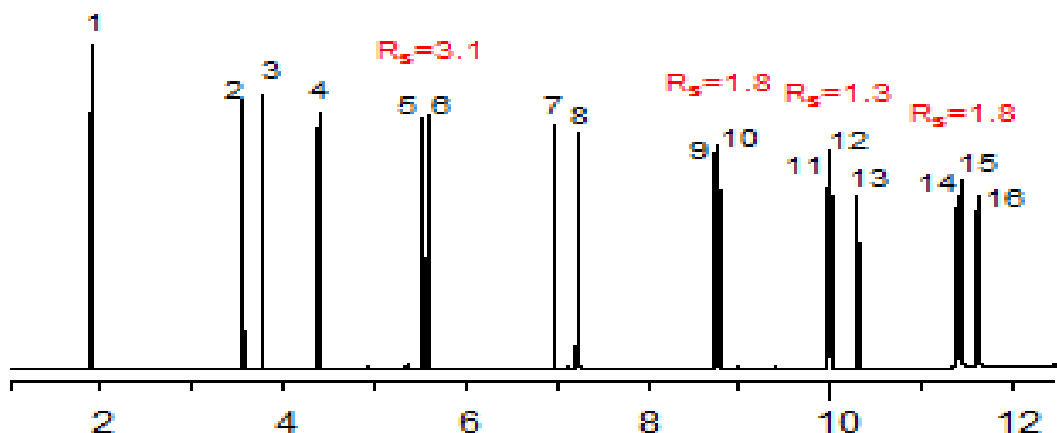


column: SLB-IL100, 30 m x 0.25 mm I.D., 0.20  $\mu$ m  
inj.: 250 °C  
det.: FID, 250 °C  
carrier gas: helium, 30 cm/sec  
injection: 1.0  $\mu$ L, 100:1 split  
liner: 4 mm I.D., split type, cup design  
sample: each analyte at various concentrations in isooctane

Higher oven temperature:

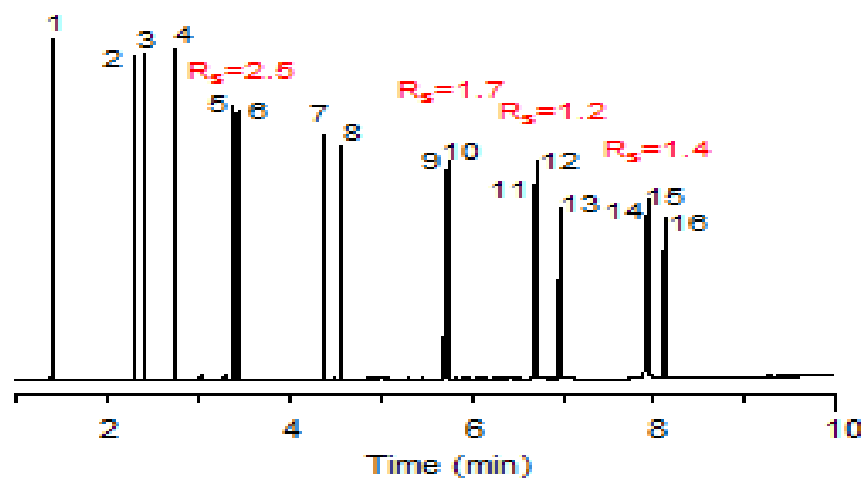
- **Decreased retention**; expected, a higher temperature will weaken all interactions
- **Selectivity changes**
  - n-Tridecane (peak 5) is primarily retained by dispersive interactions
  - The aromatics are retained by dipole-induced dipole interactions in addition to dispersive interactions

# Ramp Rate can be Linear or Stepped



SLB-5ms  
 10 m x 0.10 mm, 0.10  $\mu$ m  
**70 °C (0.2 min), 20 °C/min  
 to 325 °C (3 min)**  
 hydrogen at 60 cm/sec  
 0.5  $\mu$ L, 100:1 split

16 PAHs



SLB-5ms  
 10 m x 0.10 mm, 0.10  $\mu$ m  
**70 °C (0.2 min), 40 °C/min  
 to 175 °C, 25 °C/min to  
 270 °C, 20 °C/min to 325 °C**  
 hydrogen at 60 cm/sec  
 0.5  $\mu$ L, 100:1 split

Instrument can heat oven at different rates depending on temperature range.  
 Stepped ramp rate can decrease run time without a drastic loss of resolution.

# Chromatogram Index ([sigma-aldrich.com/gc-chros](http://sigma-aldrich.com/gc-chros))

## GC Applications

### Established Applications

### New Applications

The following chromatograms highlight some of the GC applications which may be performed with our columns, organized by **Industry**, by **Method**, by **Compound Class**, by **Phase Polarity**, by **Column Name**, and also by **Column Type**. Each entry typically includes the chromatogram, peak identifications/analyte names, and the complete analytical conditions.

- Industry: Environmental - (163)
- Industry: Industrial Hygiene - (13)
- Industry: Petroleum - (44)
- Industry: Biofuel - (15)
- Industry: Chemical - (182)
- Industry: Agriculture - (105)
- Industry: Food & Beverage - (220)
- Industry: Flavor & Fragrance - (101)
- Industry: Cosmetic and Personal Care/Cleaning Product - (17)
- Industry: Pharmaceutical - (40)
- Industry: Clinical - (66)
- Industry: Forensic - (34)
- Industry: Life Science - (6)

- Method: ASTM - (10)
- Method: EN - (5)
- Method: GPA - (2)
- Method: Japan MHLW - (7)
- Method: OSHA - (3)
- Method: Standard Methods - (2)
- Method: US EPA - (77)
- Method: US FDA - (5)
- Method: USP - (19)
- Compound Class - (586)
- Phase Polarity: Non-Polar - (247)
- Phase Polarity: Intermediate Polar - (52)
- Phase Polarity: Polar - (101)

- Phase Polarity: Highly Polar - (38)
- Phase Polarity: Extremely Polar - (23)
- Column Name - (622)
- Column Type: Ionic Liquid - (70)
- Column Type: MS-Grade - (129)
- Column Type: Fast GC - (80)
- Column Type: GCxGC - (2)
- Column Type: Chiral - (105)
- Column Type: PLOT - (23)
- Column Type: Packed - (13)
- GC Books - (3)

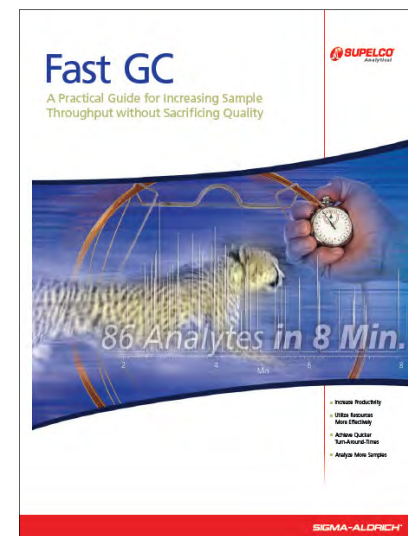
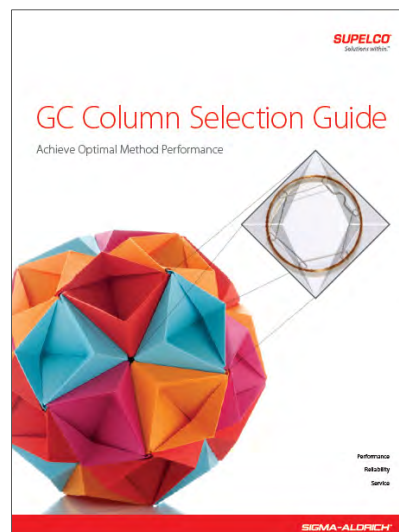
# Summary / Resources

## • Summary

- Choose stationary phase → then column I.D. → then film thickness → then length
  - If possible, start with an existing method or chromatogram
  - If not, remember “like dissolves like”
- Modify column parameters and instrument conditions to achieve desired effects
- Remember: derivatization can be used to modify analyte polarity

## • Resources

- GC Column Selection Guide (T407133 KCX)
- Fast GC Brochure (T407096 JTW)
- [sigma-aldrich.com/gc-columns](http://sigma-aldrich.com/gc-columns)
- Mike Buchanan ([mike.buchanan@sial.com](mailto:mike.buchanan@sial.com))



Supelco site  
Bellefonte, Pennsylvania (USA)

