

Practical Steps in GC Method Development

What to Consider

The Sample

Method of injection

Inlet

Detector

Carrier Gas

Column

COMPOUND REQUIREMENTS FOR GC

Only 10-20% of all compounds are suitable for GC analysis

The compounds must have:

- ✓ Sufficient volatility
- ✓ Thermal stability

NO Inorganic Acids and Bases

Be mindful of salts!

Sample Considerations

1. Sample matrix

residues?

dirty samples?

2. Analyte Composition

1. Isomers?
2. Polar vs. non-Polar?
3. Organic Acids?
4. Light Gases?
5. Nobel Gases?
6. Halogens?

Sample Residues

Semi-volatile residues

Bake out

Back flush

Non-volatile residues

Guard column

Bake out

Back flush

Dirty Samples

Sample clean up?

Back flush

Use What You Know About the Analytes

Complex Mixture?

Few analytes?

Homologous Series?

Mixture of polar and non-polar?

Labile analytes?

Volatility?

Gas or Liquid Sample?

Light Sensitive?



We have thought about the sample
...What's next?

Let's Get the Sample Onto the Column...

Manual Injection

Liquid Injection

Headspace

Purge & Trap

Gas Sampling Valve

SPME

Thermal Desorption

Custom

The Inlet

Volatiles Interface

Cool-On-Column

Purged Packed

PTV

Split / Splitless

Multi-Mode

Volatiles Interface

Used for 'volatile' samples

Sample is already a vapor

Headspace

Purge & Trap

Volatiles Interface

| Mode | Sample Concentration | Sample to Column | Comments |
|-----------|----------------------|-----------------------------|---|
| Split | High | Very little, most is vented | |
| Splitless | Low | All | Can switch to split mode electronically |
| Direct | Low | All | Must physically disconnect split vent, plug the interface, and reconfigure the GC. Maximizes sample recovery and eliminates possibility of contamination to pneumatic system. |

Cool-On-Column

- * Good for Labile Samples

 - Sample is deposited “ON” the column

 - Temperature of inlet follows Oven Temperature

- Good for ‘Active’ analytes

 - Minimizes inlet discrimination
 - No inlet Liner*

- Good for Trace Analysis

- Guard Column Highly Recommended

Purged Packed

Good for HIGH flow applications

Used with Packed columns

Can be used with 0.53 mm and 0.32 mm ID columns

**Has a minimal capacity for sample expansion

Back Flash

PTV

(Programmable Temperature Vaporization)

| Mode | Sample Concentration | Sample to Column | Comments |
|------------------|-----------------------------|-------------------------|--|
| Split | High | Very Little | |
| Pulsed Split | High | Very Little | |
| Splitless | Low | All | |
| Pulsed Splitless | Low | All | |
| Solvent Vent | Low | All | Multiple injections concentrate analytes and vent solvent. |

Split / Splitless

| Mode | Sample Concentration | Sample to Column | Comments |
|------------------|-----------------------------|-------------------------|---|
| Split | High | Very Little | |
| Pulsed Split | High | Very Little | Useful with large injections |
| Splitless | Low | All | |
| Pulsed Splitless | Low | All | Useful with large injections. *better transfer of sample to column* |

SPLIT INJECTOR

Split Ratio

- Too low: Poor peak shape
 - Column overload
- Too high: Poor sensitivity
 - Wastes carrier gas (gas saver)
- Usually non-linear
 - Do not use ratio as a dilution factor

MINIMUM RECOMMENDED SPLIT RATIO

| | mm I.D. | Lowest ratio |
|------------------------|-------------|--------------|
| Higher flow rates ↓ | 0.10 | 1:50 - 1:75 |
| | 0.18 - 0.25 | 1:10 - 1:20 |
| | 0.32 | 1:8 - 1:15 |
| | 0.53 | 1:2 - 1:5 |

Want to have 20 mL/min flow through the inlet

Multimode

| Mode | Sample Concentration | Sample to Column | Discussion |
|------------------|-----------------------------|-------------------------|---|
| Split | High | Low | |
| Pulsed Split | High | Low | |
| Splitless | Low | All | |
| Pulsed Splitless | Low | All | |
| Solvent Vent | Low | All | Multiple Injections concentrate sample and vent solvent |
| Direct | Low | All | |

Sample Expansion...Liners?

Split / Splitless Inlet

Multimode Inlet

Packed inlet

PTV

Inlet Liners - Purpose

Glass Inlet Liners provide an “inert” space for liquid samples to be uniformly vaporized to a gas and moved to the column.

Liquid-gas phase change involves a significant change in volume.

Gaseous sample volume depends on

- the solvent type
- column head pressure
- temperature of inlet

These aspects should be optimized for your sample volume and application.

| Solvent (1 μ L, ambient) | Volume (μ L at 250°C and 20psig) |
|---------------------------------|--|
| n-Hexane | 140 |
| Acetone | 245 |
| Acetonitrile | 350 |
| Methanol | 450 |
| Water | 1010 |

See “A Practical Guide to the Care, Maintenance, and Troubleshooting of Capillary GC Systems”, Third Revised Edition, by Dean Rood, Wiley-VCH, New York, 2001.

Liners - 3 Key Aspects Govern Applications

Liner Volume

Liner Treatments or Deactivation

Special Characteristics (glass wool, cup, taper, etc.)

When choosing a liner for your application, consider all three aspects to give you the best chromatography.

You must also determine what type of inlet is in your GC

Then consider the application itself, and the types of liners and injection techniques used for it:

 Split

 Splitless

Liner Volume

Choose a liner with enough volume to accommodate the vaporized sample.

Important, especially for polar solvents with large vapor volumes.

If vapor volume of sample exceeds liner volume, samples may back up (backflash) into carrier gas supply lines, causing ghost peaks and reproducibility problems in chromatography.

Liner Volume (contd.)

Agilent liners are primarily 2mm or 4mm in inner diameter (without tapers and additional features) and 78mm long.

- Thus, 2mm liners hold approx. 0.245 mL or 245 μ L of vapor
4mm liners hold approx. 0.972 mL or 972 μ L of vapor

Recommended injection volumes are 1-2 μ L or less for organic solvents, 0.5 μ L for water.

Liner Volume

How Do we Calculate the Vapor Volume?

Pressure / Flow Calculator

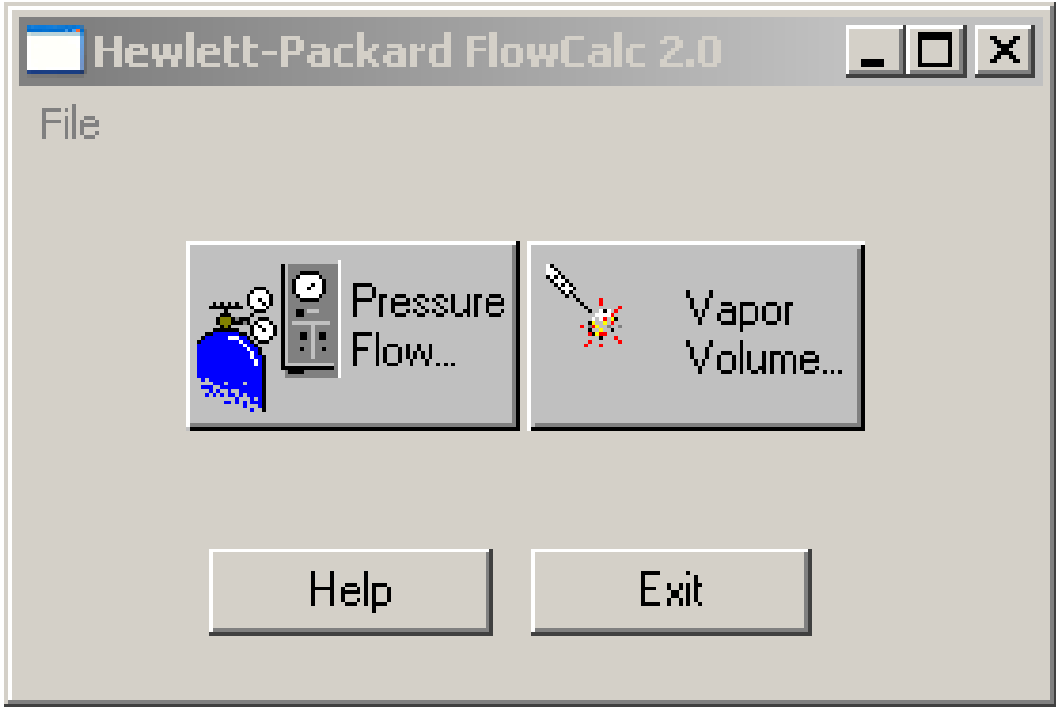
Free download from our Website

www.chem.agilent.com

<http://www.chem.agilent.com/en-US/Technical-Support/Instruments-Systems/Gas-Chromatography/utilities/Pages/GCCalculators.aspx>



Pressure / Flow Calculator



Determine what the inlet pressure will be:

Column Pressure/Flow Calculator

Column Parameters

Length (m)

i.d. (mm)

Temp (C)

Carrier Gas Parameters

Inlet Pressure (gauge)

Outlet Flow (mL/min)

Average Velocity (cm/s)

Outlet Pressure (Absolute)

1 Atm Vacuum Other

Split Ratio

Split vent flow

Split Ratio(vent flow/col flow) :1

Holdup time

1.67 minutes

Inlet

Inlet Temperature (C)

Inlet Flow (mL/min)

Carrier gas

Helium Opt. Vel. range 20 40

Pressure Units KPa psi bar

Agilent Instrument Utilities


The screenshot displays the Agilent Instrument Utilities software interface. The window title is "Agilent Instrument Utilities". The top right corner shows the current user as "AGILENT\simjones [Administrator]" and the current instrument as an empty dropdown menu. The main title bar reads "Agilent Instrument Utilities Version B.1.06.11343.1852".


The interface is divided into several sections:

- Left Sidebar:** Contains navigation options: "Lab at a Glance", "Configuration", "Documentation", "Firmware Update", and "Calculators". A "Help" section is also present with links to "The Vapor Volume Calculator", "Comparison with liner volume", and "Using the Vapor Volume Calculator".
- Calculators Tab:** The "Vapor Volume Calculator" is selected, with other tabs for "Pressure Flow Calculator", "Method Translator", and "Solvent Vent Calculator".
- Solvent Properties:** A dropdown menu shows "Methanol". Below it, the boiling point is 64.7°C, density is 0.791 g/cm³, and molecular weight is 32 amu.
- Injection Liner:** A dropdown menu shows "5190-2295" with a liner volume of 850 µL.
- Injection Parameters:** Includes sliders and input fields for "Injection Volume (µL)" (1.00), "Inlet Temperature (°C)" (250), and "Inlet Pressure (gauge)" (8.600). Units are set to psi.
- Results:** Shows an "Estimated Volume" of 669 µL and a "% Capacity" of 78%. A yellow progress bar indicates the capacity level.
- Management:** "Solvents" and "Liners" sections each have "Add", "Remove", and "Defaults" buttons.

Test Inlet Conditions For Solvent Expansion

Solvent Vapor Volume Calculator

Approximate vapor volume(ul): **669 ul**  **79 %**




Injection Volume (ul)
Slider:

Inlet Temp (C)
Slider:

Inlet Pressure
Slider:

Pressure Units
 KPa psi bar

Solvent Properties
Methanol
Boiling Pt (C): 64.7
Denisty (g/cm3): 0.791
Mol Wt. (amu): 32
 Solvents


Injection Liner Volume (ul)
5183-4647 single-t 850

Capacity limits (%)
75 100

Water as Solvent

Solvent Vapor Volume Calculator [X]

Approximate vapor volume(ul): **1499 ul**

Overload:  **176%**

Injection Volume (ul): 1.0

Inlet Temp (C): 250


Inlet Pressure: 8.6

Pressure Units: KPa psi bar

Solvent Properties

Water

Boiling Pt (C): 100
Denisty (g/cm3): 0.998
Mol Wt. (amu): 18.02

 Solvents

Injection Liner Volume (ul): 5183-4647 single-t 850

Capacity limits (%): 75 100

Print Help OK Edit Liner list

Water as Solvent

Cut Injection Volume in Half

The screenshot shows the 'Solvent Vapor Volume Calculator' window. At the top, it displays 'Approximate vapor volume (ul): 750 ul' and '88 %'. Below this is a progress bar. The main interface is divided into several sections: 'Injection Volume (ul)' with a slider set to 0.5; 'Inlet Temp (C)' with a slider set to 250; 'Inlet Pressure' with a slider set to 8.6; 'Solvent Properties' with 'Water' selected, showing Boiling Pt (C): 100, Density (g/cm3): 0.998, and Mol Wt. (amu): 18.02; 'Pressure Units' with 'psi' selected; and 'Injection Liner' with '5183-4647 single-t' selected and a volume of 850 ul. A 'Capacity limits (%)' table shows 75 and 100. Buttons for 'Print', 'Help', 'OK', and 'Edit Liner list' are at the bottom.

Solvent Vapor Volume Calculator

Approximate vapor volume (ul): **750 ul** **88 %**

Injection Volume (ul): **0.5**

Inlet Temp (C): **250**

Inlet Pressure: **8.6**

Pressure Units: KPa psi bar

Solvent Properties:

- Solvent: **Water**
- Boiling Pt (C): 100
- Density (g/cm3): 0.998
- Mol Wt. (amu): 18.02

Solvents

Injection Liner: **5183-4647 single-t** Volume (ul): **850**

Capacity limits (%): 75 100

Buttons: Print, Help, OK, Edit Liner list

Water as Solvent

Pulsed Injection

The screenshot shows the 'Solvent Vapor Volume Calculator' window. At the top, it displays 'Approximate vapor volume(ul): 750 ul' and '88 %'. Below this is a progress bar. The main interface is divided into several sections: 'Injection Volume (ul)' with a slider set to 1.0; 'Inlet Temp (C)' with a slider set to 250; 'Inlet Pressure' with a slider set to 31.9; 'Solvent Properties' with a dropdown menu set to 'Water' and values for Boiling Pt (100), Density (0.998), and Mol Wt. (18.02); 'Pressure Units' with radio buttons for KPa, psi (selected), and bar; and 'Injection Liner' with a dropdown set to '5183-4647 single-t' and a volume of 850. At the bottom right, there are 'Capacity limits (%)' of 75 and 100, and an 'Edit Liner list' button. The window has 'Print', 'Help', and 'OK' buttons at the bottom left.

Solvent Vapor Volume Calculator

Approximate vapor volume(ul): **750 ul** **88 %**

Injection Volume (ul): **1.0**

Inlet Temp (C): **250**

Inlet Pressure: **31.9**

Pressure Units: KPa psi bar

Solvent Properties: **Water**
Boiling Pt (C): 100
Denisty (g/cm3): 0.998
Mol Wt. (amu): 18.02

Solvents

Injection Liner: **5183-4647 single-t** Volume (ul): **850**

Capacity limits (%): 75 100

Print Help OK

Liner Treatments or Deactivation

Minimizes possibility of active sample components from adsorbing on active sites on the liner or glass wool surface.

Unwanted sample adsorption leads to tailing peaks and loss of response for polar compounds.

Although not necessary for all applications, deactivated liners provide added insurance against possible sample adsorption.

Deactivation of borosilicate glass liners is often done with a silylating reagent like Dimethyldichlorosilane (DMDCS)

Special Characteristics

Some liners have special features that are necessary for different injection techniques. For example:

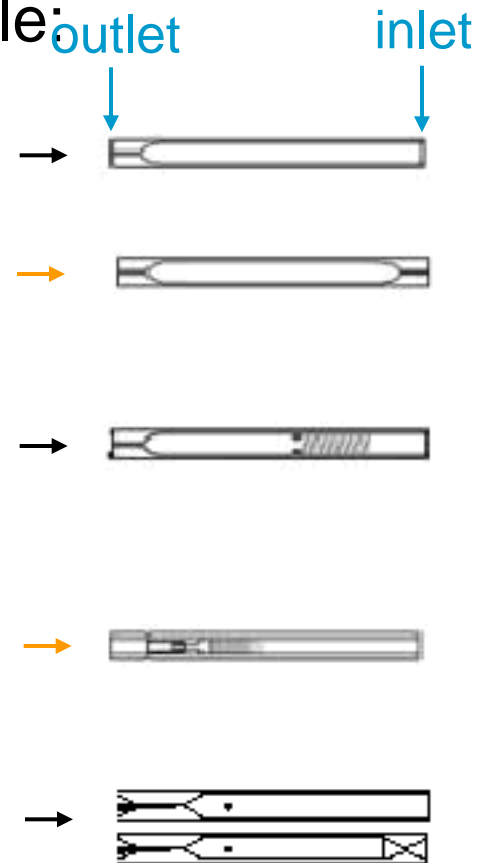
Taper (gooseneck), minimizes sample contact with gold seal.

Dual taper, also minimizes sample contact with inlet weldment and reduces potential for backflash.

Glass wool and shelf to hold it in place, prevents non-volatiles from reaching column and removes residual sample from needle. Glass wool should be deactivated.

Jennings cup, normally used for efficient sample mixing in split inlets, reduces sample discrimination and prevents non-volatiles from reaching the column. Not for very dirty samples.

Press fit (direct) connection end to hold capillary column firmly (virtually all sample goes onto the column). Side hole needed for Electronic Pressure Control with direct connect liners.



Special Characteristics (contd.)





Other special characteristics include:

- Baffles
- Spiral paths
- Glass or ceramic frits or beads
- Laminar cups (elongated version of Jennings cups)
- Column packings with stationary phases





All designed to provide:

- a turbulent sample flow path for sample mixing
- protrusions, barriers, or adsorbents to collect high molecular weight sample components or particles
- surfaces for efficient vaporization of sample components.

Split Injection Liners

| Liner | Part No. | Comments |
|--|--------------------|---|
|  | <p>19251-60540</p> | <p>Simplest split liner, glass wool, no-deactivation, large volume, 990μL volume. Use for general purpose applications for compounds with low glass adsorption activity. Also used for Splitless mode.</p> |
|  <p>Glass nub</p> | <p>5183-4647</p> | <p>Glass wool (held near needle entrance to remove residual sample on needle), deactivated, 870μL volume. Glass nub ensures that gap remains below liner for split injection. Efficient, for most applications, including active compounds. Fail-safe insertion into injection port. Needle length is important.</p> |
|  | <p>18740-80190</p> | <p>Liner with Jennings cup, no glass wool, 800μL volume. For manual injection only. Use for general purpose applications, high and low MW compounds. Reduces inlet discrimination.</p> |
|  | <p>18740-60840</p> | <p>Liner with Jennings cup, glass wool, and column packing, 800μL volume. For manual injection only. For dirty samples, traps non-volatiles and particulates well. For high and low MW compounds. Not recommended for use with EPC.</p> |

Splitless Injection Liners

| Liner | Part No. | Comments |
|--|----------------------------|---|
|  | 5181-3316 | Single taper, deactivated, 900µL volume. Taper isolates sample from metal seal, reducing breakdown of compounds that are active with metals. For trace samples, general application. |
|  | 5062-3587 | Single taper, deactivated, with glass wool, 900µL volume. Glass wool aides volatilization and protects column. For trace (dirty) samples. |
|  | 5181-3315 | Double taper, deactivated, 800µL volume. Taper on inlet reduces chance for backflash into carrier gas lines. High efficiency liner for trace, active samples. |
|  <p style="color: red; margin-left: 100px;">Side hole</p> | G1544-80730 G1544-80700 | Direct connect liners, single and dual taper, deactivated. Capillary column press fits into liner end, eliminating sample exposure to inlet. Ultimate protection for trace, active samples. Side hole permits use with EPC. |

GLASS WOOL

Liner Packing Recommendations

Amount, size and placement must be consistent for consistent results

Can be broken upon installation into the liner, exposing active sites

Liner deactivation with glass wool plug in place is ideal

GLASS WOOL

Placement in Liner

Near top of liner:

- Wipes syringe needle of sample
- Can improve injector precision
- Helps to prevent backflash

Near bottom of liner:









- Helps in volatilization of high MW components
- Increases mixing

Both positions help retain some non-volatile residues from reaching the column

Ultra Inert Liners

Liner is deactivated with glass wool in place!

Agilent Ultra Inert Liners

| Description | Volume (µL) | ID (mm) | 1/pk | 5/pk | 25/pk | 100/pk* |
|--|-------------|---------|-----------|-----------|-----------|-----------|
| Split Inlet Liners | | | | | | |
|  Low pressure drop, Ultra Inert Liner with glass wool | 870 | 4 | 5190-2295 | 5190-3165 | 5190-3169 | 5190-3173 |
|  Straight, Ultra Inert Liner with glass wool | 990 | 4 | 5190-2294 | 5190-3164 | 5190-3168 | 5190-3172 |
| Splitless Inlet Liners | | | | | | |
|  Single taper, Ultra Inert Liner | 900 | 4 | 5190-2292 | 5190-3162 | 5190-3166 | 5190-3170 |
|  Single taper, Ultra Inert Liner with glass wool | 900 | 4 | 5190-2293 | 5190-3163 | 5190-3167 | 5190-3171 |
|  Splitless, double taper Ultra Inert Liner, no wool | 800 | 4 | 5190-3983 | 5190-4007 | | |
|  Dimpled, splitless, Ultra Inert Liner | 200 | 2 | 5190-2297 | 5190-4006 | | |
|  Straight, Ultra Inert Liner | | 1 | 5190-4047 | | | |
|  Straight Ultra Inert Liner for SPME | | .75 | 5190-4048 | | | |

*The 100/pk is not in the Touchless packaging. O-rings must be purchased separately.

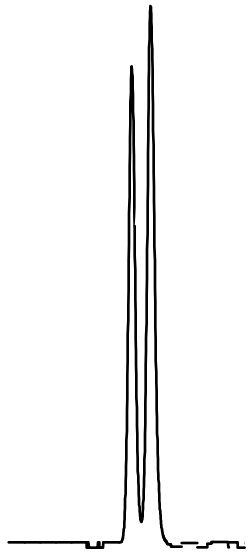
Carrier Gas Considerations

- Carries the solutes down the column
- Selection and velocity influences efficiency and retention time

RESOLUTION VS. LINEAR VELOCITY

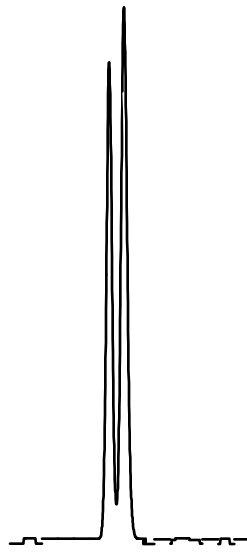
Helium

Resolution of 1.5 = baseline resolution



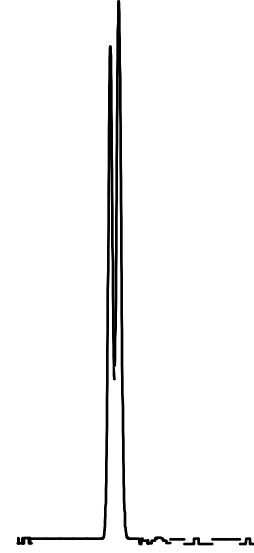
$R = 1.46$
30 cm/sec

4.4 psig



$R = 1.31$
35 cm/sec

5.1 psig

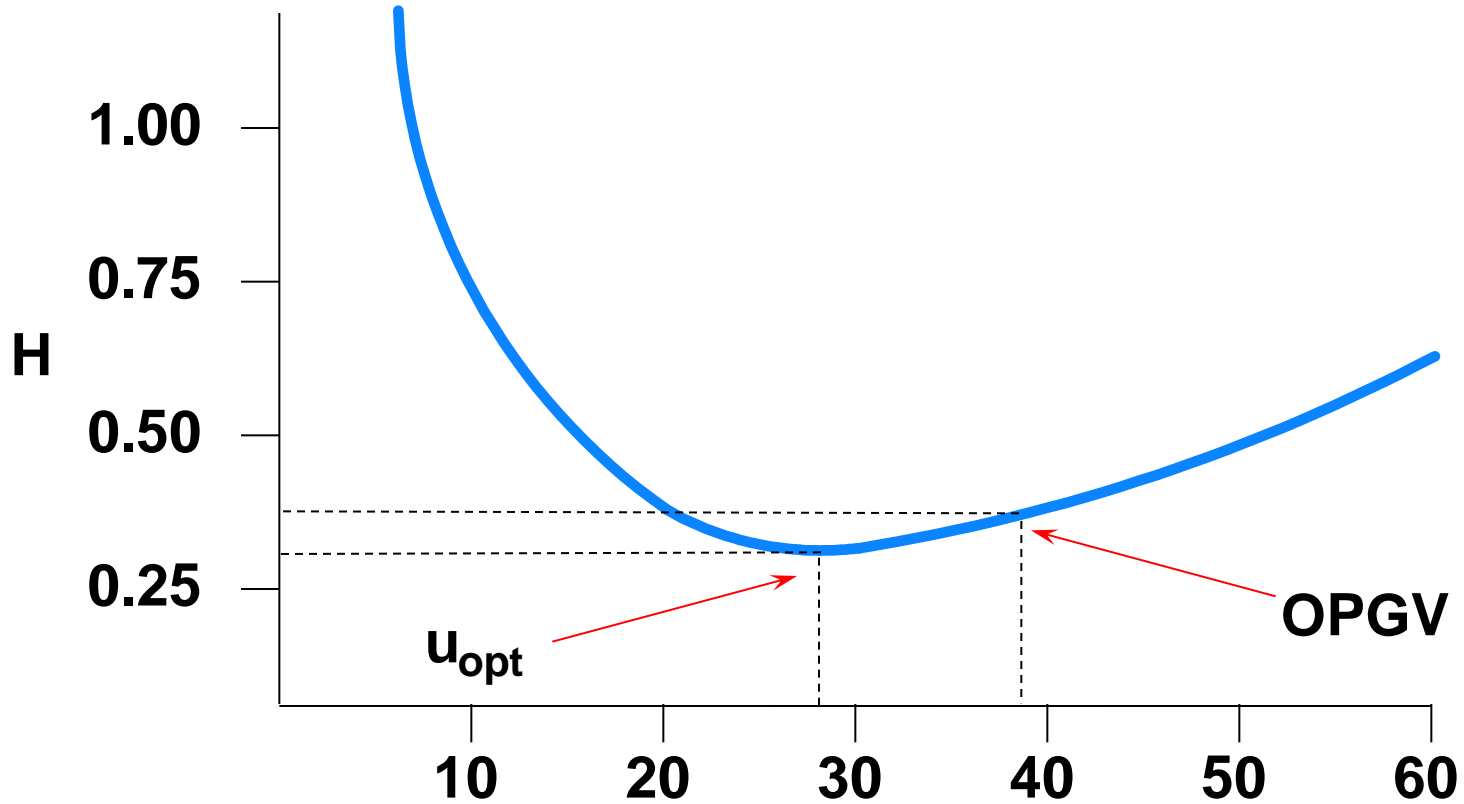


$R = 0.97$
40 cm/sec

5.8 psig

DB-1, 15 m x 0.32 mm ID, 0.25 μ m
60°C isothermal
1,3- and 1,4-Dichlorobenzene

VAN DEEMTER CURVE



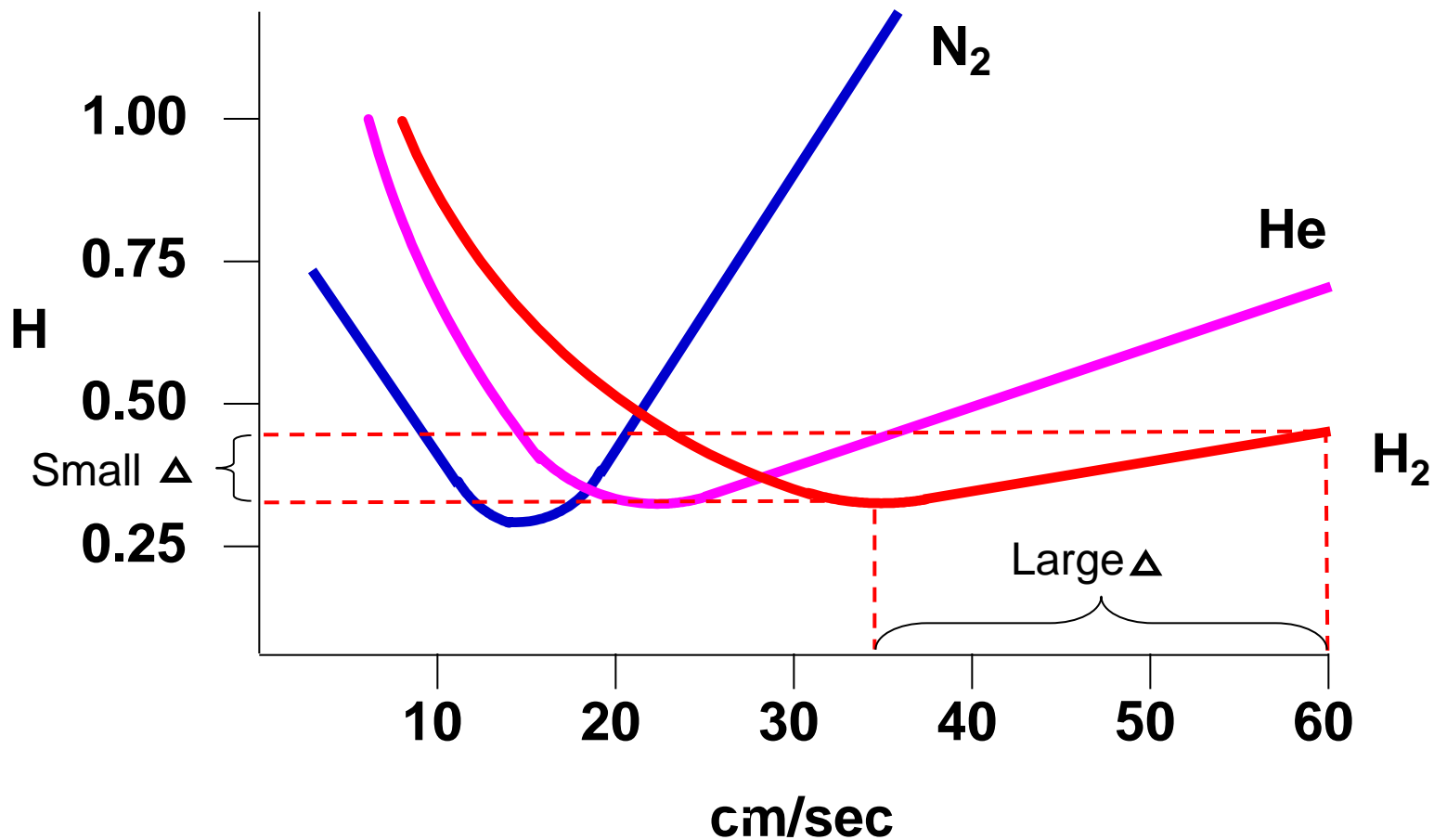
\bar{u}_{opt} and OPGV

\bar{u}_{opt} : Maximum efficiency

OPGV: Optimal practical gas velocity
Maximum efficiency per unit time

$$1.5 - 2x \bar{u}_{opt}$$

VAN DEEMTER CURVES



What Happens to the Flow as Oven Temp Increases?

Column Pressure/Flow Calculator

Column Parameters

Length (m)

i.d. (mm)

Temp (C)

Carrier Gas Parameters

Inlet Pressure (gauge)

Outlet Flow (mL/min)

Average Velocity (cm/s)

Outlet Pressure (Absolute)

1 Atm Vacuum Other

Split Ratio

Split vent flow

Split Ratio(vent flow/col flow) :1

Holdup time

1.67 minutes

Inlet

Inlet Temperature (C)

Inlet Flow (mL/min) 1.81

Carrier gas

Helium Opt. Vel. range 20 40

Pressure Units

KPa psi bar

Help Plot... Print OK

Carrier Gas: Constant Pressure

Column Pressure/Flow Calculator

Column Parameters

Length (m)

i.d. (mm)

Temp (C)

Carrier Gas Parameters

Inlet Pressure (gauge)

Outlet Flow (mL/min)

Average Velocity (cm/s)

Outlet Pressure (Absolute)

1 Atm Vacuum Other

Split Ratio

Split vent flow

Split Ratio(vent flow/col flow) :1

Holdup time

2.62 minutes

Inlet

Inlet Temperature (C)

Inlet Flow (mL/min)

Carrier gas

Opt. Vel. range

Pressure Units

KPa psi bar

Carrier Gas: Constant Flow

Column Pressure/Flow Calculator

Column Parameters

Length (m)

i.d. (mm)

Temp (C)

Carrier Gas Parameters

Inlet Pressure (gauge)

Outlet Flow (mL/min)

Average Velocity (cm/s)

Outlet Pressure (Absolute)

1 Atm Vacuum Other

Split Ratio

Split vent flow

Split Ratio(vent flow/col flow) :1

Holdup time

Inlet

Inlet Temperature (C)

Inlet Flow (mL/min) 1.24

Carrier gas

Opt. Vel. range 20 40

Pressure Units

KPa psi bar

Detectors

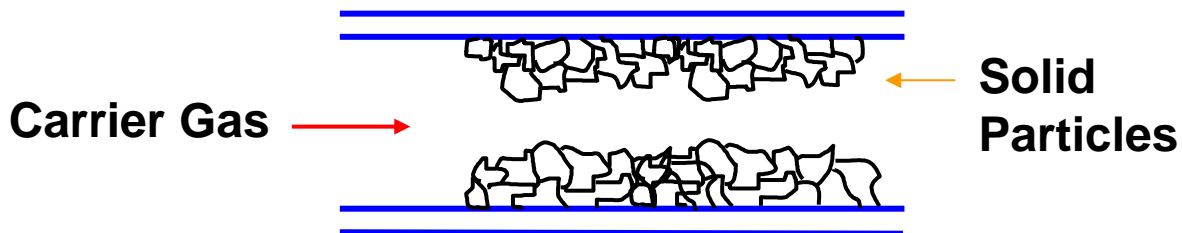
| Detector | Dynamic Range | | MDL |
|----------|--------------------|----------------------------|------------------------|
| TCD | 10^5 | Universal | 400 pg Tridecane |
| FID | 10^7 | Responds to C-H bonds | 1.8 pg Tridecane |
| ECD | 5×10^5 | Responds to free electrons | 6 fg/mL Lindane |
| NPD | 10^5 | Specific to N or P | 0.4 pgN/s 0.06 pg P /s |
| FPD | 10^3 S, 10^4 P | Specific to S or P | 60 fg P/s 3.6 pg S/s |
| SCD | 10^4 | Specific & Selective to S | 0.5 pg S/s |
| NCD | 10^4 | Specific & Selective to N | 3 pg N/s |
| MSD | | Universal | S/N 400:1 1 pg/uL OFN |

Selecting the RIGHT Column

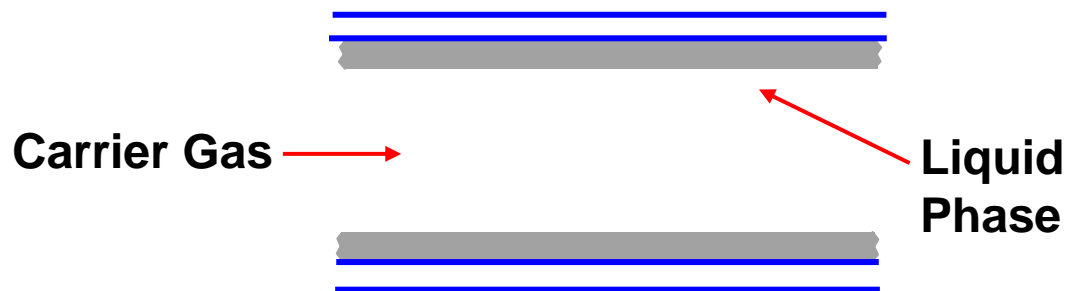
Understanding the Stationary Phase

CAPILLARY COLUMN TYPES

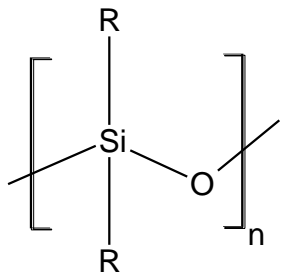
Porous Layer Open Tube (PLOT)



Wall Coated Open Tube (WCOT)

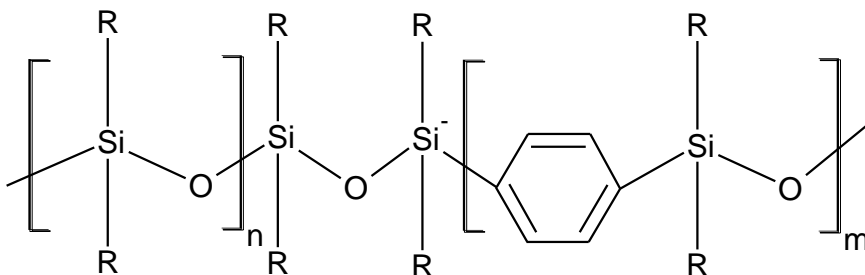


STATIONARY PHASE POLYMERS

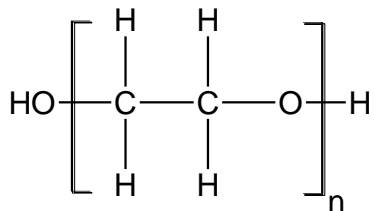


**R= methyl, cyanopropyl, cyanopropylphenyl,
trifluoropropyl**

Siloxane



Arylene



Polyethylene glycol backbone

Selectivity Interactions

- Dispersion
- Dipole
- Hydrogen bonding

Selectivity

Interaction Strengths

| Phase | Dispersion | Dipole | H Bonding |
|-----------------|------------|----------|-----------|
| Methyl | Strong | None | None |
| Phenyl | Strong | None | Weak |
| Cyanopropyl | Strong | Strong | Moderate |
| Trifluoropropyl | Strong | Moderate | Weak |
| PEG | Strong | Strong | Moderate |

Starting Parameters

Inlet

Start with a Split/Splitless inlet in split mode ~50:1

Inlet temperature hot enough to vaporize the sample
~250°C to start

Oven

Start cold to trap the sample on the head of the column
ramp conservatively if you are not certain of where analytes will elute
to the isothermal limit of the column*

Detector

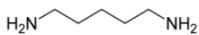
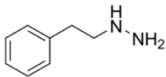
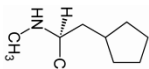
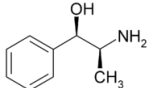
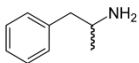
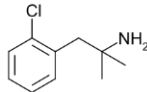
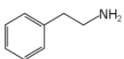
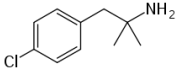
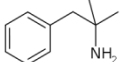
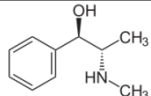
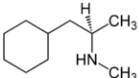

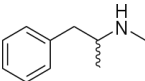
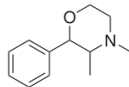



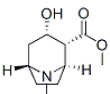
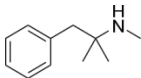

Set the detector temperature ~20°C above the highest oven temperature

Carrier Gas

Set the velocity to the midpoint of the velocity range for the carrier gas type

Now Let's Apply What We've Learned

Sample List (drugs)

| | | | |
|--------------------|---|---------------------------|---|
| 1. Cadaverine |  | 11. Phenelzine |  |
| 2. Cyclopentamine |  | 12. Phenylpropanolamine |  |
| 3. Amphetamine |  | 13. Clortermine |  |
| 4. Phenethylamine |  | 14. Chlorphentermine |  |
| 5. Pentermine |  | 15. Ephedrine |  |
| 6. Propylhexedrine |  | 16. Pseudoephedrine |  |
| 7. Methamphetamine |  | 17. Phendimetrazine |  |
| 8. Methenamine |  | 18. MDA |  |
| 9. Amantidine |  | 19. Ecgonine methyl ester |  |
| 10. Mephentermine |  | 20. diethylpropion |  |

Starting Method Parameters

Column: DB-5 30m X 0.32mm X 0.25um

S/SI Inlet: Split 50:1 Temp 250°

FID: Temp 350°

Carrier: He

Constant flow 30 cm/sec

Oven: 50°C Hold for 5 min

10°C/min to 325°C Hold for 5 min

Am I Going to Have Backflash?

Column Pressure/Flow Calculator

Column Parameters

Length (m)

i.d. (mm)

Temp (C)

Carrier Gas Parameters

Inlet Pressure (gauge)

Outlet Flow (mL/min)

Average Velocity (cm/s)

Outlet Pressure (Absolute)

1 Atm Vacuum Other

Split Ratio

Split vent flow

Split Ratio(vent flow/col flow) :1

Holdup time

1.67 minutes

Inlet

Inlet Temperature (C)

Inlet Flow (mL/min)

Carrier gas


Opt. Vel. range


Pressure Units

KPa psi bar

Injection Volume / Solvent Expansion

Solvent Vapor Volume Calculator [X]

Approximate vapor volume(ul): **669 ul**  **79 %**




Injection Volume (ul)
Slider: [] [1.0] [◀ ▶]

Inlet Temp (C)
Slider: [] [250] [◀ ▶]

Inlet Pressure
Slider: [] [8.6] [◀ ▶]

Pressure Units
 KPa psi bar

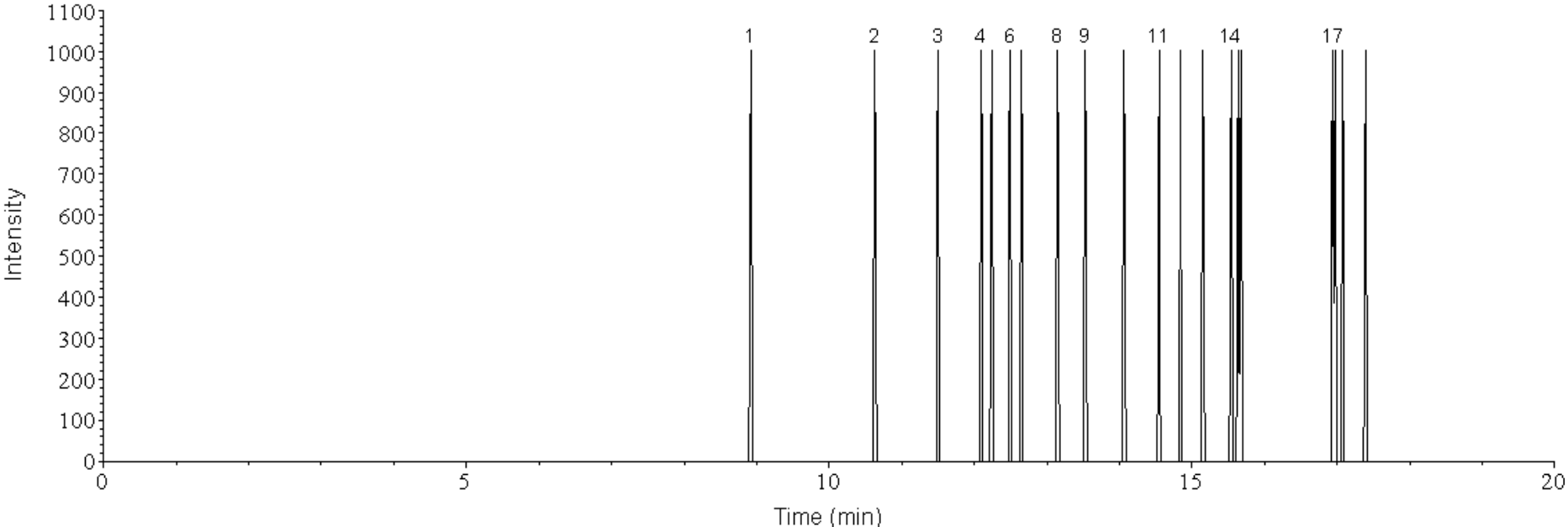
Solvent Properties
Methanol [▼]
Boiling Pt (C): 64.7
Denisty (g/cm3): 0.791
Mol Wt. (amu): 32


Injection Liner Volume (ul)
5183-4647 single-t [▼] 850
Capacity limits (%)
75 100

Developing Temperature Program

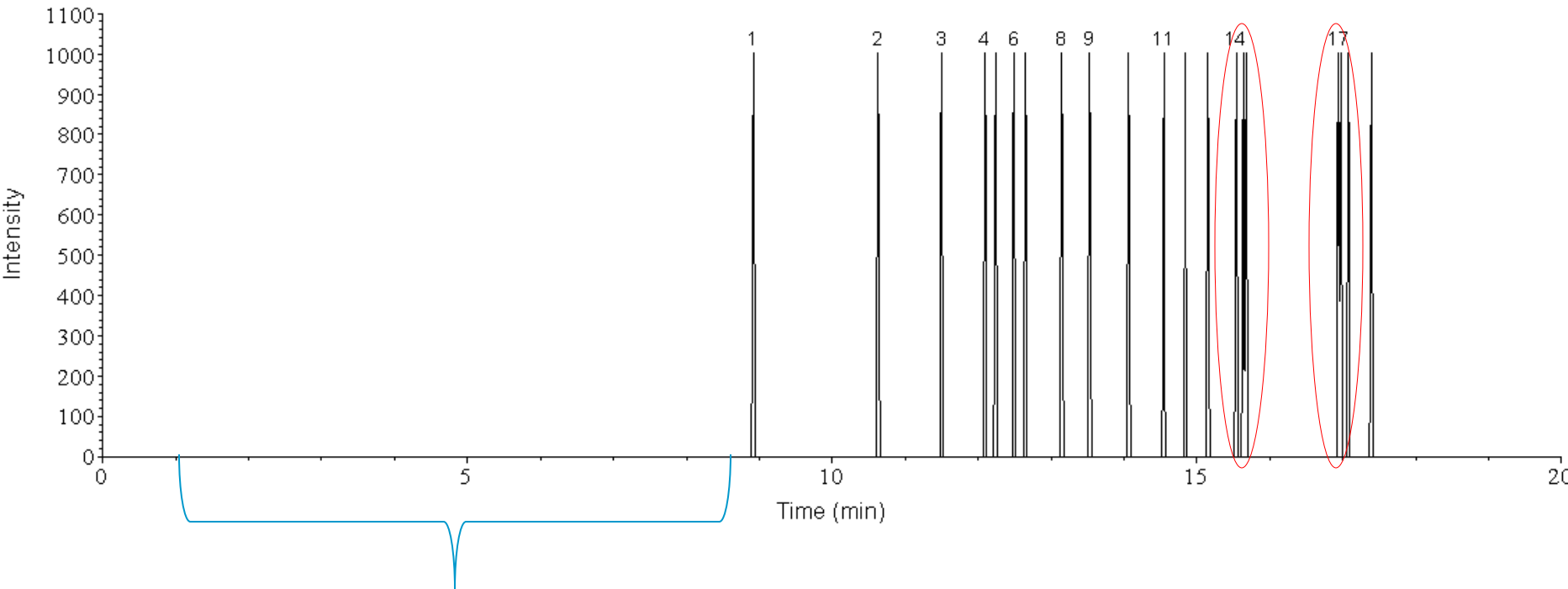
Initial Run

Initial Temp 50°C Hold for 5 min
Ramp 10°C/min to 325°C Hold for 5 min



Developing Temperature Program

Initial Run - Define Areas for Improvement



Next Step...

When does the first peak come out?

~9 minutes

What temperature does it come out at?

Temp program:

50°C for 5 minutes

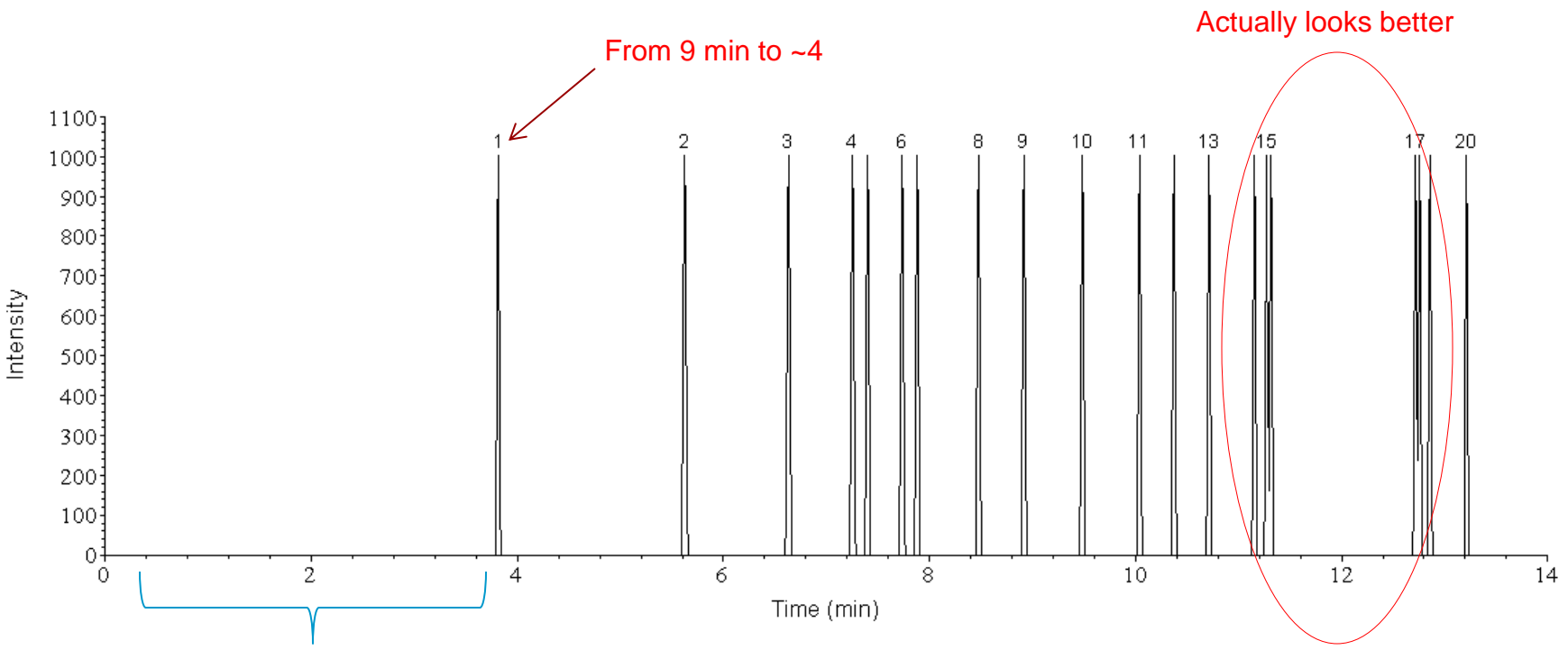
10°C to 325°C

1st Peak comes out at 90°C

Developing Temperature Program

2nd Try

Initial Temp 90°C Hold for 5 min
Ramp 10°C/min to 325°C Hold for 5 min

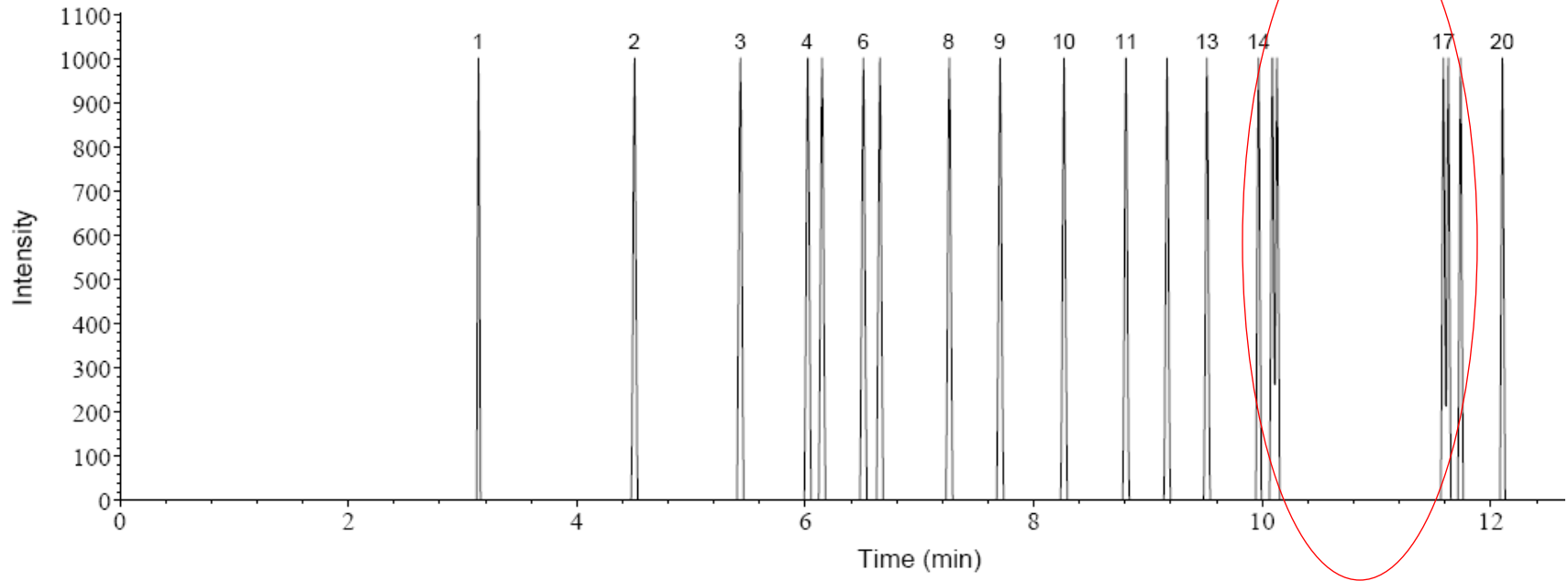


Developing Temperature Program

3rd Try

Initial Temp 100°C Hold for 5 min
Ramp 10°C/min to 325°C Hold for 5 min

Time to resolve these peaks



Resolve Co-elutions

Add a hold 20-30° below the elution temperature

Co-elutions occur at 10 minutes

100°C hold for 5 minutes

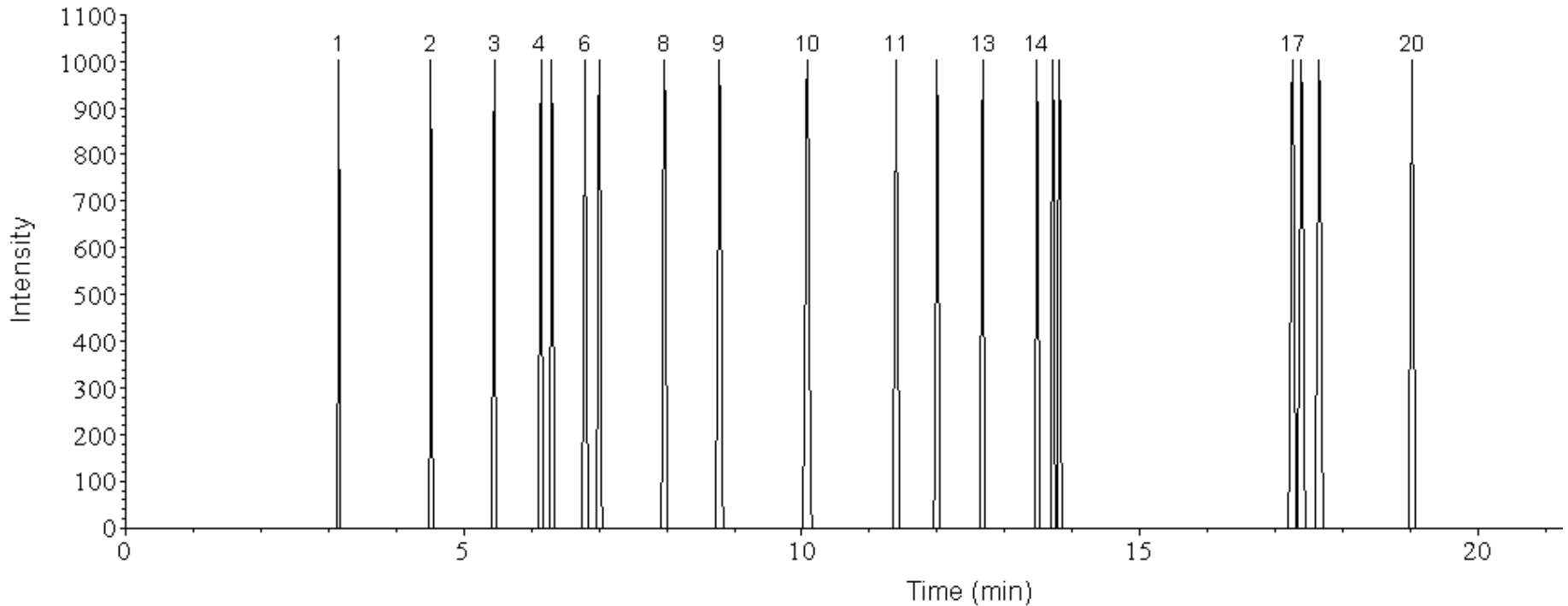
10°C/min to 325°C

Co-elutions occur at 150°C

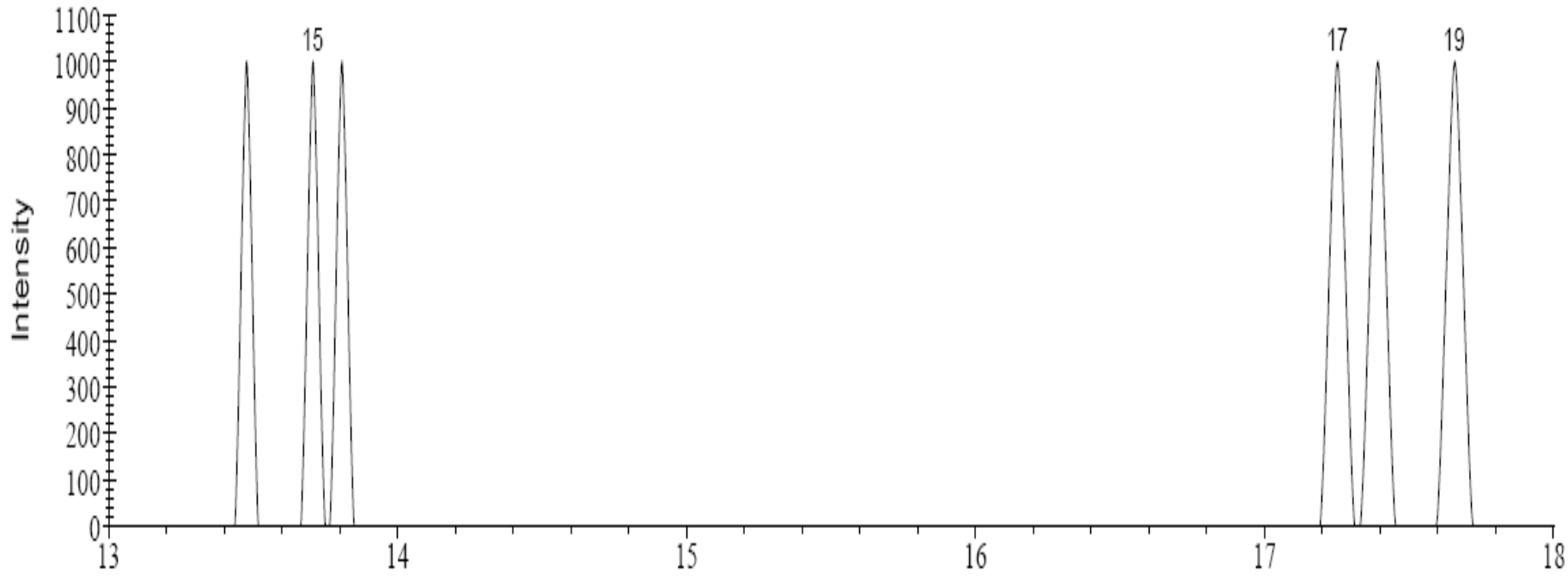
Set hold at 130°C

Developing a Temperature Program

Oven: 100°C Hold for 10 minutes
10°C/min to 130°C hold for 5 min
10°C/min to 325°C



Developing a Temperature Program



Conclusions:

Think about the sample first

****Is it chromatographable by GC?**

sample composition

sample clean up

level of detection

Use information sources first when choosing a column

Mild oven program to begin with

Split injection if possible

Utilize Technical Support

Agilent J&W Scientific Technical Support

800-227-9770 (phone: US & Canada)*

** Select option 3, then 3, then 1.*

866-422-5571 (fax)

GC-Column-support@agilent.com



www.chem.agilent.com