Secrets of GC Column Dimensions

GC Columns and Consumables

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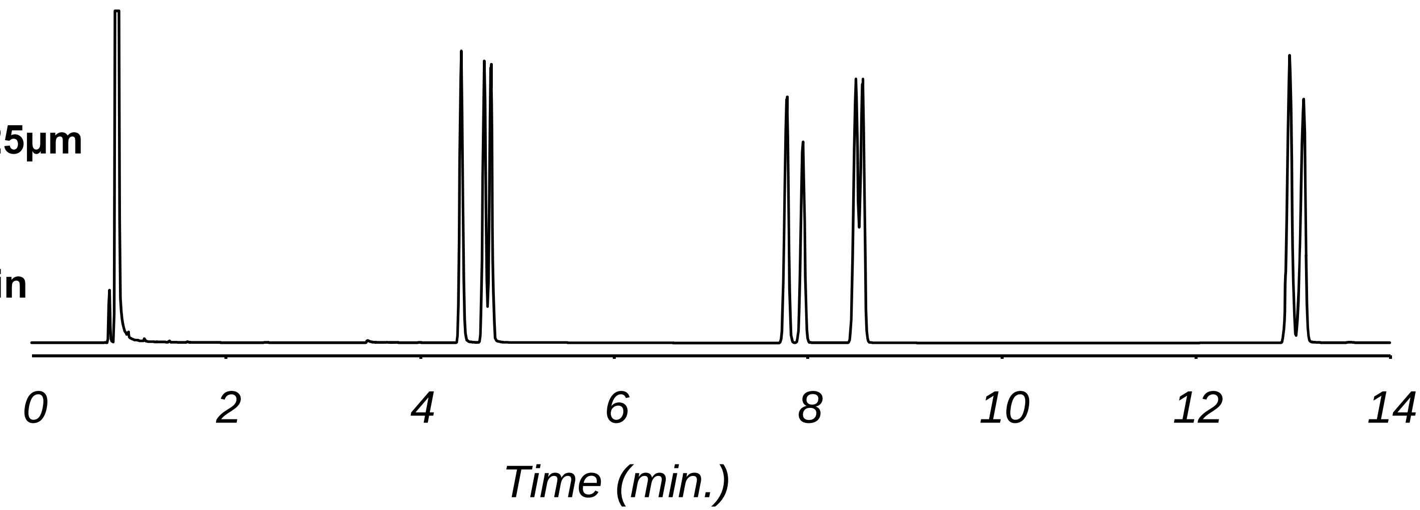
Secrets of GC Column Dimensions

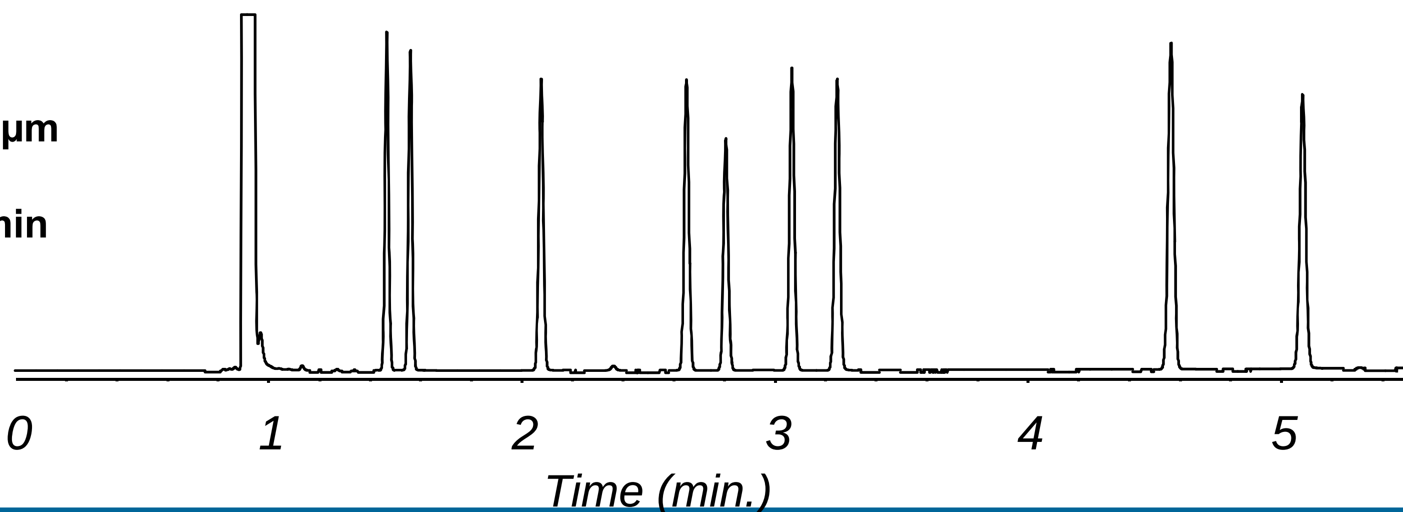
- Do I have the right column phase?
- Resolution Equation
- Changes in Dimensions:
  - Length
  - Diameter
  - Film Thickness
- Method Translation Software
- Carrier gas (if time)

Start with the Right Phase

DB-1
15m x 0.32mm, 0.25µm
Oven:
40°C for 2 min
40-120°C at 5°C/min

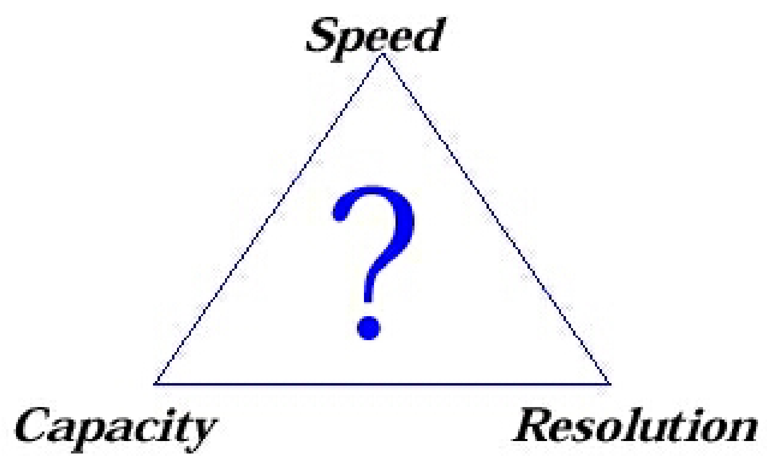


DB-Wax
15m, 0.32mm, 0.25µm
Oven:
80-190°C at 20°C/min



Variables that affect chromatography

- Stationary Phase
- Temperature Programming
- Carrier Gas: type and linear velocity
- Column Length
- Film Thickness
- Internal Diameter



"There's no such thing as a free lunch"

Resolution

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

Efficiency $N = f\ (\text{gas}, L, r_c)$

Retention $k = f\ (T, d_f, r_c)$

Selectivity $\alpha = f\ (T, \text{phase})$

L = Length

r_c = column radius

d_f = film thickness

T = temperature

Column Dimensions

Length

Diameter

Film Thickness

Resolution

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

Efficiency $N = f\ (\text{gas}, L, r_c)$

Retention $k = f\ (T, d_f, r_c)$

Selectivity $\alpha = f\ (T, \text{phase})$

L = Length

r_c = column radius

d_f = film thickness

T = temperature

Column Length and Efficiency (Theoretical Plates)

Length (m)	N
15	69,450
30	138,900
60	277,800

0.25 mm ID
n/m = 4630 (for k = 5)

Column Length and Resolution

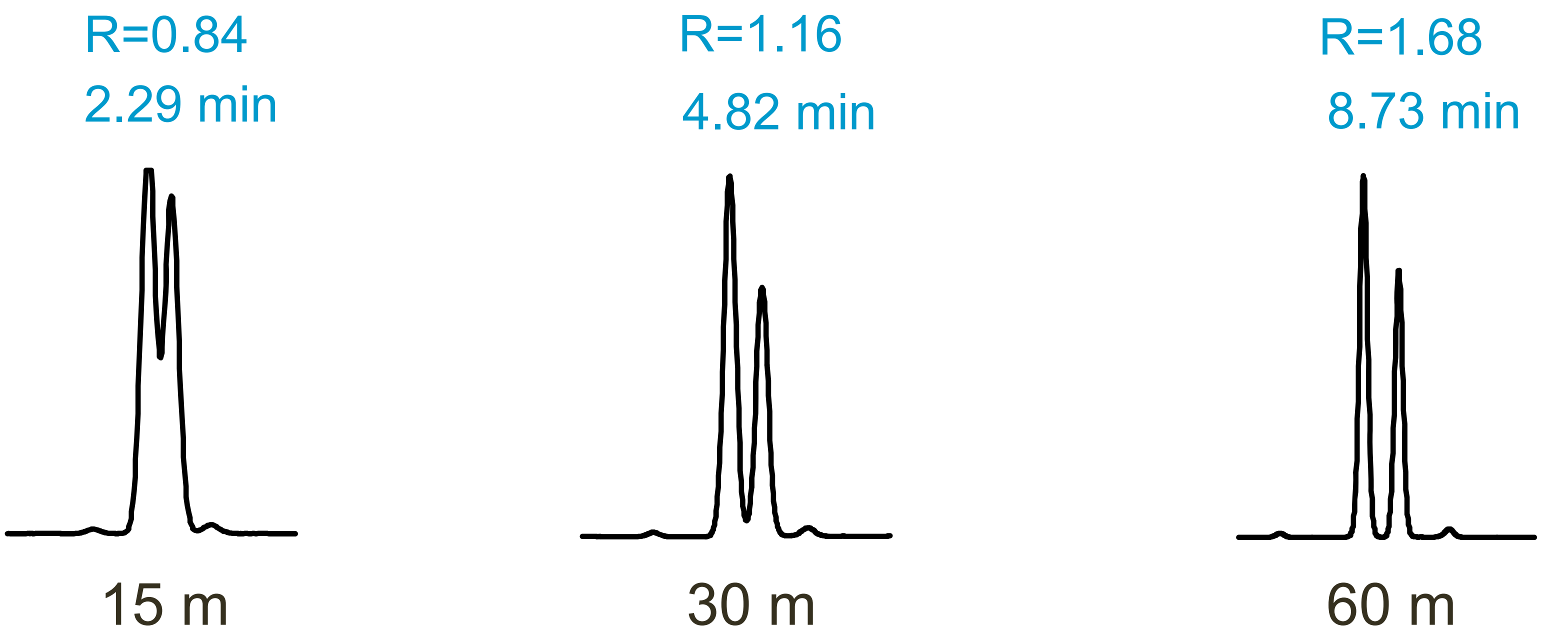
$$R \propto \sqrt{N} \propto \sqrt{L}$$

Length X 4 = Resolution X 2

$$t \propto L$$

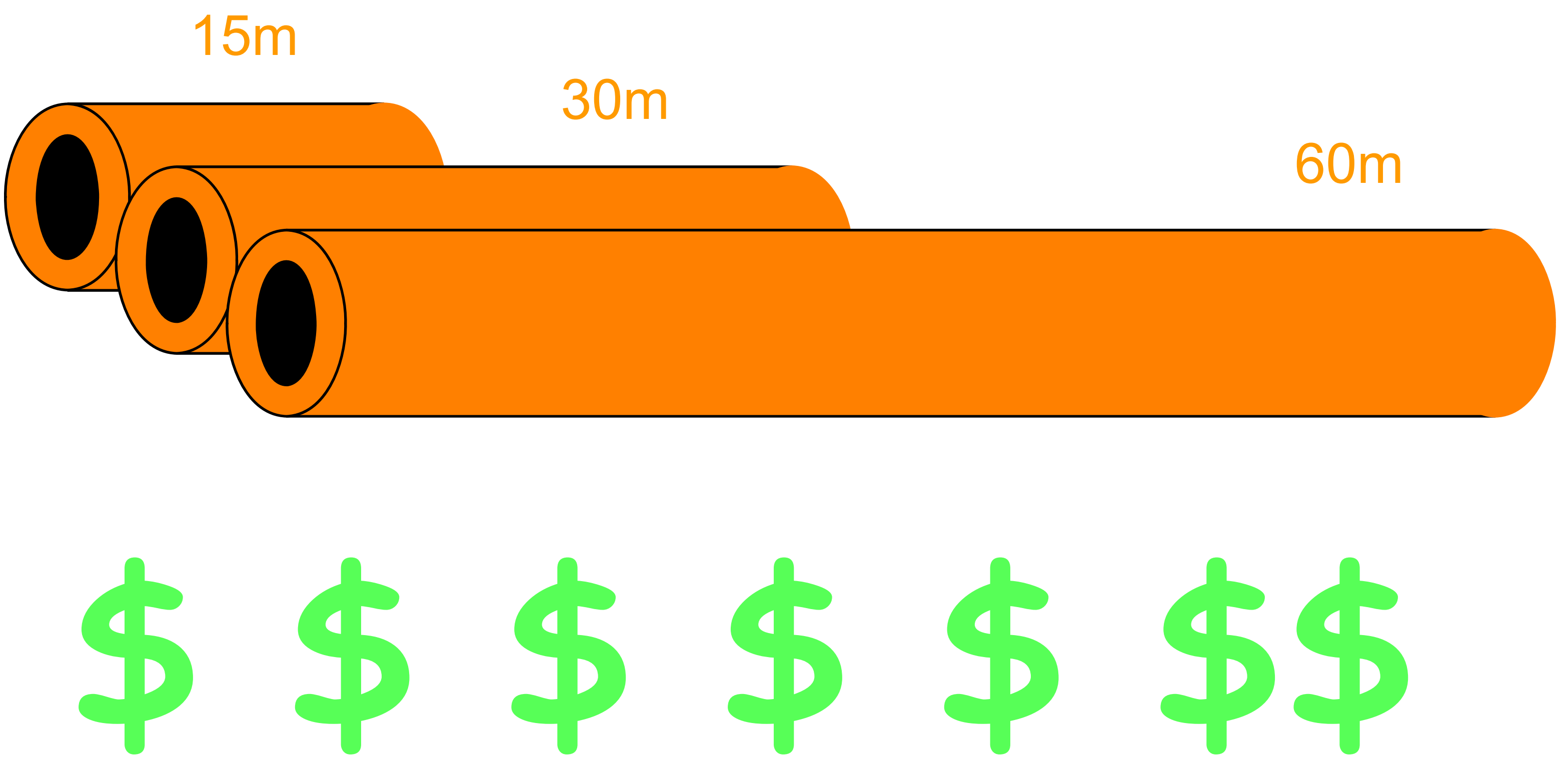
Upside = Cut a bunch off during routine inlet maintenance and not lose a lot of Resolution

Column Length vs Resolution and Retention: Isothermal



Double the plates, double the time
but not double the resolution

Column Length and Cost



Length Summary

If you Increase Length:

Efficiency	Increase
Resolution	Increase
Analysis Time	Increase
Pressure	Increase
Cost	Increase

Resolution

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

Efficiency	$N = f\ (\text{gas}, L, r_c)$	L = Length
Retention	$k = f\ (T, d_f, r_c)$	r_c = column radius
Selectivity	$\alpha = f\ (T, \text{phase})$	d_f = film thickness
		T = temperature

Column Diameter and Carrier Gas Flow

Lower flow rates: Smaller diameter columns

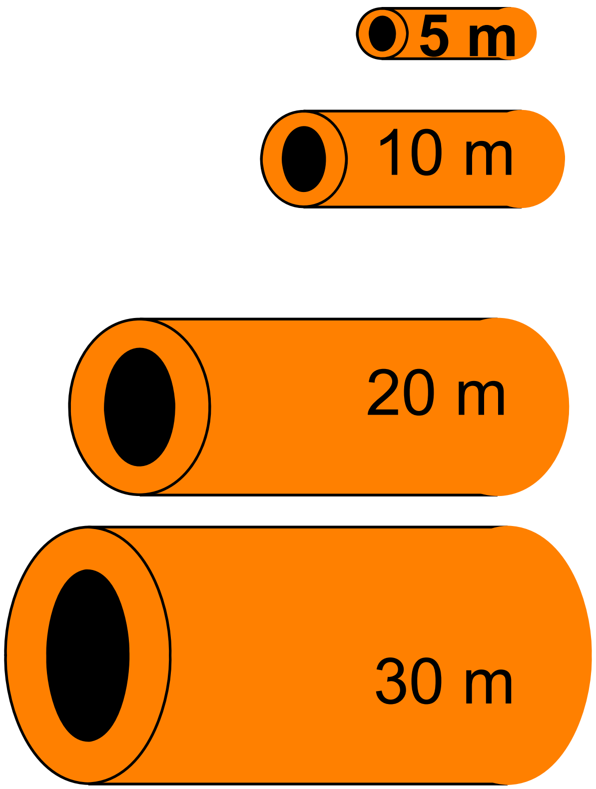
Higher flow rates: Larger diameter columns

Low flow rates : GC/MS
High flow rates: Headspace, purge & trap

Column Diameter
Capillary Columns

I.D. (mm)	Common Name
0.53	Megabore
0.45	High speed Megabore
0.32	Widebore
0.20-0.25	Narrowbore
0.18	Minibore
0.10	Microbore
0.05	“Nanobore”

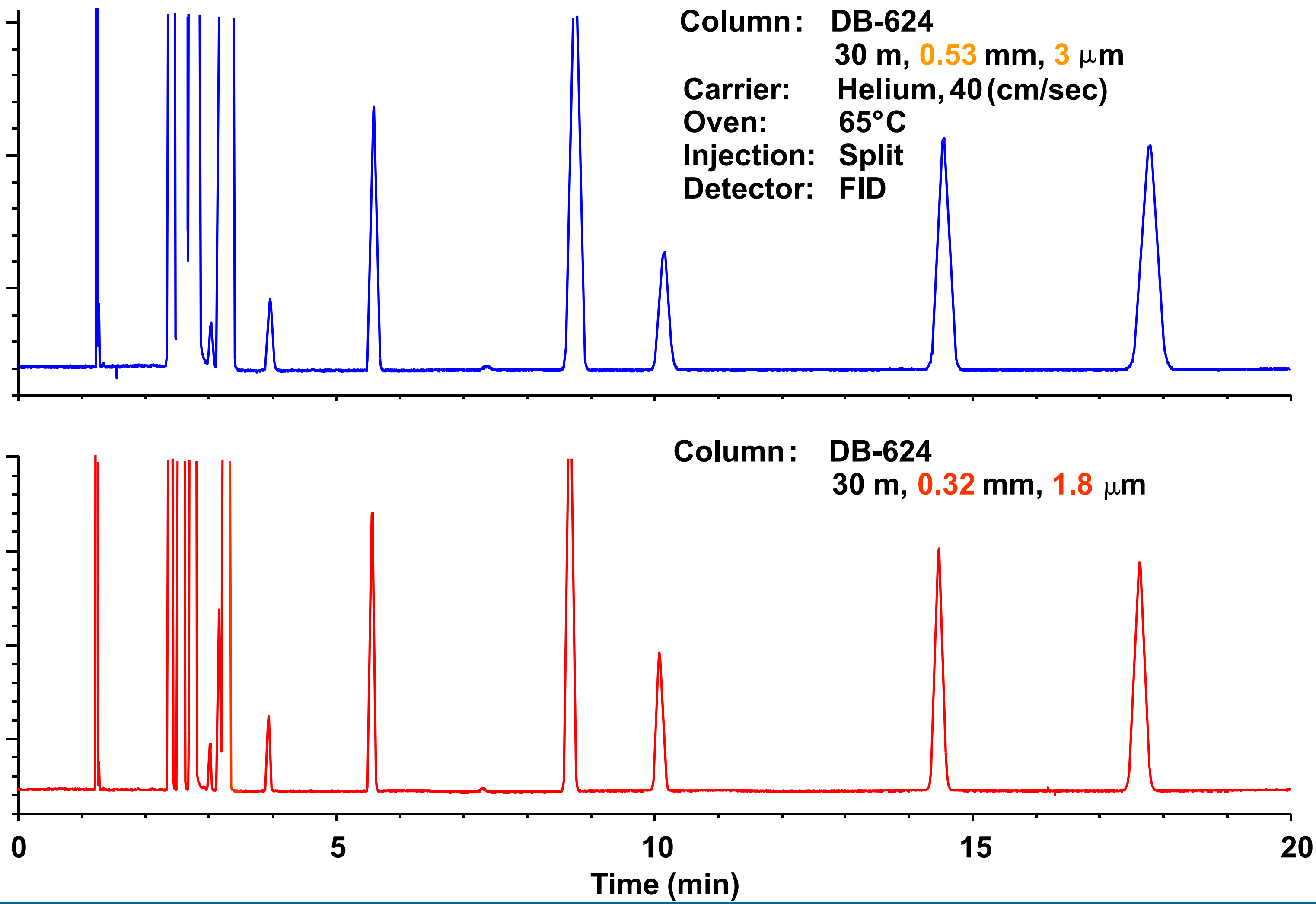
Column Diameter - Theoretical Efficiency



Total Plates	I.D. (mm)	n/m
N ~ 112,000	0.05	23,160
N ~ 112,000	0.10	11,580
	0.18	6,660
N ~ 112,000	0.20	5830
	0.25	4630
N ~ 112,000	0.32	3660
	0.45	2840
	0.53	2060

k = 5

Different Column I. D.
Equal Phase Ratios



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PHASE RATIO (β)

Film Thickness

Column Dimensions	Phase Ratio β
30 m x .53 mm x 3.0 μm	44
30 m x .32 mm x 1.8 μm	44

$$K_C = k\,\beta$$

$$\beta = \frac{r}{2d_f}$$

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High Resolution Megabore (0.45 mm diameter)

- **Same outer diameter as the Megabore**
- **No special hardware required**
- **Smaller inner diameter (0.45mm)**
- **Maintain phase ratio (Beta)**
- **Methods are easy to translate!**

High Resolution Megabore

Column Dimensions	Phase Ratio β
30 m x .53 mm x 3.0 μm	44
30 m x .45 mm x 2.55 μm	44

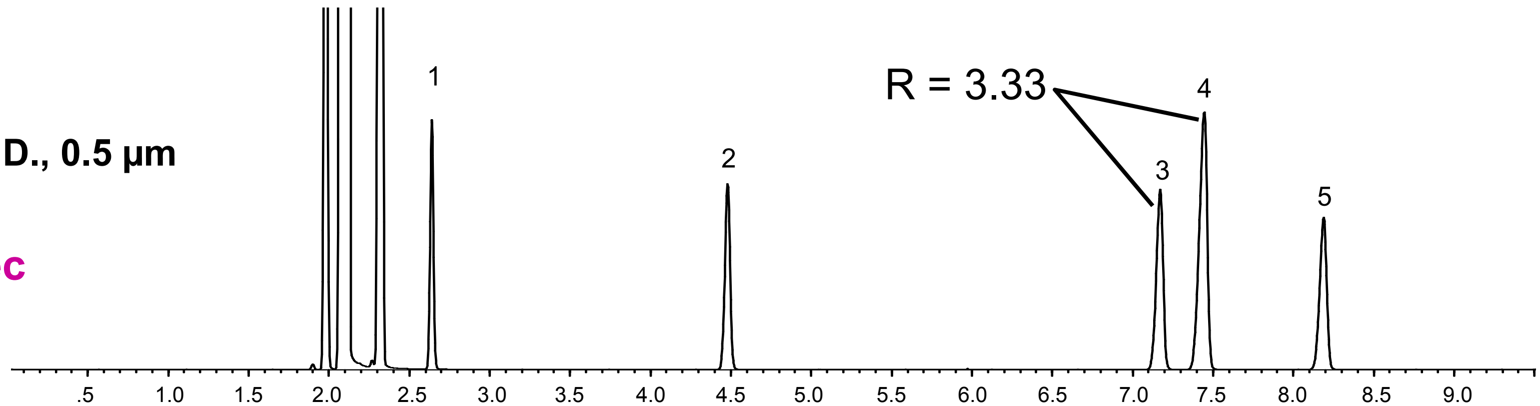
Column phase	Column length	Internal diameter	Film thickness
DB-VRX	75 meters	0.449 mm	2.55 μm
	75 meters	0.540 mm	3.00 μm
DB-624	75 meters	0.446 mm	2.55 μm
	75 meters	0.546 mm	3.00 μm
DB-502.2	75 meters	0.453 mm	2.55 μm
	105 meters	0.544 mm	3.00 μm
DB-WAX	30 meters	0.447 mm	0.85 μm
	30 meters	0.544 mm	1.00 μm
DB-1	30 meters	0.455 mm	1.30 μm
	30 meters	0.551 mm	1.50 μm
DB-5	30 meters	0.446 mm	1.30 μm
	30 meters	0.540 mm	1.50 μm
DB-608	30 meters	0.450 mm	0.71 μm
	30 meters	0.535 mm	0.83 μm

High-SPEED Megabore

Same Resolution - Faster Analysis!

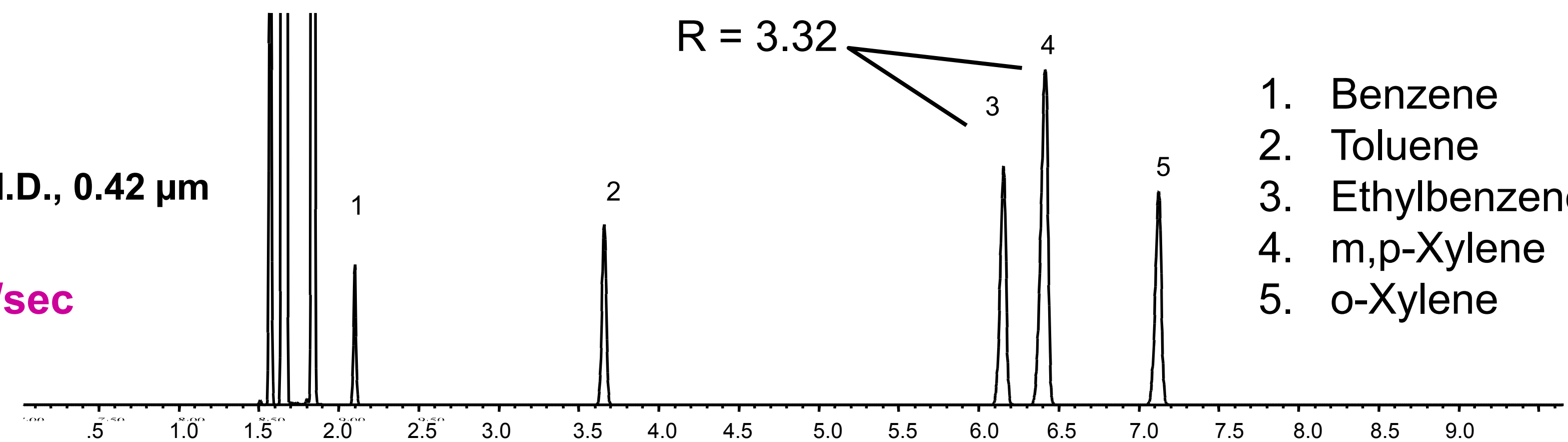
DB-5
30 m,
0.53 mm I.D., 0.5 µm

36 cm/sec



DB-5
30 m,
0.45 mm I.D., 0.42 µm

45.9 cm/sec

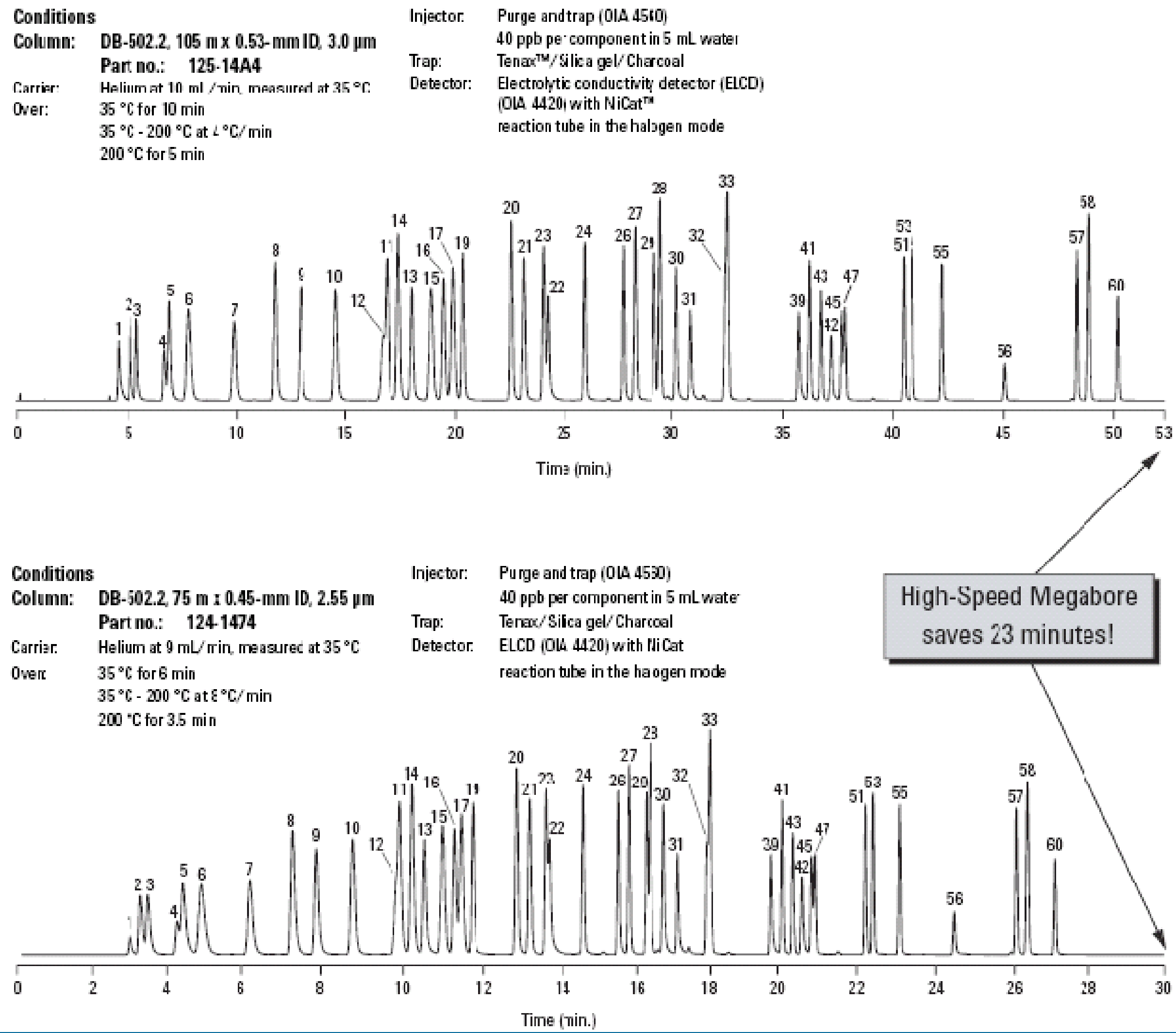


1. Benzene
2. Toluene
3. Ethylbenzene
4. m,p-Xylene
5. o-Xylene

BTEX
Carrier: Helium
Oven : 40°C for 3 min, 5°/min to 100°C

High SPEED Megabore

Same Resolution - Faster Analysis!



"Increasing Sample Throughput With High-Speed Megabore"

Application note 5988-5271EN

Column Diameter and Capacity

I.D. (mm)	Capacity (ng)
0.05	1-2
0.10	6-13
0.18	25-55
0.20	35-70
0.25	80-160
0.32	110-220
0.45	600-800
0.53	1000-2000

Like Polarity
Phase/Solute
0.25 µm film thickness

Column Diameter - Inlet Head Pressures (Helium)

I.D (mm)	Pressure (psig)
0.05	275-400
0.10	90-130
0.18	30-45
0.20	25-40
0.25	15-25
0.32	10-20
0.45	3-7
0.53	2-4

30 meters
Hydrogen pressures x 1/2

Diameter Summary

If you decrease the inside diameter:

Efficiency	Increase
Resolution	Increase
Pressure	Increase
Capacity	Decrease
Flow rate	Decrease

Resolution

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

Efficiency $N = f\ (\text{gas}, L, r_c)$

Retention $k = f\ (T, d_f, r_c)$

Selectivity $\alpha = f\ (T, \text{phase})$

L = Length

r_c = column radius

d_f = film thickness

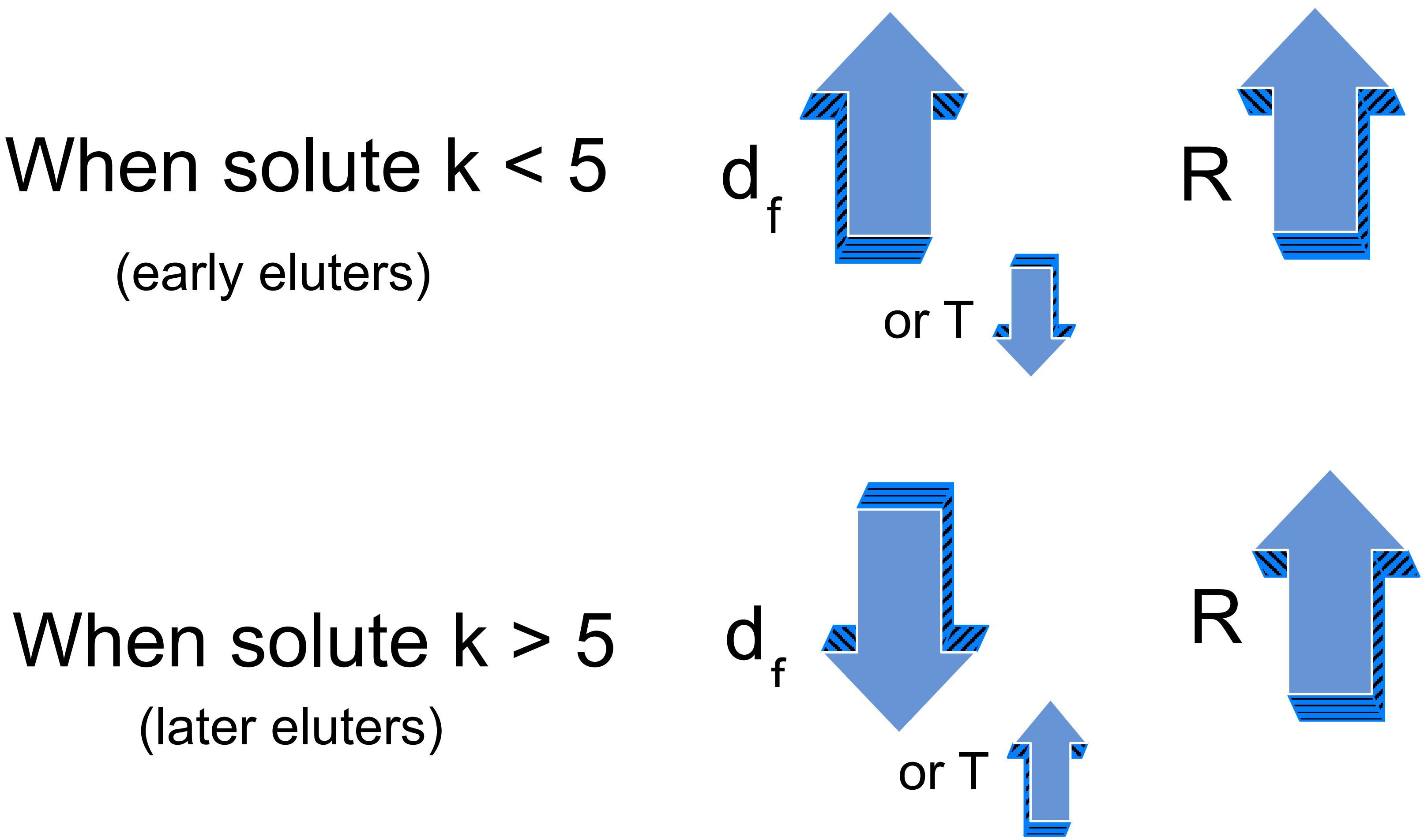
T = temperature

Film Thickness and Retention: Isothermal

Thickness (µm)	Retention Change
0.10	0.40
0.25	*1.00*
1.0	4.00
3.0	12.0
5.0	20.0

Constant Diameter
Normalized to 0.25 µm

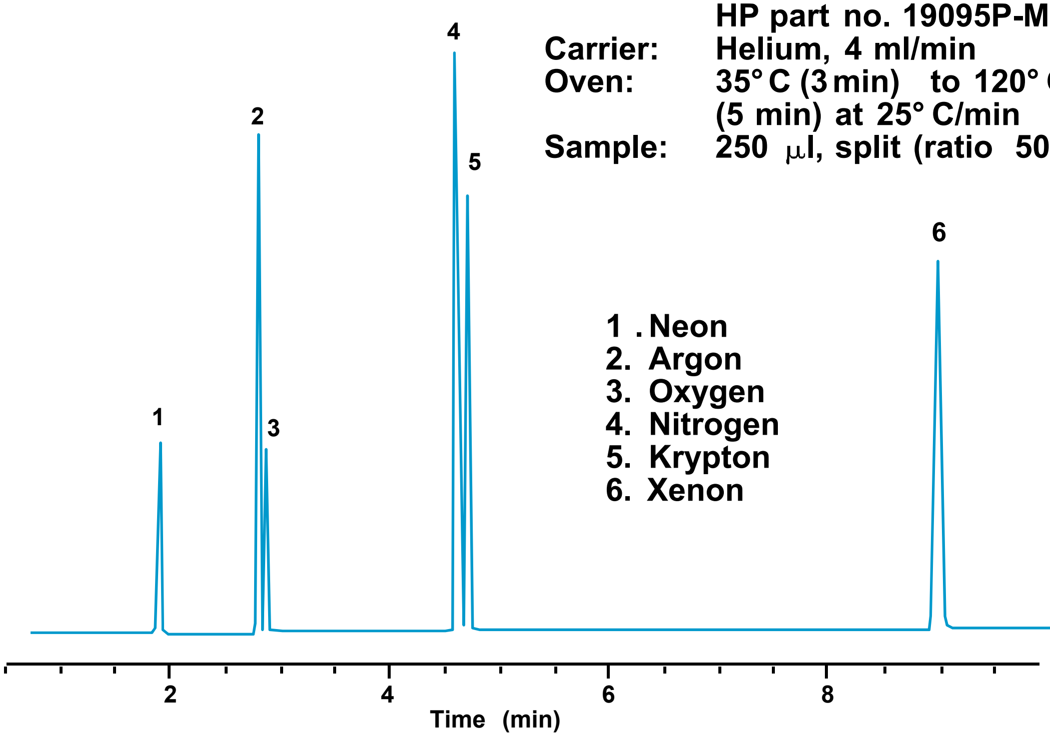
Film Thickness and Resolution



Other Retention - Adsorption

Analysis of Noble & Fixed Gases Using HP PLOT MoleSieve

Column:	**HP-PLOT/MoleSieve**
	30 m x 0.53 mm x 50 μm
	HP part no. 19095P-MS0
Carrier:	**Helium, 4 ml/min**
Oven:	**35° C (3 min) to 120° C**
	(5 min) at 25° C/min
Sample:	**250 μl, split (ratio 50:1)**



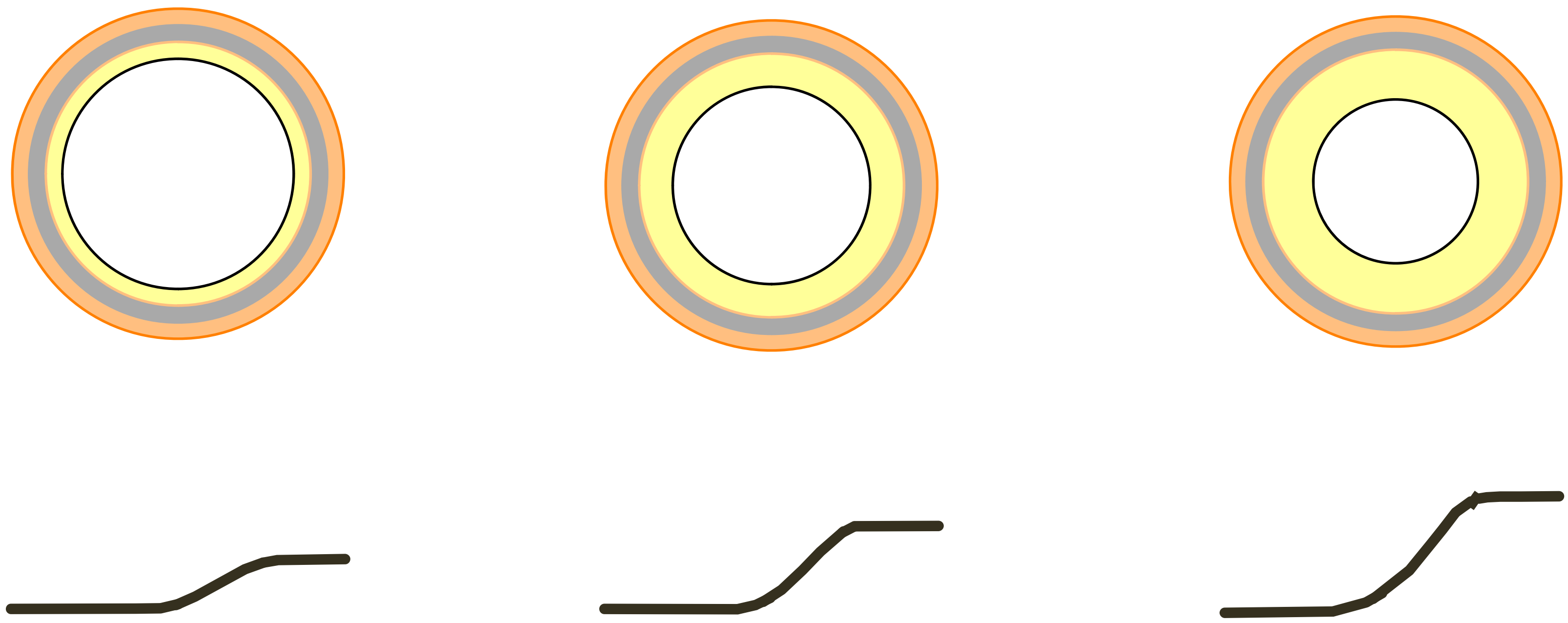
Film Thickness and Capacity

Thickness (µm)	Capacity (ng)
0.10	50-100
0.25	125-250
0.50	250-300
1	500-1000
3	1500-3000
5	2500-5000

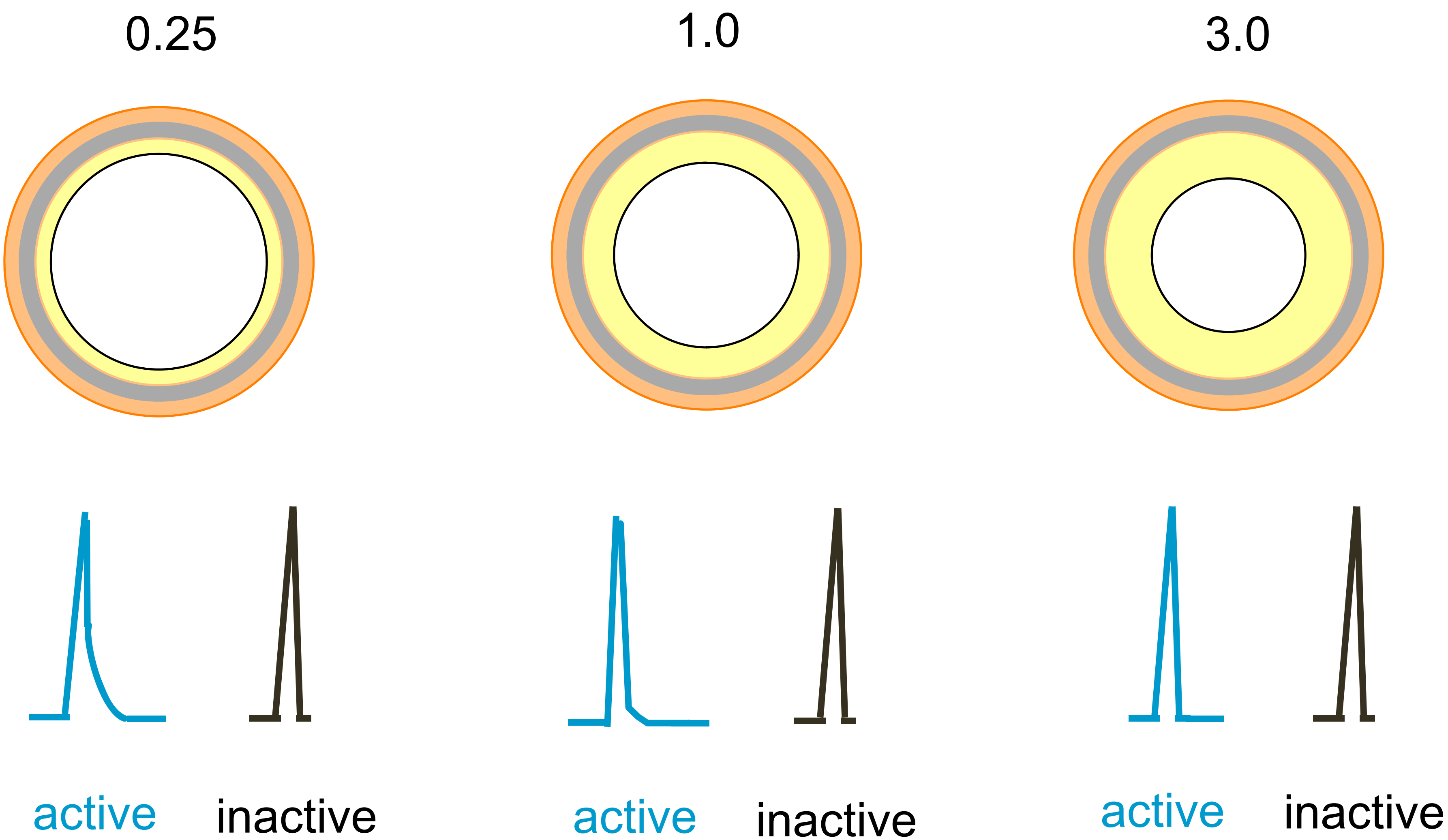
0.32 mm I.D.
Like Polarity Phase/Solute

Film Thickness and Bleed

More stationary phase = More degradation products



Film Thickness and Inertness



Film Thickness Summary

If you increase the film thickness:

Retention	Increase
Resolution (k<5)	Increase
Resolution (k>5)	Decrease
Capacity	Increase
Bleed	Increase
Inertness	Increase
Efficiency	Decrease

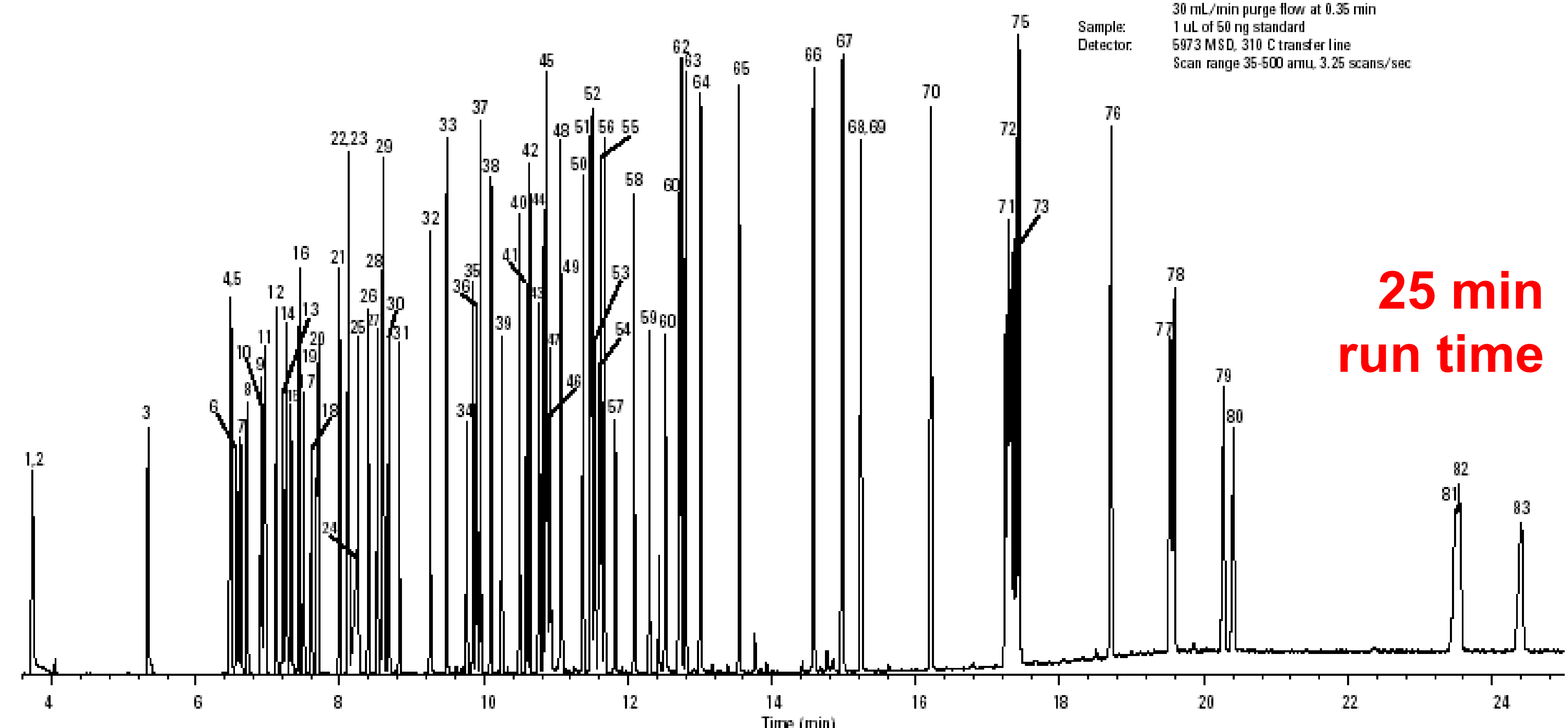
GC Column Dimensions

Examples…….

Method 8270
– 30m x 0.25mm ID, 0.50 µm

USEPA Method 8270 Semivolatile Compounds

Column:	HP-5ms
	30 m x 0.25 mm I.D., 0.5 um
P/N:	19091S-133
Carrier:	Ramped flow
	1.2 mL/min for 0.0 min
	ramp at 99 mL/min to 2.0 mL/min
	2.0 mL/min for 0.35 min
	ramp at 10 mL/min to 1.2 mL/min
Oven:	40 C for 1.0 min
	40-100 C at 15 C/min
	100-240 C at 20 C/min
	240-310 C at 10 C/min
Inlet:	Splitless, 250 C
	30 mL/min purge flow at 0.35 min
Sample:	1 uL of 50 ng standard
Detector:	5973 MSD, 310 C transfer line
	Scan range 35-500 amu, 3.25 scans/sec



25 min run time

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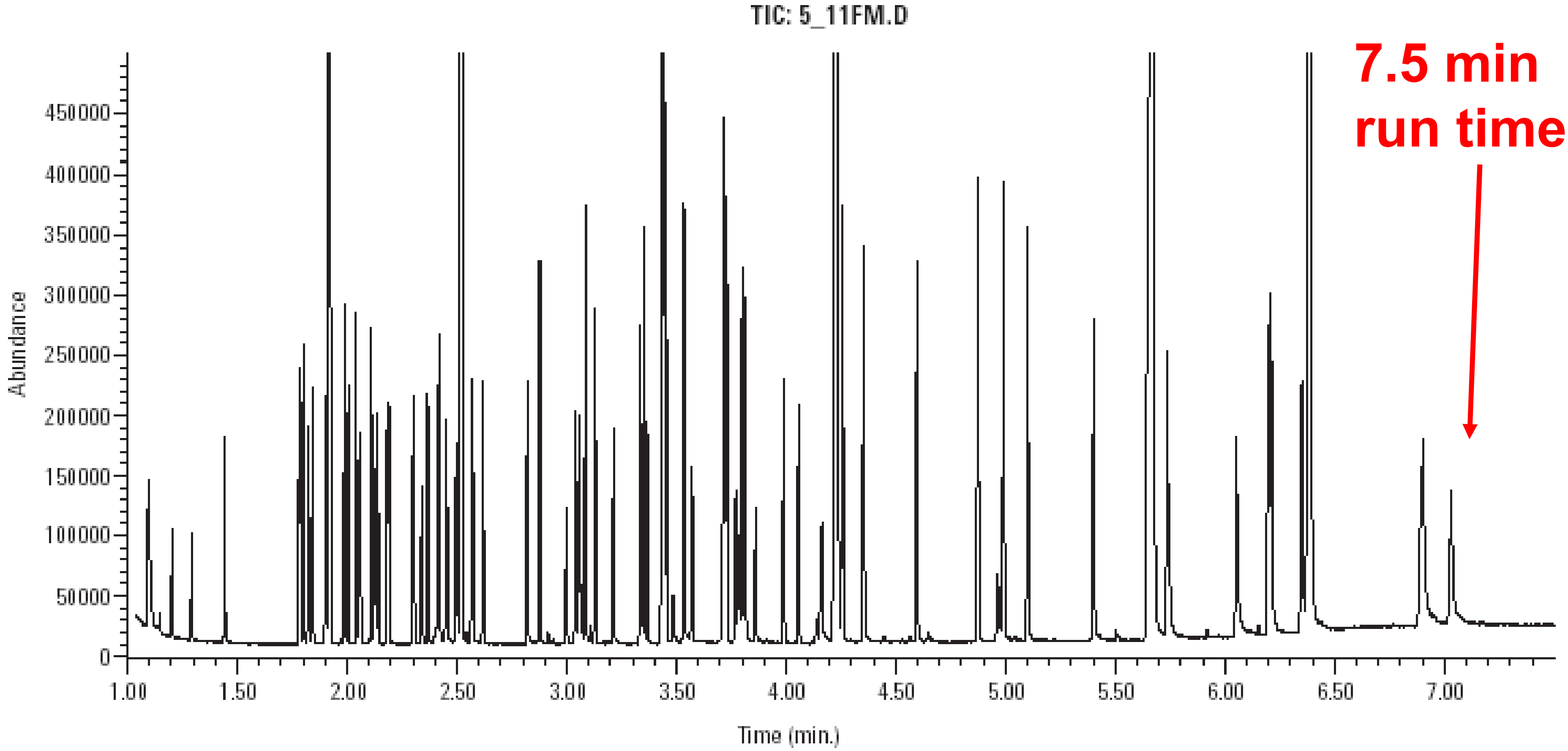
Fast 8270 Semivolatile Analysis
12.5m X 100 µm ID HP-5ms column



But….is this practical?

See Agilent application note #5989-0207EN for more details.

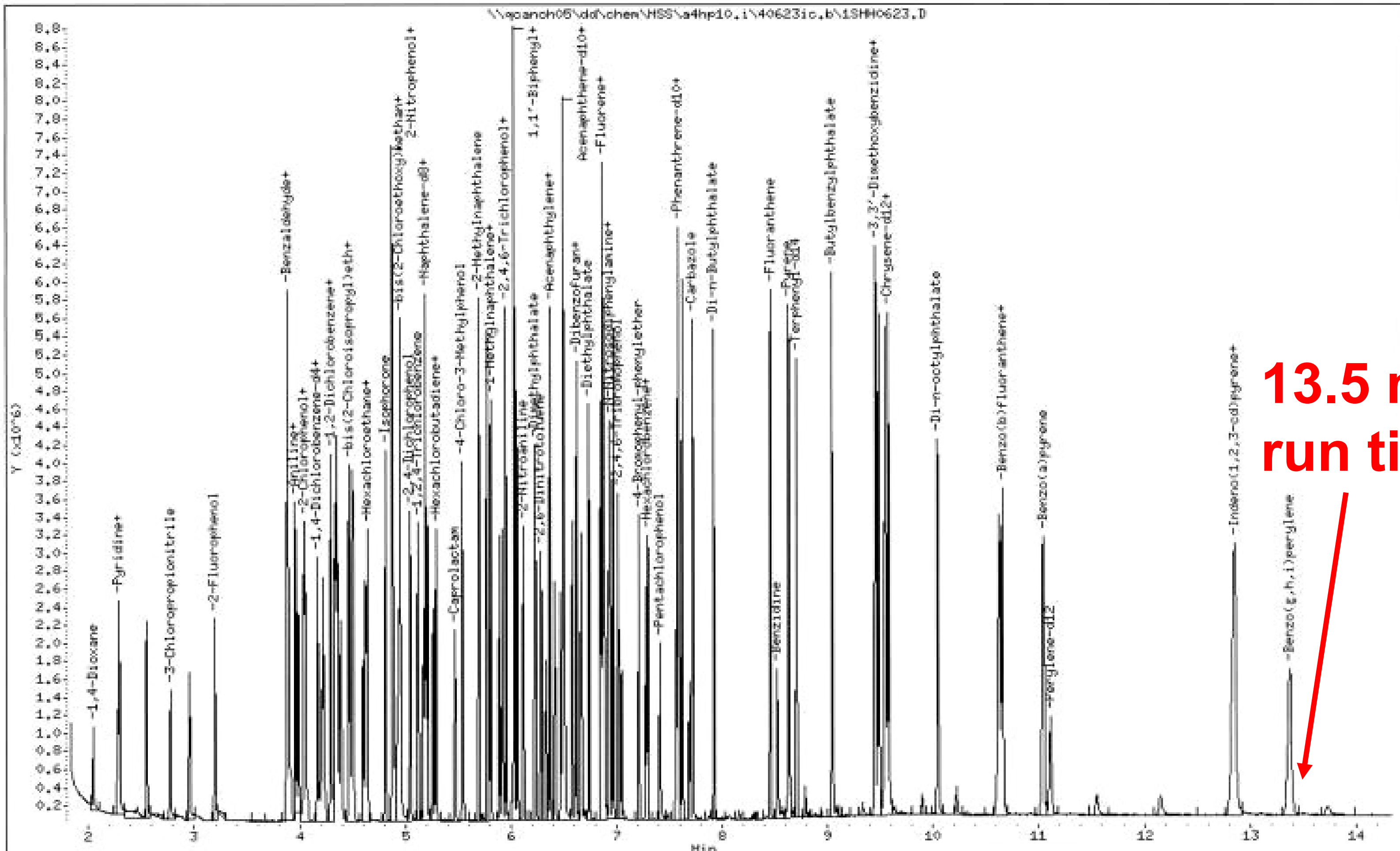
Running Samples on 100 µm ID Columns – Practical?

Environmental type samples = high contaminant residue potential. Smaller ID columns have reduced capacity for matrix contaminants due to lower surface area, shorter length (less forgiving).

Less surface area…a 10 m x .10 mm column has 7.5X less overall surface area than a 30 m x 0.25 mm ID column.

A more robust solution might be to switch to a 20 m x 0.18 mm ID column (nice middle-ground between 0.25 and 0.10). These columns could be used in splitless or in split mode, whereas the 0.10 mm ID columns are practically limited to split introduction.

Fast 8270 Semivolatile Analysis
20m x 0.18mm ID x 0.36um, DB-5.625



13.5 min run time

Flow program or faster temperature program?

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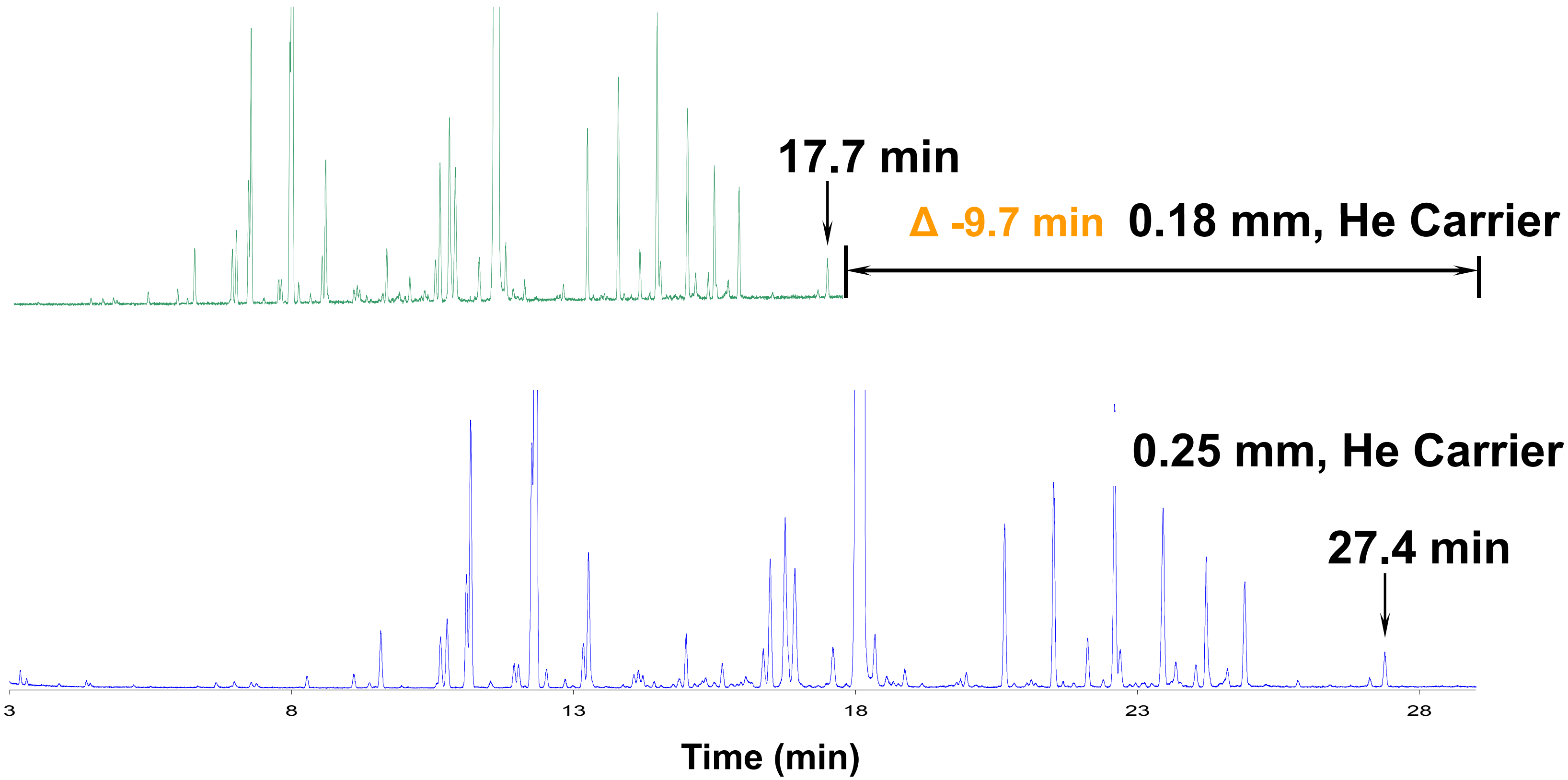
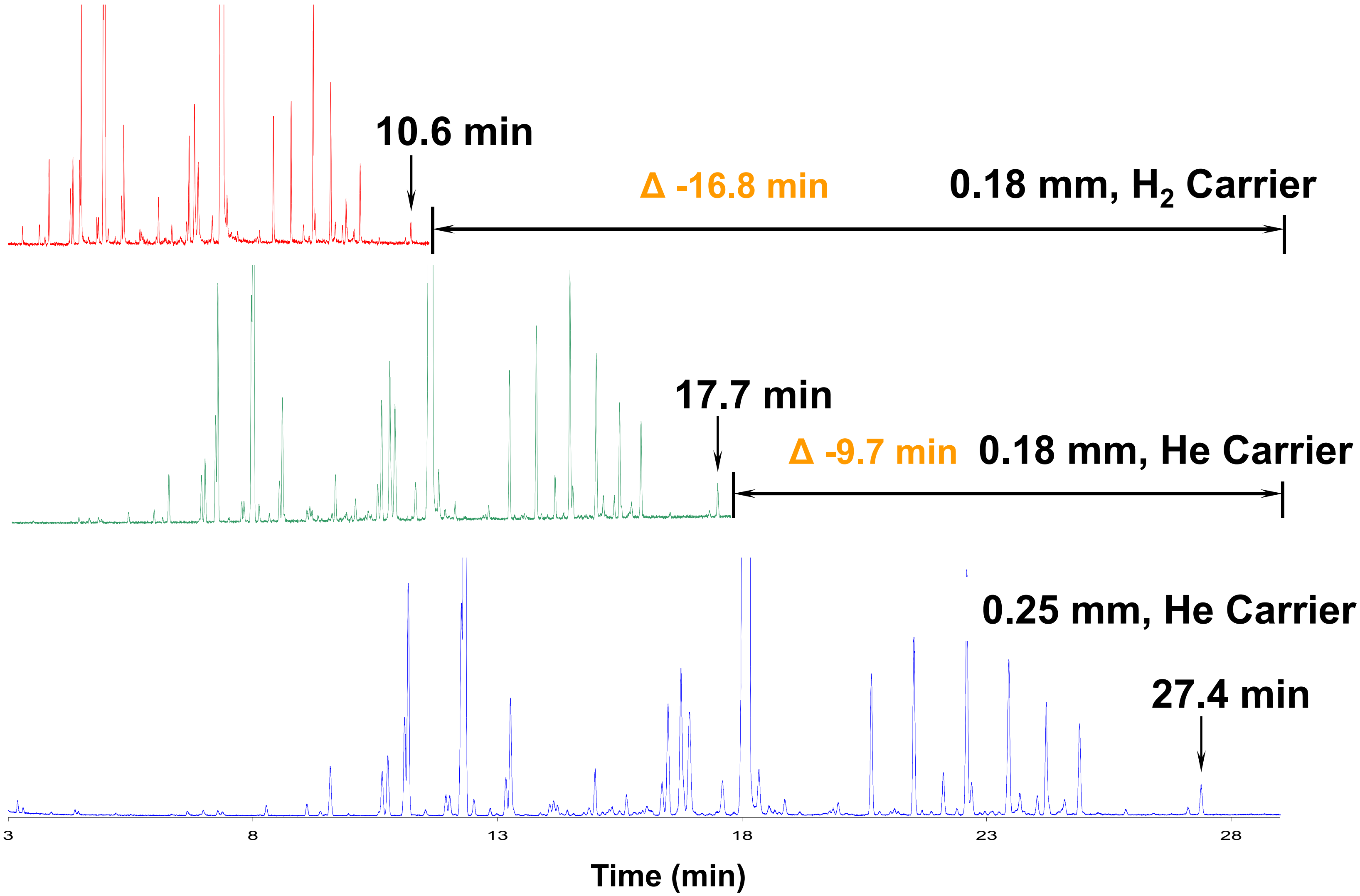
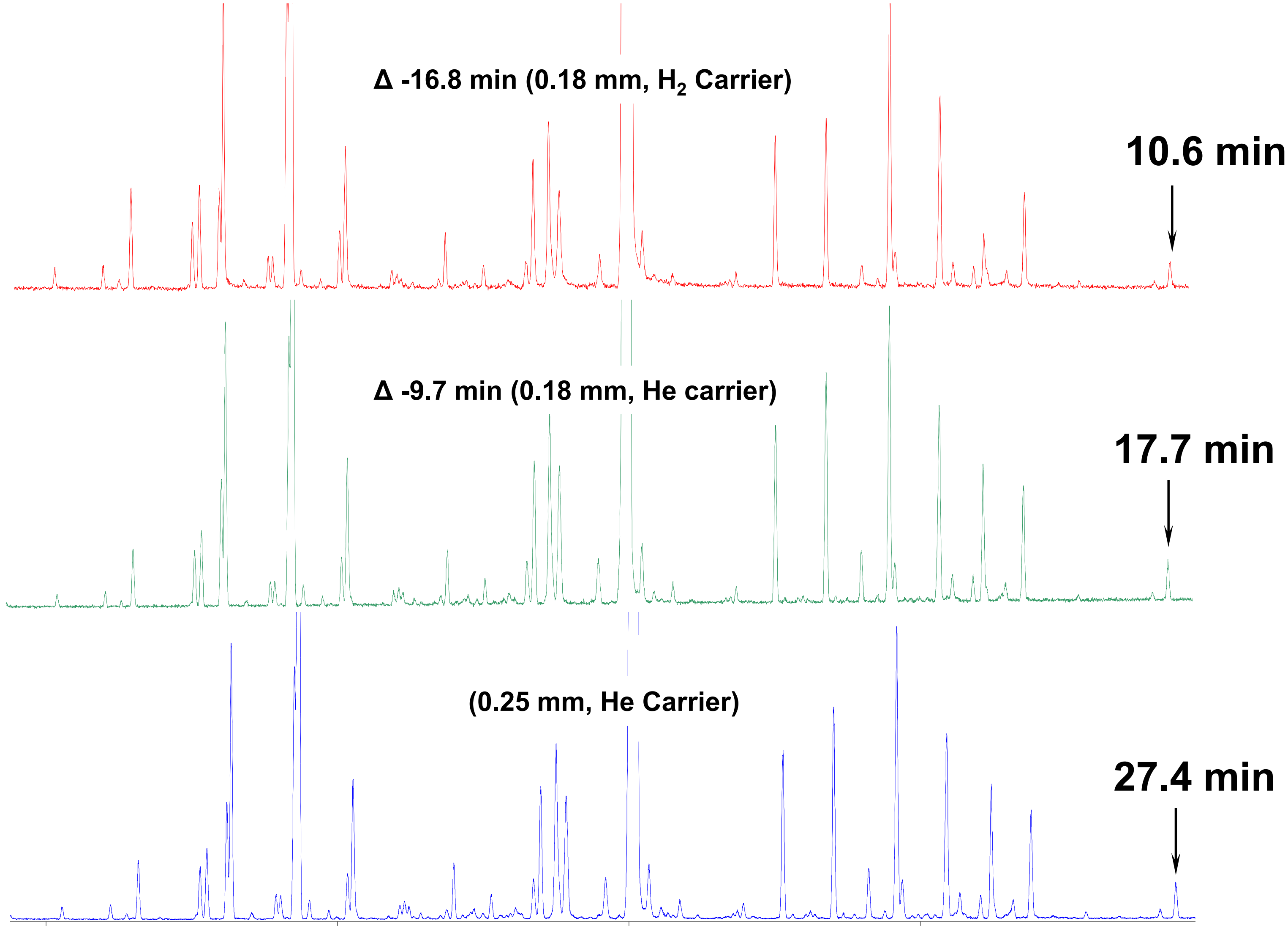
Spearmint Oil on DB-1



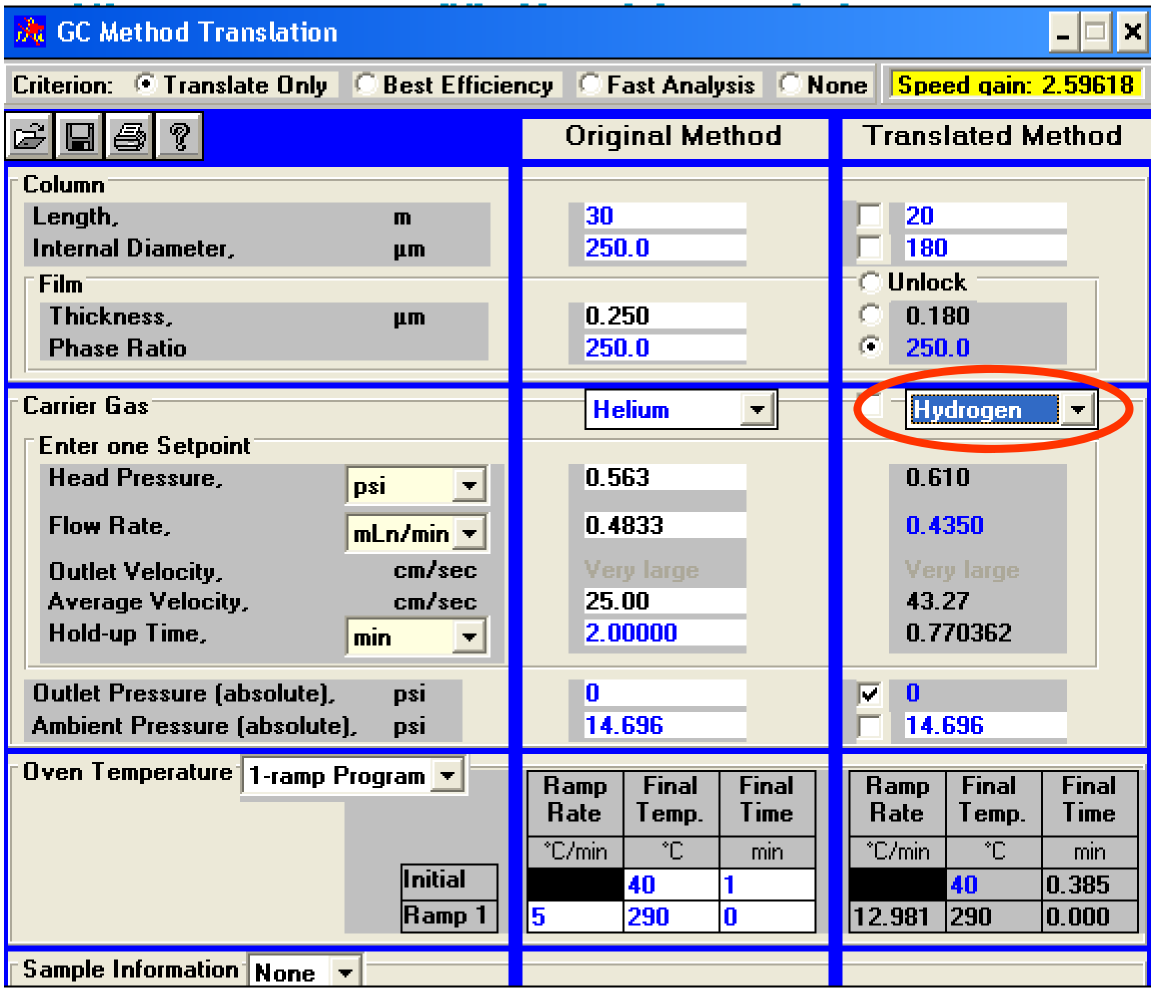
Spearmint Oil on DB-1, (App. Note 5989-7509EN)



Spearmint Oil on DB-1 – Resolution Check



Food/Fragrance – Method translation, Hydrogen



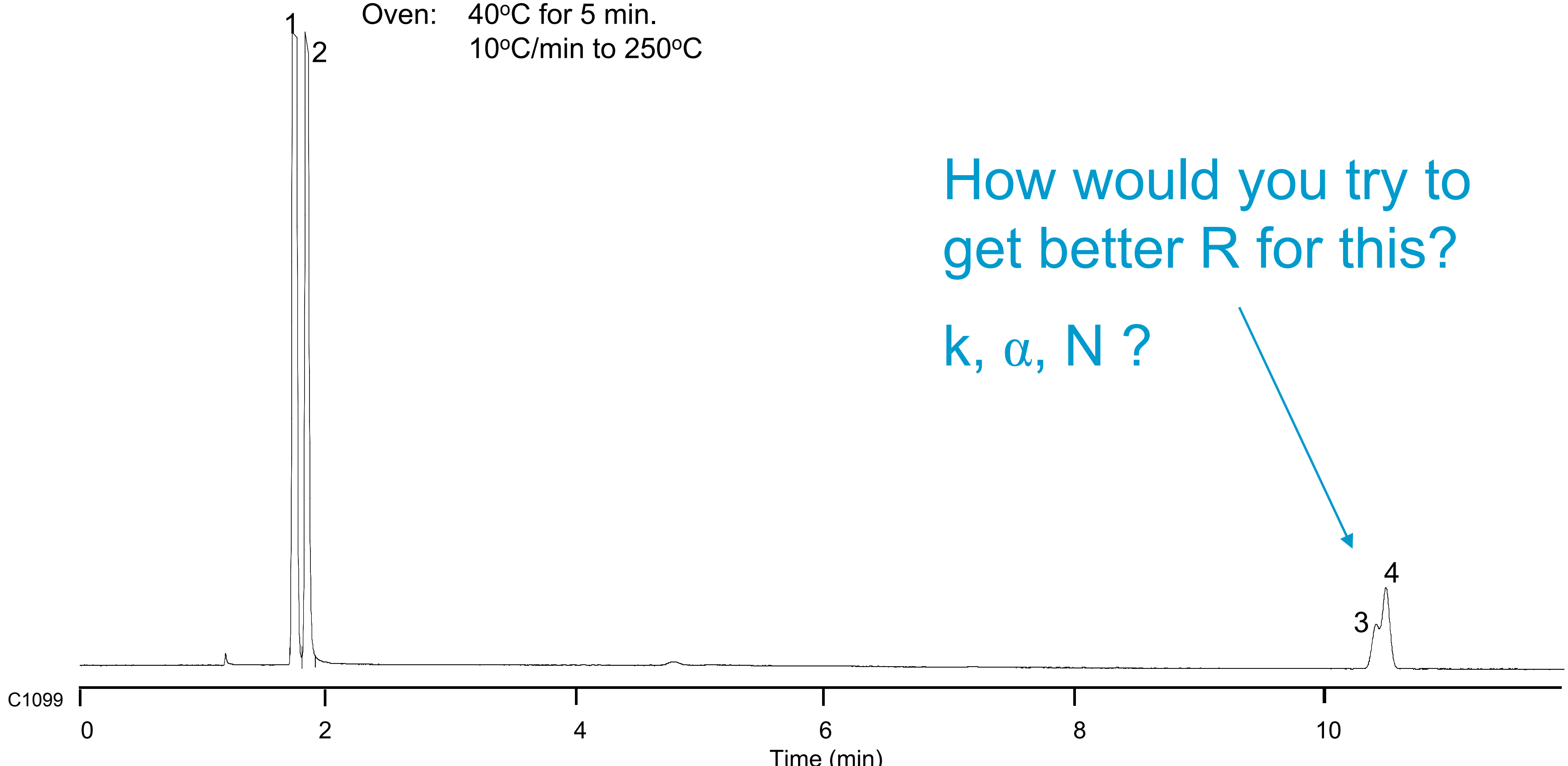
OK, Test Time

Fusel Oil Simple Standard

DB-624
25 m x .53 mm I.D. x 3.0 µm

Inlet: 250ºC, split
FID: 300ºC
Carrier: H_2, 50 cm/sec
Oven: 40ºC for 5 min.
10ºC/min to 250ºC

1. acetaldehyde
2. methanol
3. 3-methyl-butanol (isoamyl alcohol)
4. 2-methyl-butanol (active amyl alcohol)



How would you try to get better R for this?

k, α, N ?

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The Secrets of GC Column Dimensions
Agilent Restricted

Need Plates?…
Length AND Column Diameter

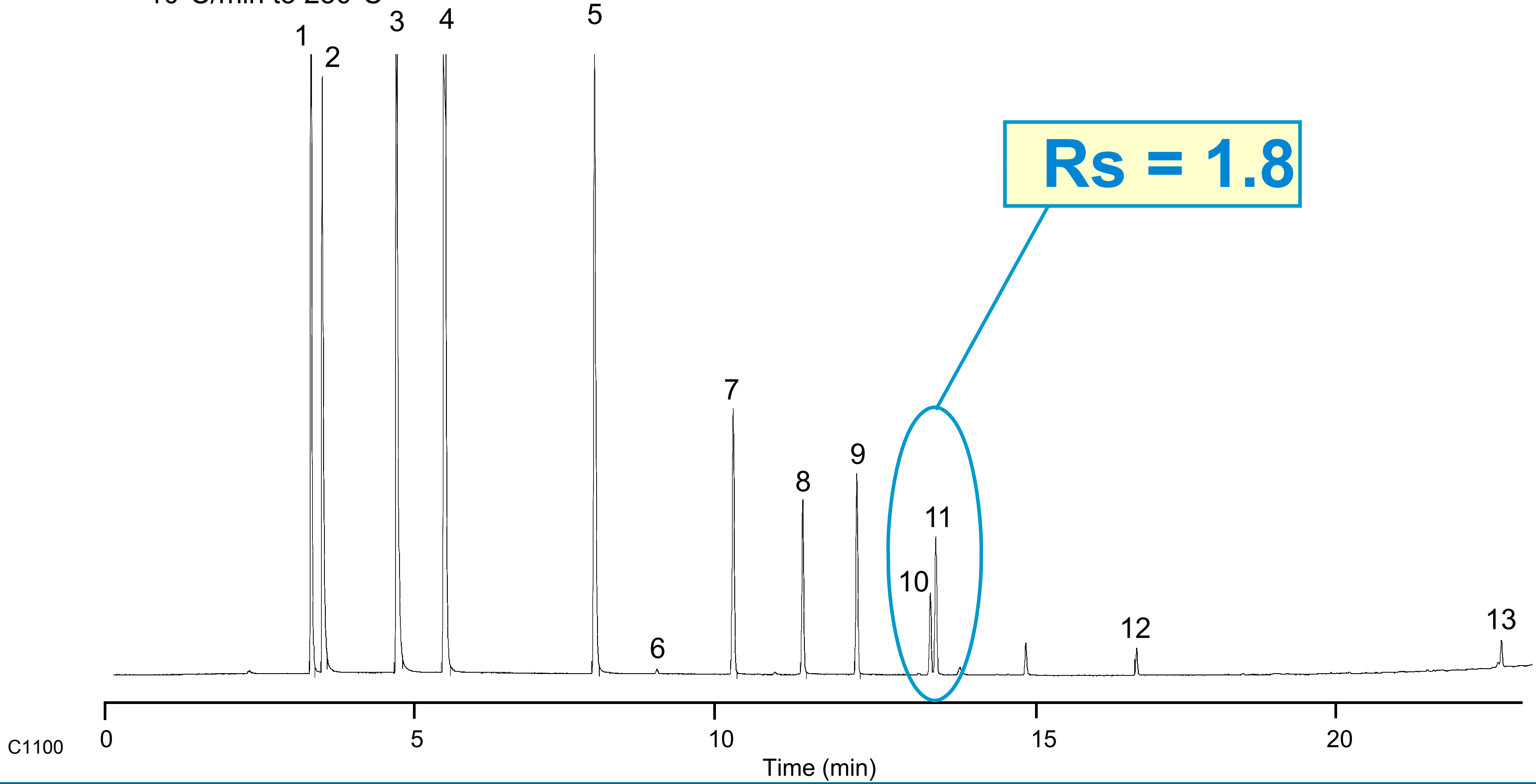
Column Dimensions	Theoretical Plates
25 m x .53 mm x 3.0 µm	34,530
60 m x .25 mm x 1.4 µm	181,860

Fusel Oil Standard

DB-624
60 m x .25 mm I.D. x 1.4 µm

Inlet: 250°C, split
FID: 300°C
Carrier: H_2, 50 cm/sec
Oven: 40°C for 5 min.
10°C/min to 250°C

1. acetaldehyde
2. methanol
3. ethanol
4. acetone
5. 1-propanol
6. ethyl acetate
7. isobutanol
8. 1-butanol
9. 3-pentanol (IS)
10. 3-methyl-butanol (isoamyl alcohol)
11. 2-methyl-butanol (active amyl alcohol)
12. hexanol
13. phenylethanol



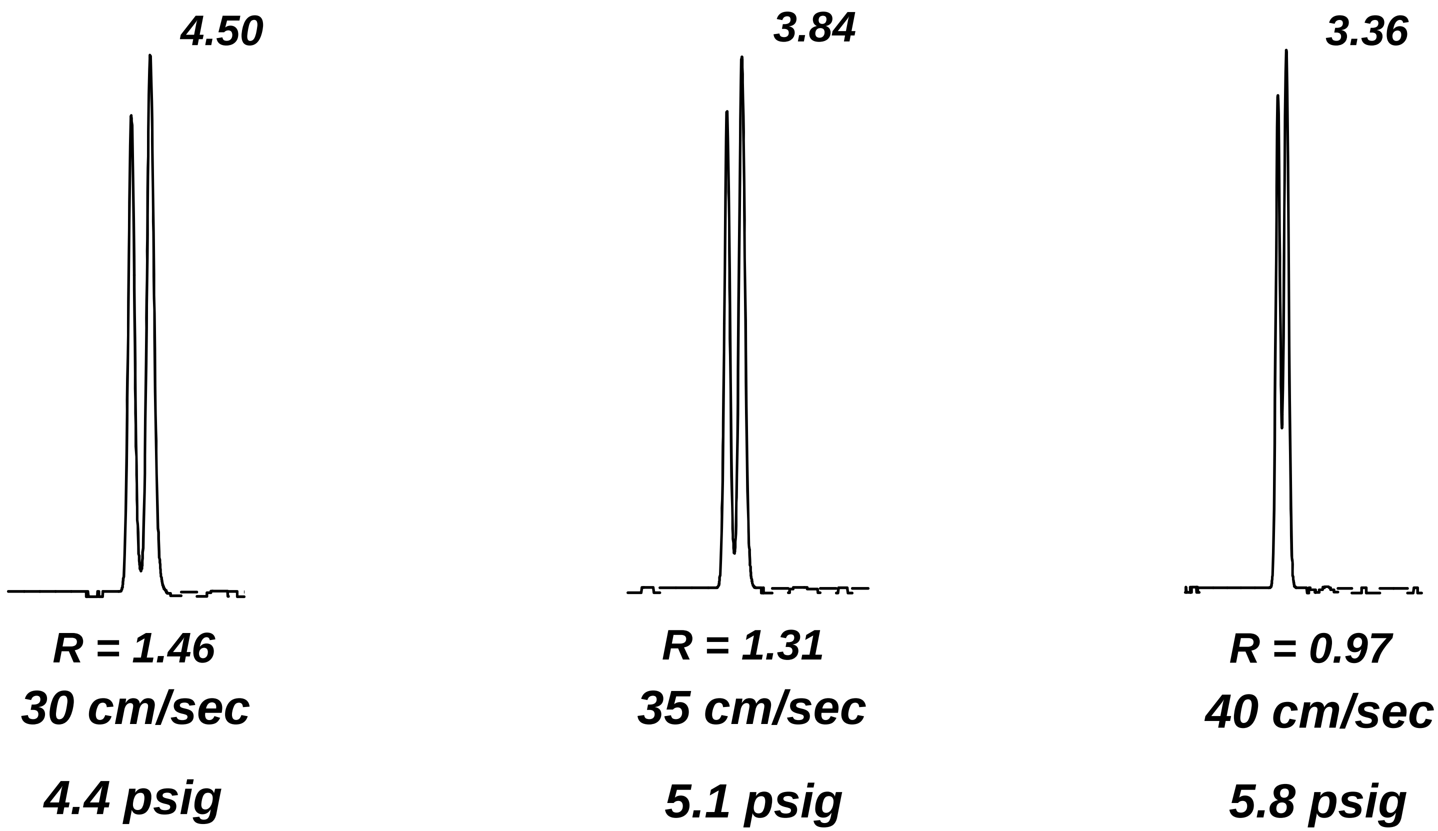
CARRIER GAS

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

RESOLUTION VS. LINEAR VELOCITY

Helium

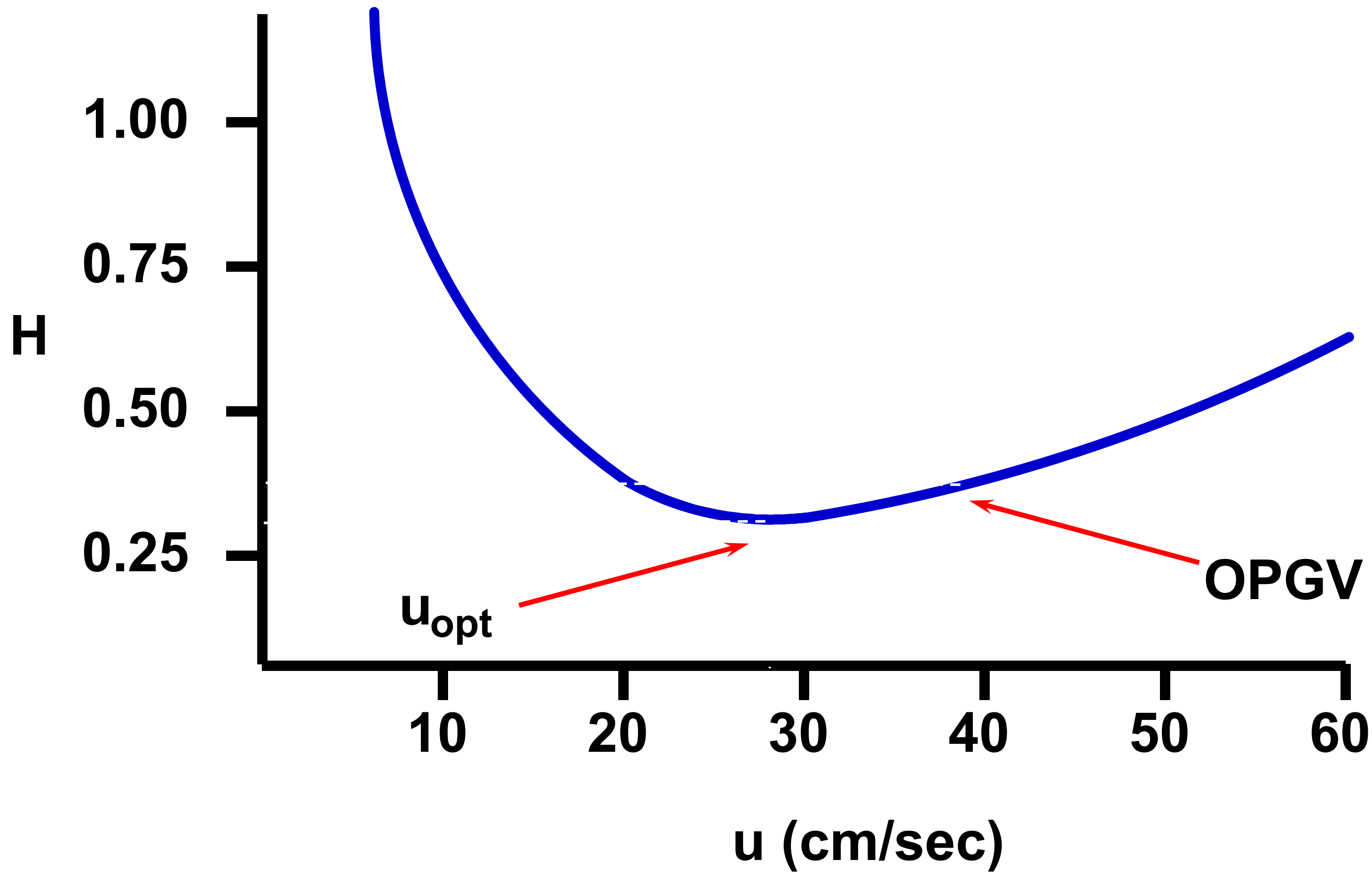
Resolution of 1.5 = baseline resolution



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

VAN DEEMTER CURVE

OPGV = Optimum Practical Gas Velocity



$\bar{\mu}_{opt}$ and OPGV

$\bar{\mu}_{opt}$:

Maximum efficiency

OPGV:

Optimal practical gas velocity

Maximum efficiency per unit time

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

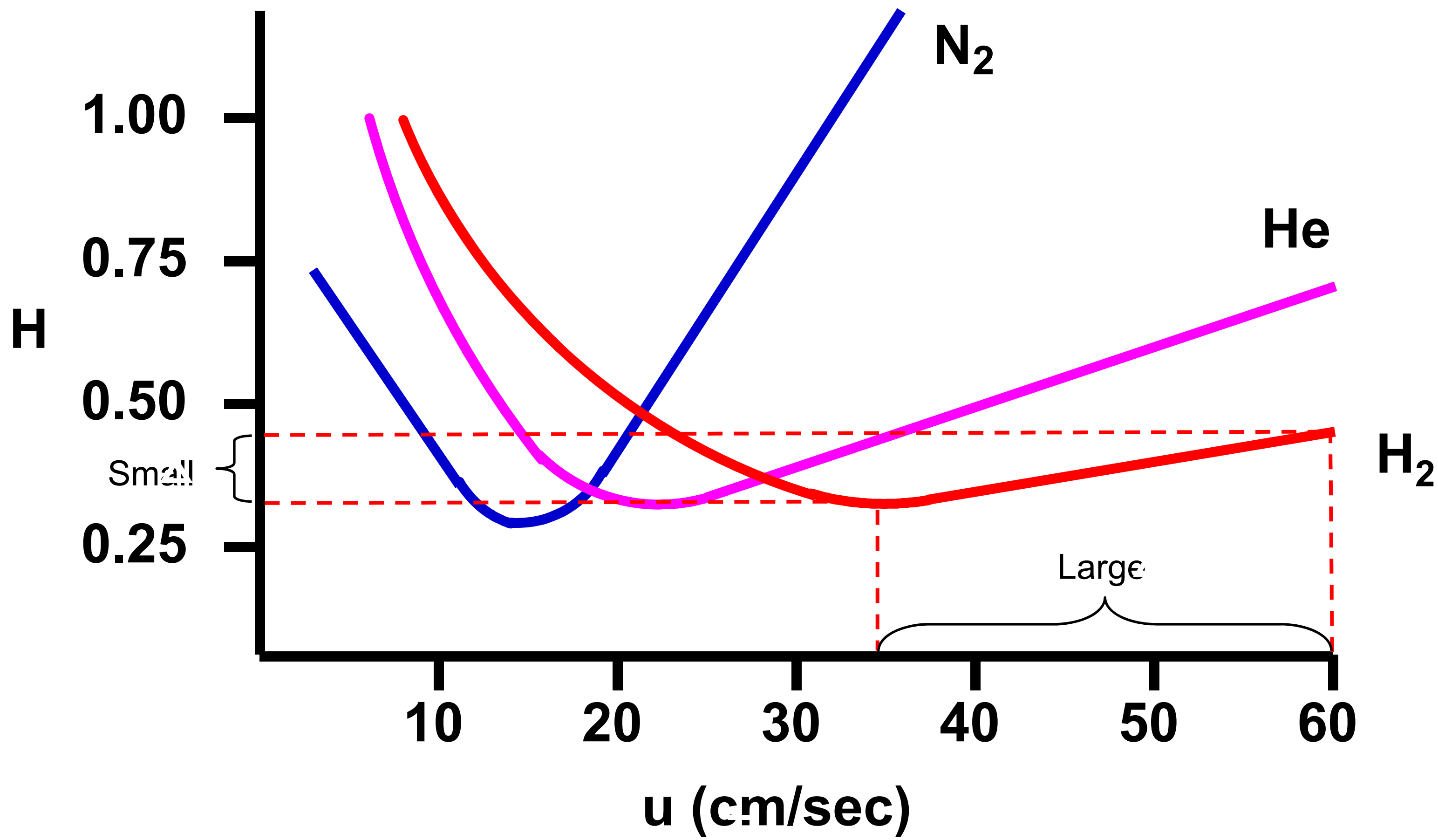
COMMON CARRIER GASES

Nitrogen

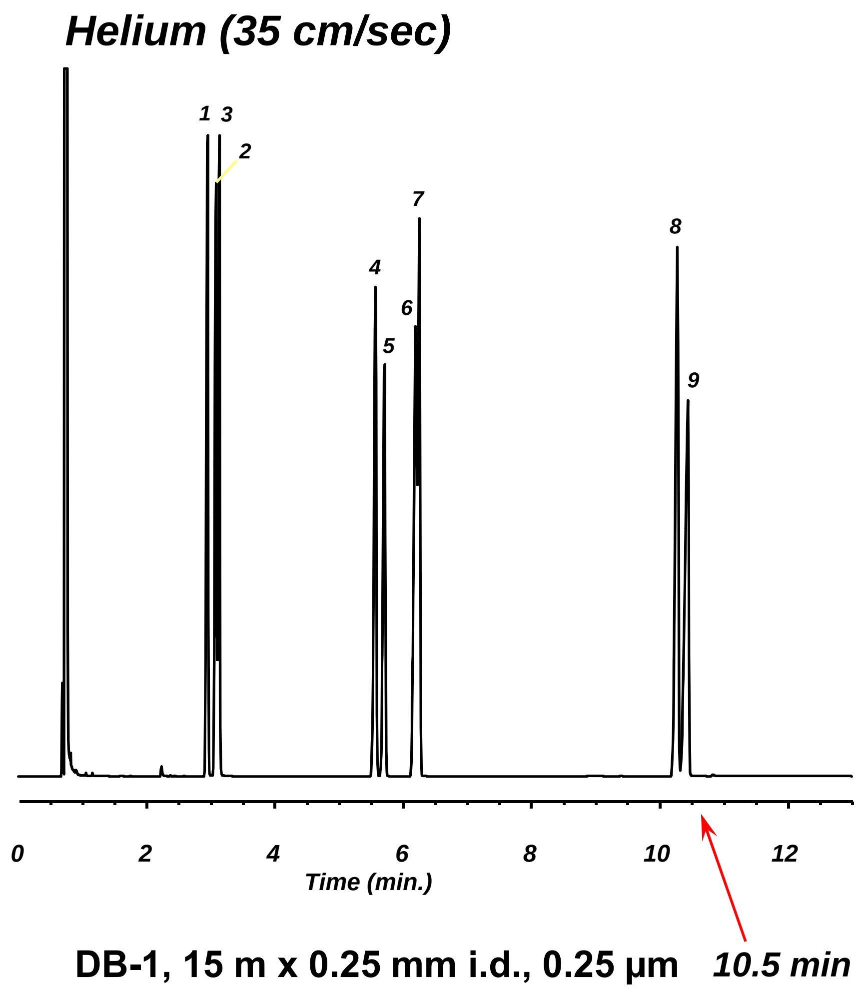
Helium

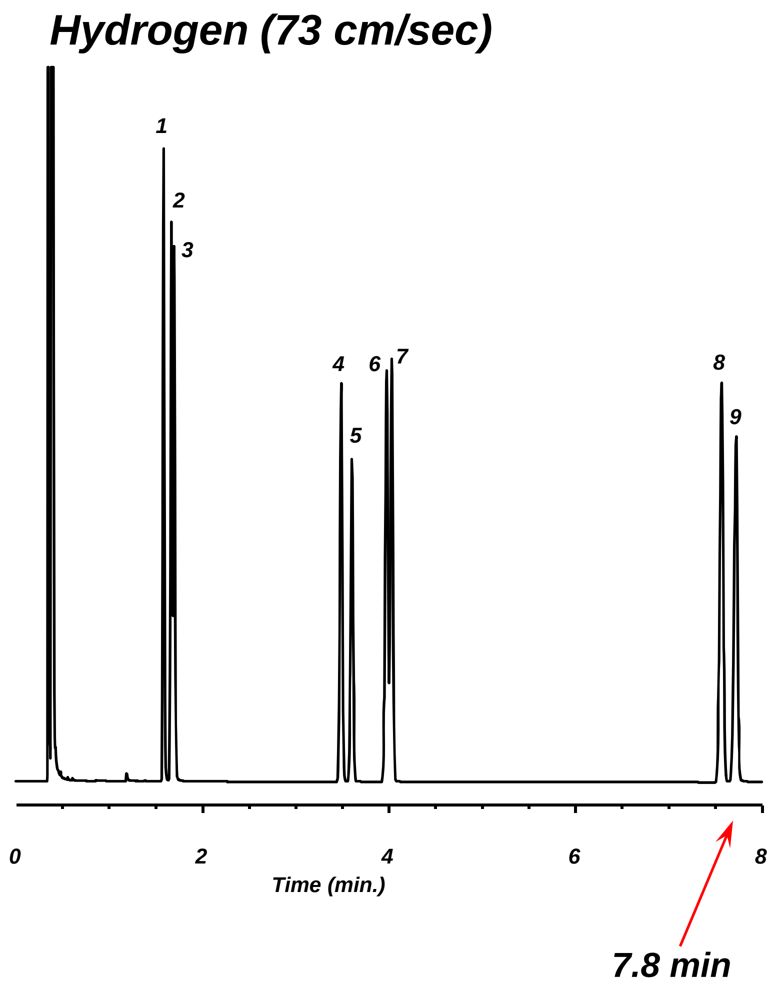
Hydrogen

VAN DEEMTER CURVES



CARRIER GAS
Helium vs. Hydrogen





DB-1, 15 m x 0.25 mm i.d., 0.25 µm
50°C for 2 min, 50-110°C at 20°/min

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CARRIER GAS

Gas	Advantages	Disadvantages
Nitrogen	Cheap, Readily available	Long run times
Helium	Good compromise, Safe	Expensive
Hydrogen	Shorter run times, Cheap	Explosive*

*Hydrogen is difficult to explode under GC conditions

Conclusions

- ✓ Make sure what you are doing make sense
- ✓ Try not to make a big change in diameter, take small steps
- ✓ A good place to start is to switch from 0.53 to 0.45 mm id, or 0.32 to 0.25 mm
- ✓ Remember that with a decrease in diameter you will also have a decrease in capacity and flow
- ✓ Avoid 0.100 mm id columns unless you are proficient in 0.18 mm id
- ✓ Use Method Translator

Thank you!

Agilent Technical support can be reached at:

1-800-227-9770…3…3…1

Questions?