

Secrets of GC Column Dimensions

GC Columns and Consumables

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Application Engineer
May 20, 2008



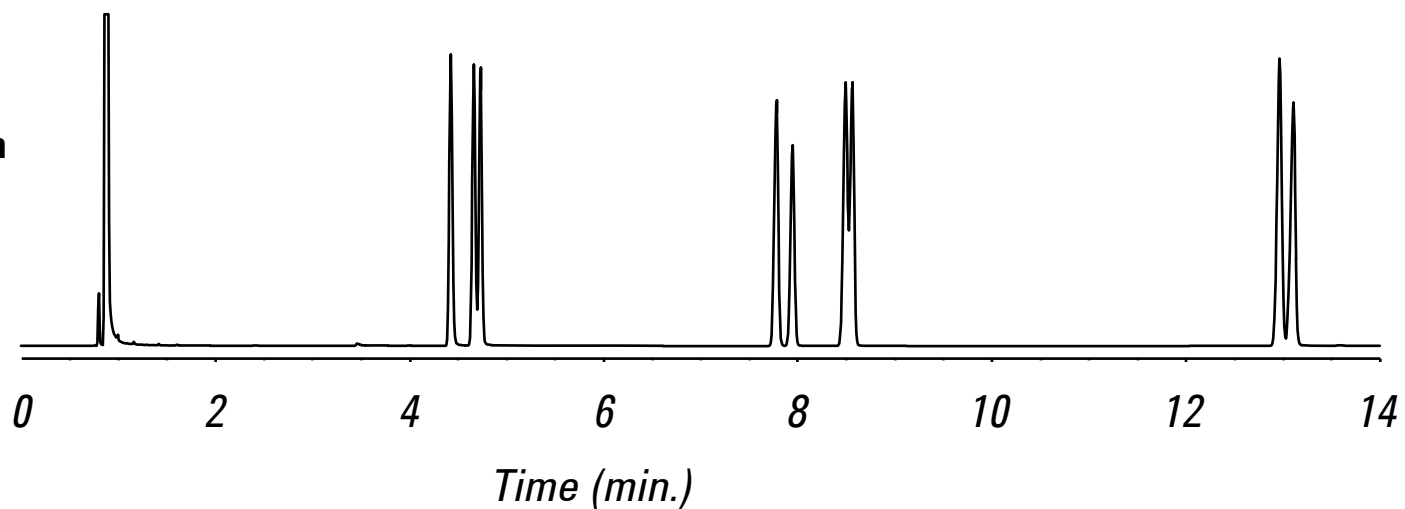
Secrets of GC Column Dimensions

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

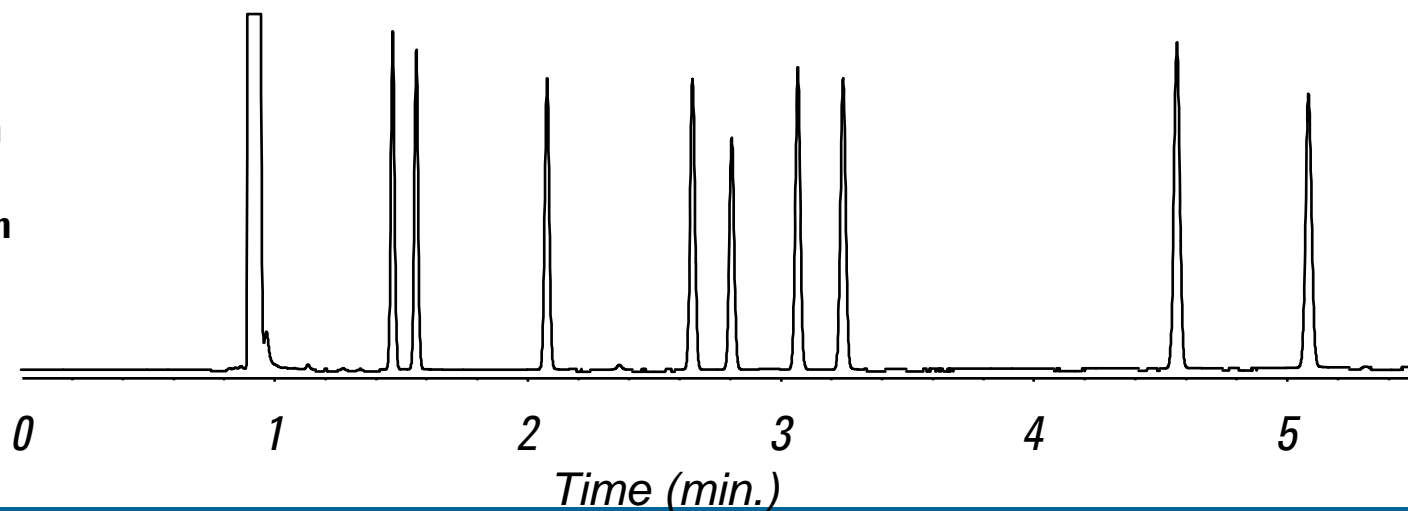


Start with the Right Phase

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

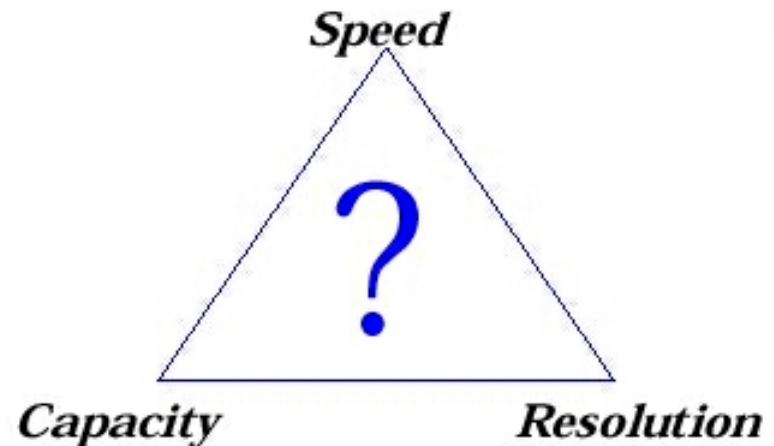


DB-Wax
15m, 0.32mm, 0.25 μ m
Oven:
80-190°C at 20°C/min



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

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

L = Length

Retention

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

r_c = column radius

d_f = film thickness

Selectivity

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

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

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

L = Length

Retention

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

r_c = column radius

d_f = film thickness

Selectivity

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

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

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

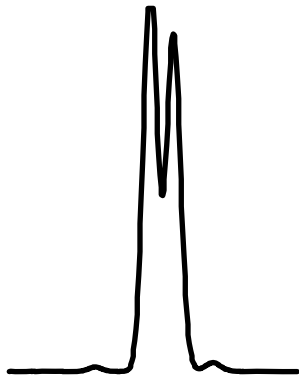
Length X 4 = Resolution X 2

$$t \propto L$$

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

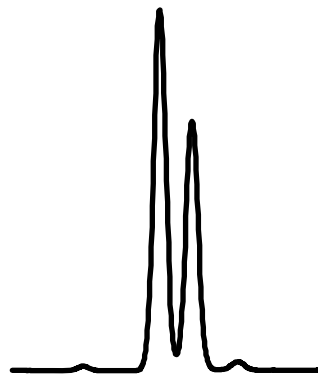
Column Length VS Resolution and Retention: Isothermal

R=0.84
2.29 min



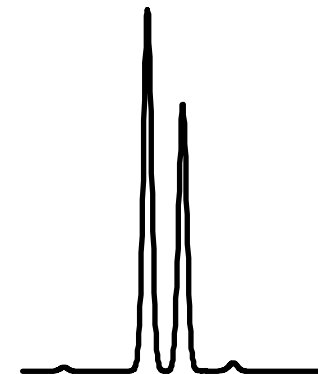
15 m

R=1.16
4.82 min



30 m

R=1.68
8.73 min

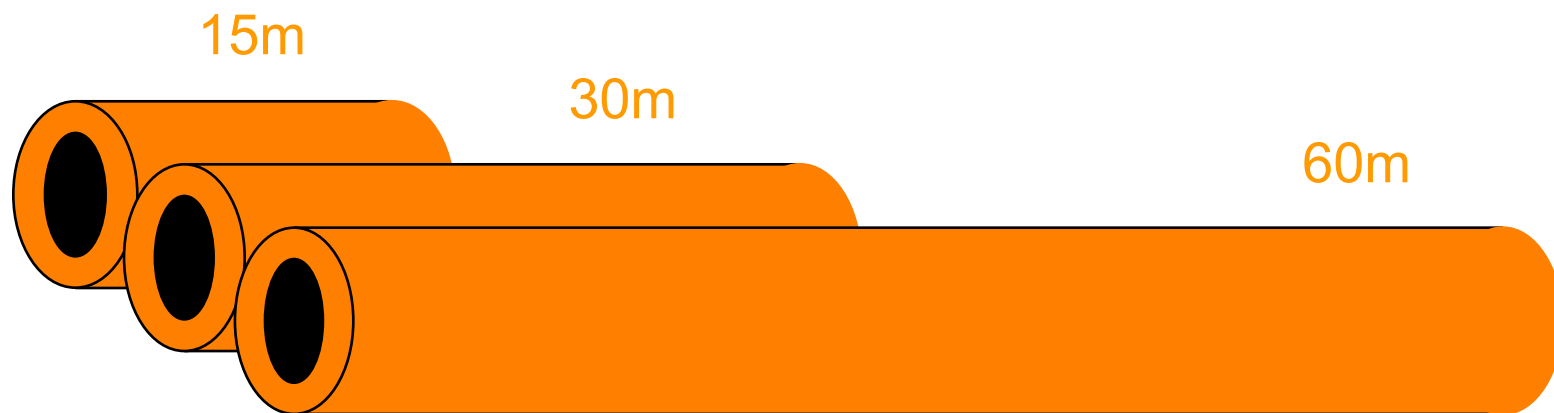


60 m

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

d_f = film thickness

Selectivity

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

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

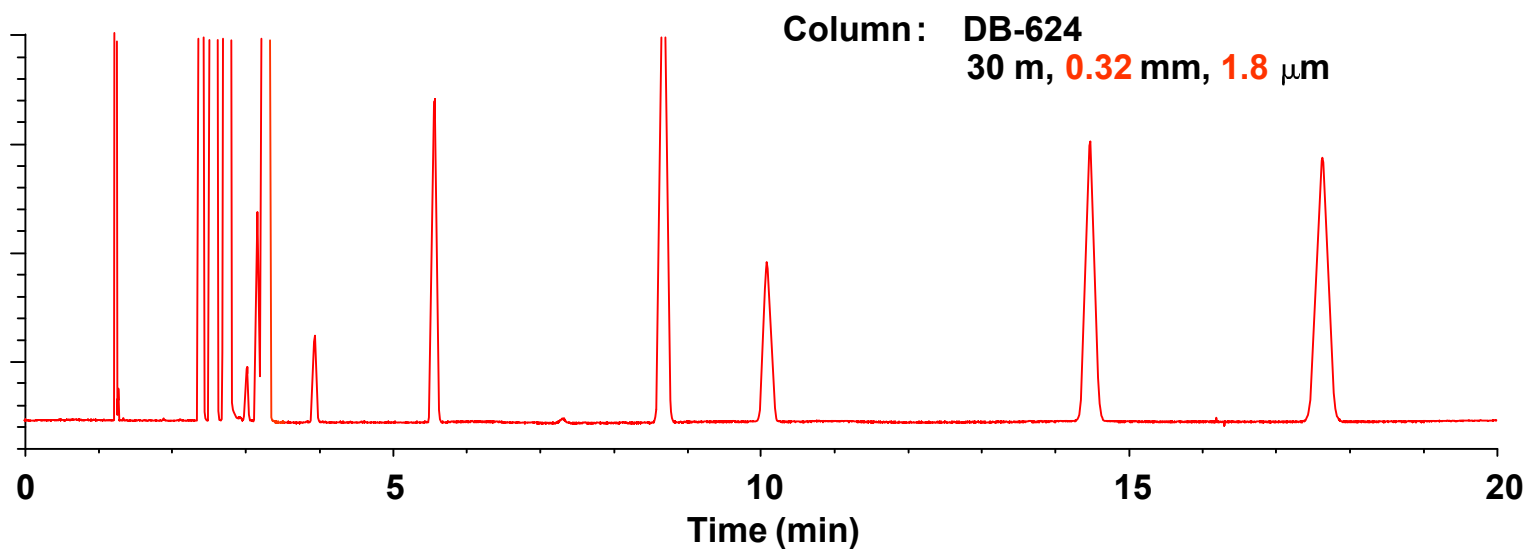
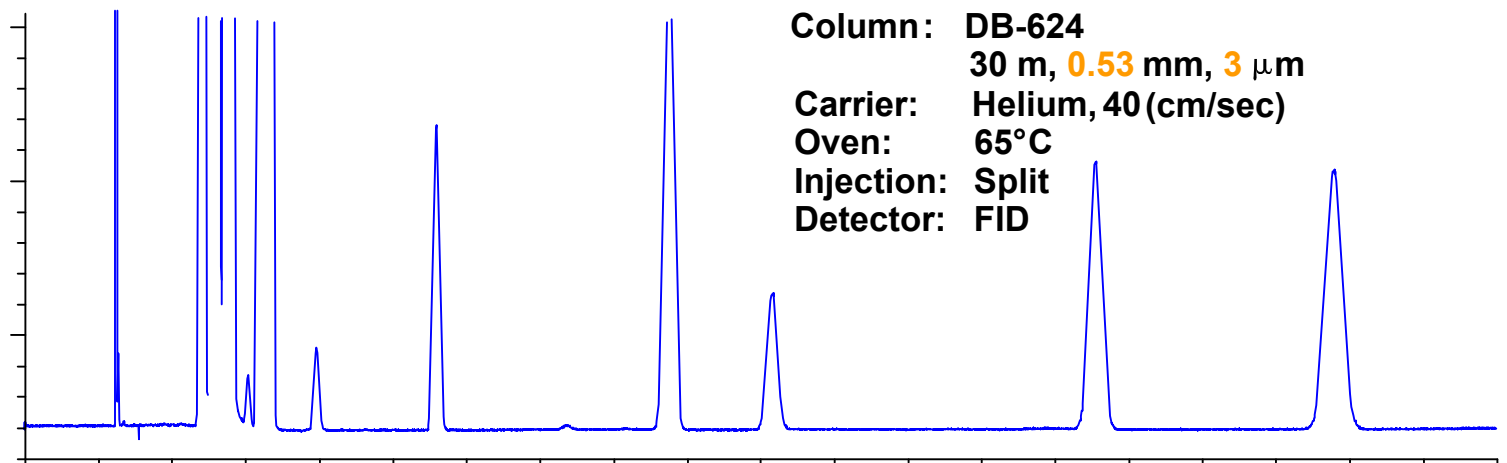
I.D. (mm)	Common Name
0.53	Megabore
0.45	High speed Megabore
0.32	Widebore
0.20-0.25	Narrowbore
0.18	Minibore
0.10	Microbore
0.05	“Nanobore”

Column Diameter - Theoretical Efficiency

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

k = 5

Different Column I. D. Equal Phase Ratios



PHASE RATIO (β)

Film Thickness

Column Dimensions

30 m x .53 mm x 3.0 μm

30 m x .32 mm x 1.8 μm

Phase Ratio β

44

44

$$K_C = k \beta$$

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

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

30 m x .53 mm x 3.0 μm

30 m x .45 mm x 2.55 μm

Phase Ratio β

44

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

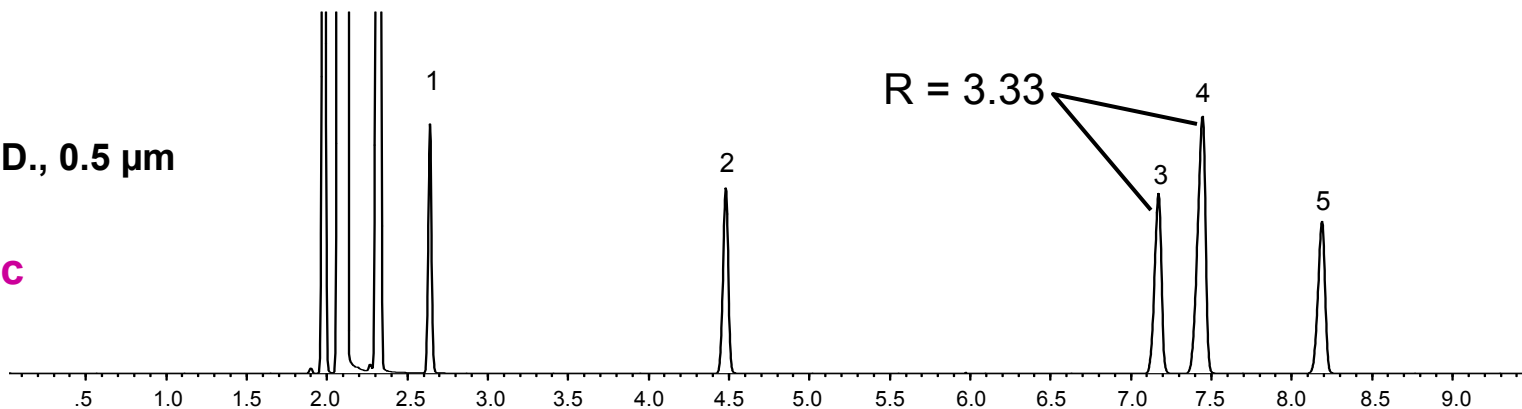


High-SPEED Megabore

Same Resolution - Faster Analysis!

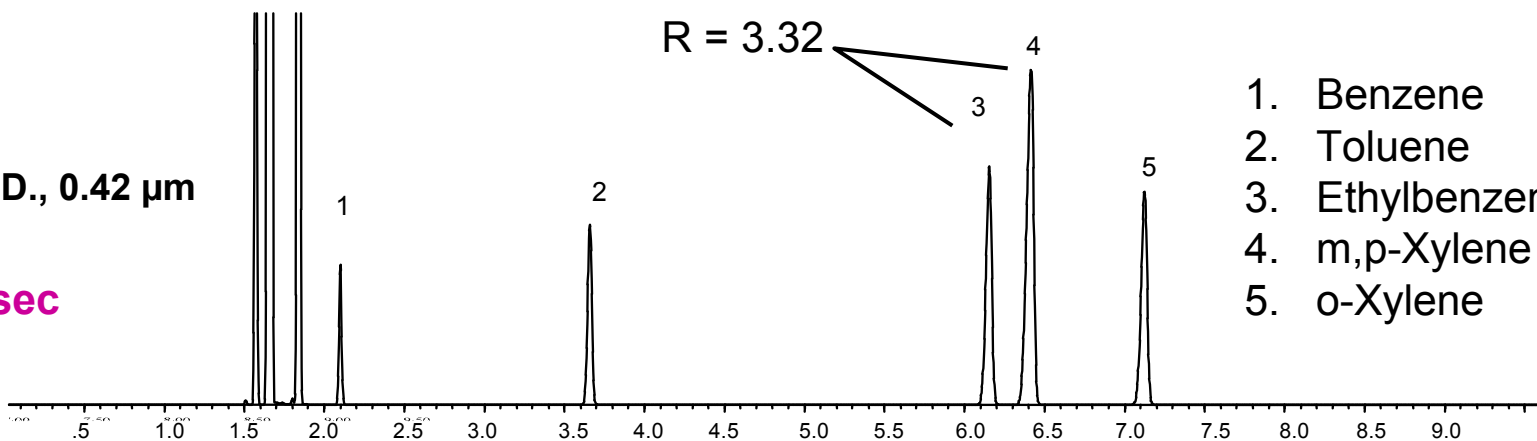
DB-5
30 m,
0.53 mm I.D., 0.5 μ m

36 cm/sec



DB-5
30 m,
0.45 mm I.D., 0.42 μ m

45.9 cm/sec



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

BTEX

Carrier: Helium

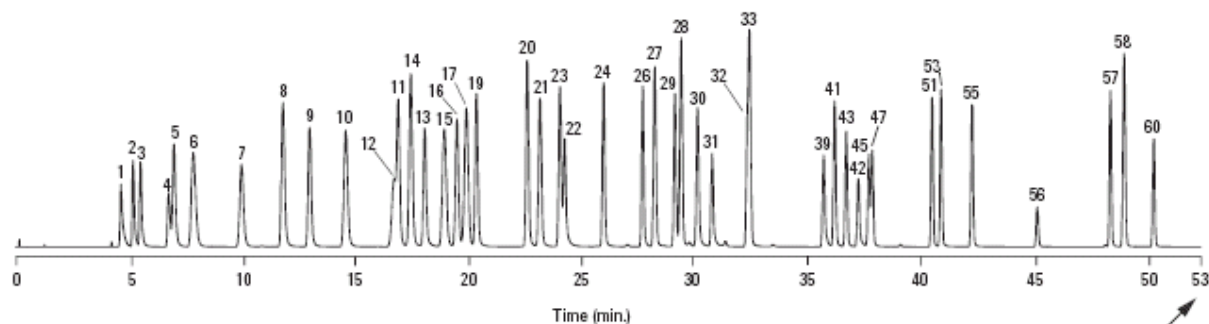
Oven: 40°C for 3 min, 5°/min to 100°C

High SPEED Megabore

Same Resolution - Faster Analysis!

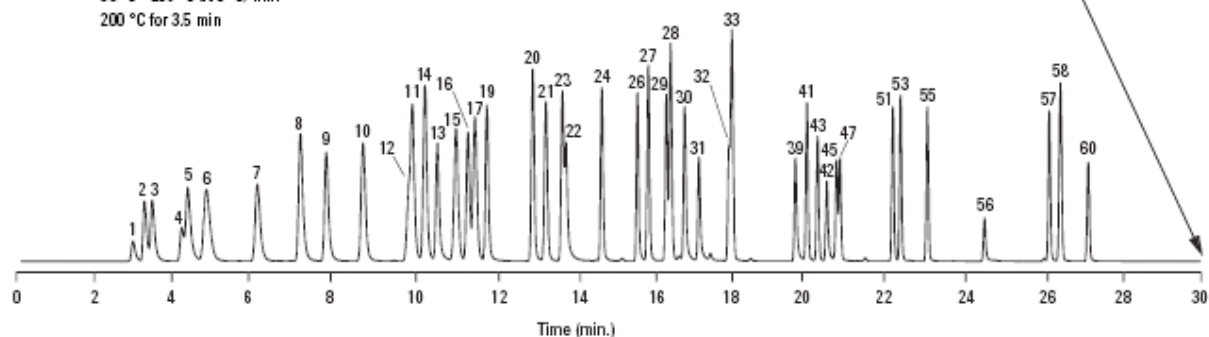
Conditions
Column: DB-502.2, 105 m x 0.53-mm ID, 3.0 µm
Part no.: 125-14A4
Carrier: Helium at 10 mL/min, measured at 35 °C
Oven: 35 °C for 10 min
 35 °C - 200 °C at 4 °C/min
 200 °C for 5 min

Injector: Purge and trap (OIA 4560)
 40 ppb per component in 5 mL water
Trap: Tenax™/Silica gel/Charcoal
Detector: Electrolytic conductivity detector (ELCD)
 (OIA 4420) with NiCat™
 reaction tube in the halogen mode



Conditions
Column: DB-502.2, 75 m x 0.45-mm ID, 2.55 µm
Part no.: 124-1474
Carrier: Helium at 9 mL/min, measured at 35 °C
Oven: 35 °C for 6 min
 35 °C - 200 °C at 8 °C/min
 200 °C for 3.5 min

Injector: Purge and trap (OIA 4560)
 40 ppb per component in 5 mL water
Trap: Tenax™/Silica gel/Charcoal
Detector: ELCD (OIA 4420) with NiCat
 reaction tube in the halogen mode



High-Speed Megabore
 saves 23 minutes!

“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
<hr/>	
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	$N = f$ (gas, L, r_c)	L = Length
Retention	$k = f$ (T, d_f , r_c)	r_c = column radius d_f = film thickness
Selectivity	$\alpha = f$ (T, phase)	T = temperature

Film Thickness and Retention: Isothermal

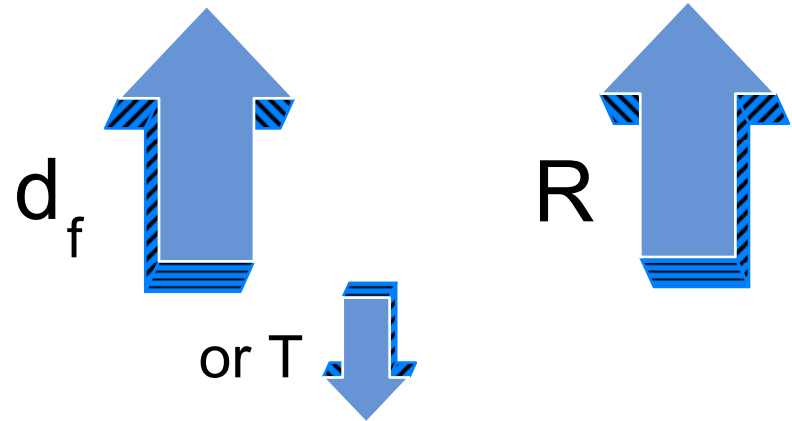
Thickness (μm) Retention Change

0.10	0.40
0.25	1.00
1.0	4.00
3.0	12.0
5.0	20.0

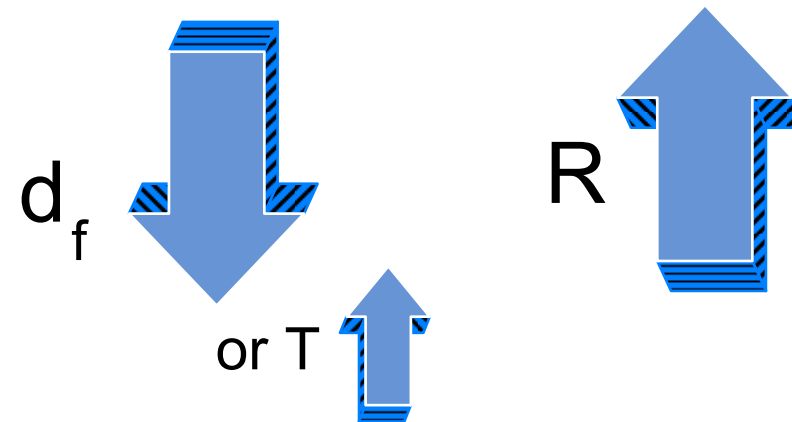
Constant Diameter
Normalized to 0.25 μm

Film Thickness and Resolution

When solute $k < 5$
(early eluters)



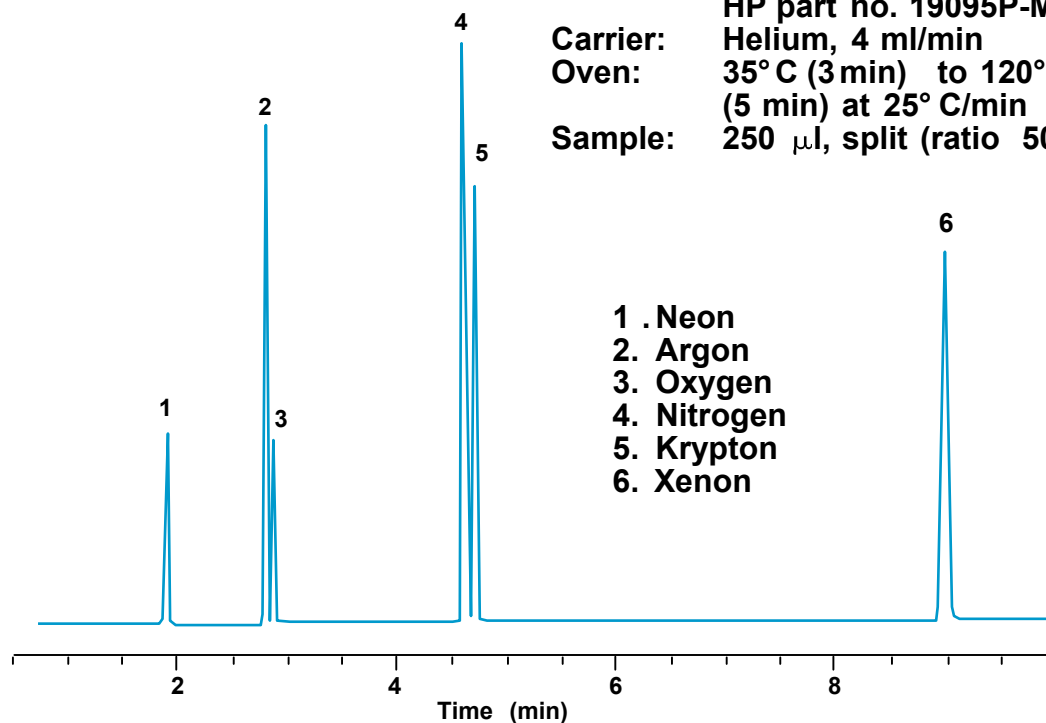
When solute $k > 5$
(later eluters)



Other Retention - Adsorption

Analysis of Noble & Fixed Gases Using HP PLOT MoleSieve

Column: HP-PLOT/MoleSieve
30 m x 0.53 mm x 50 μ m
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 μ l, split (ratio 50:1)



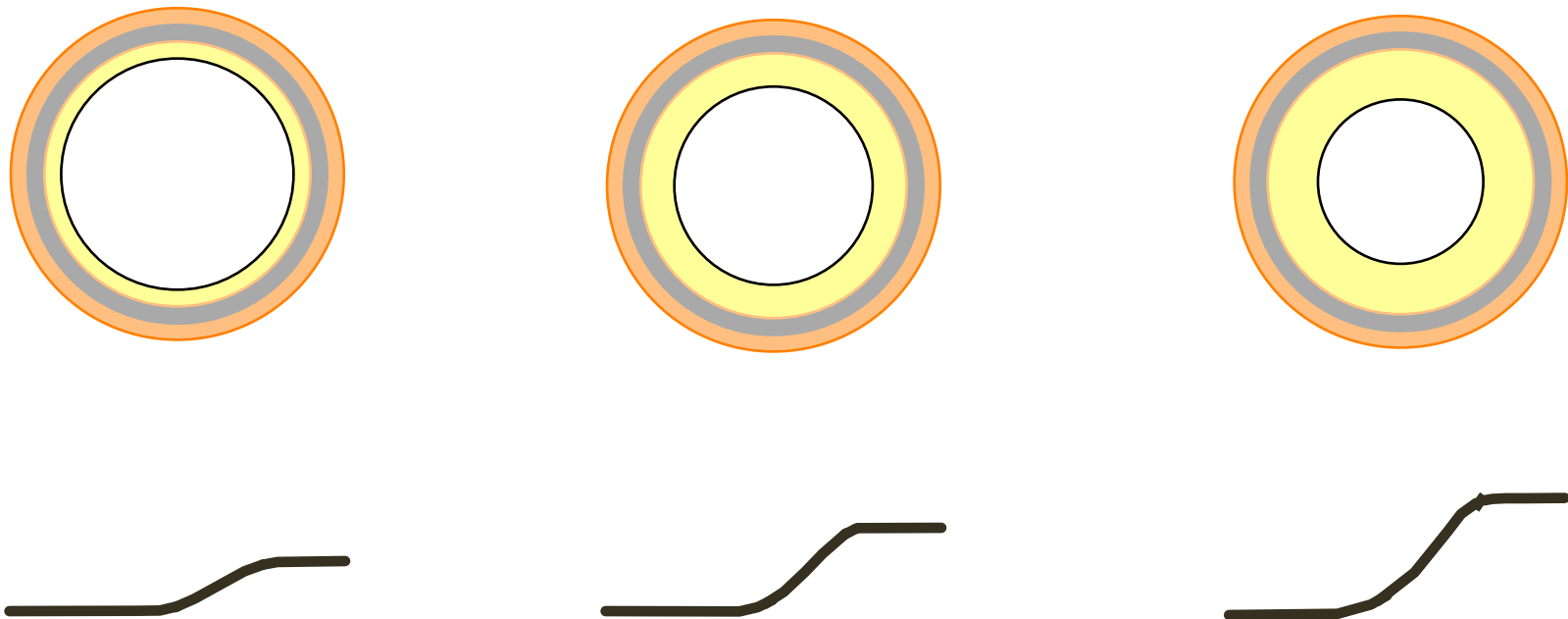
Film Thickness and Capacity

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

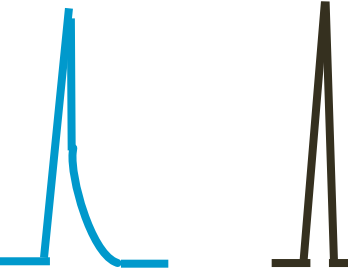
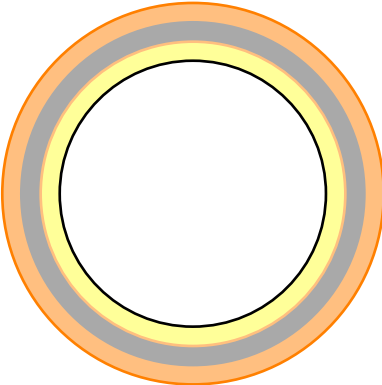
Film Thickness and Bleed

More stationary phase = More degradation products



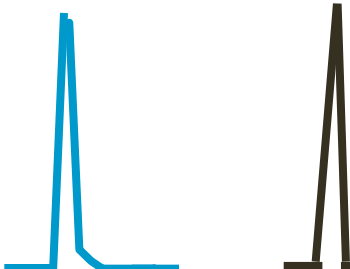
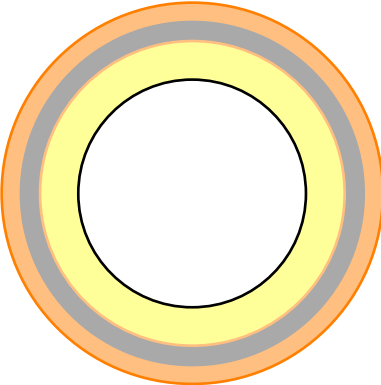
Film Thickness and Inertness

0.25



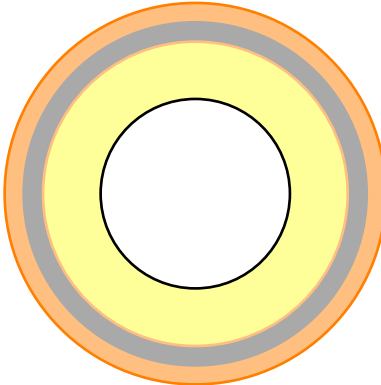
active inactive

1.0



active inactive

3.0



active inactive

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

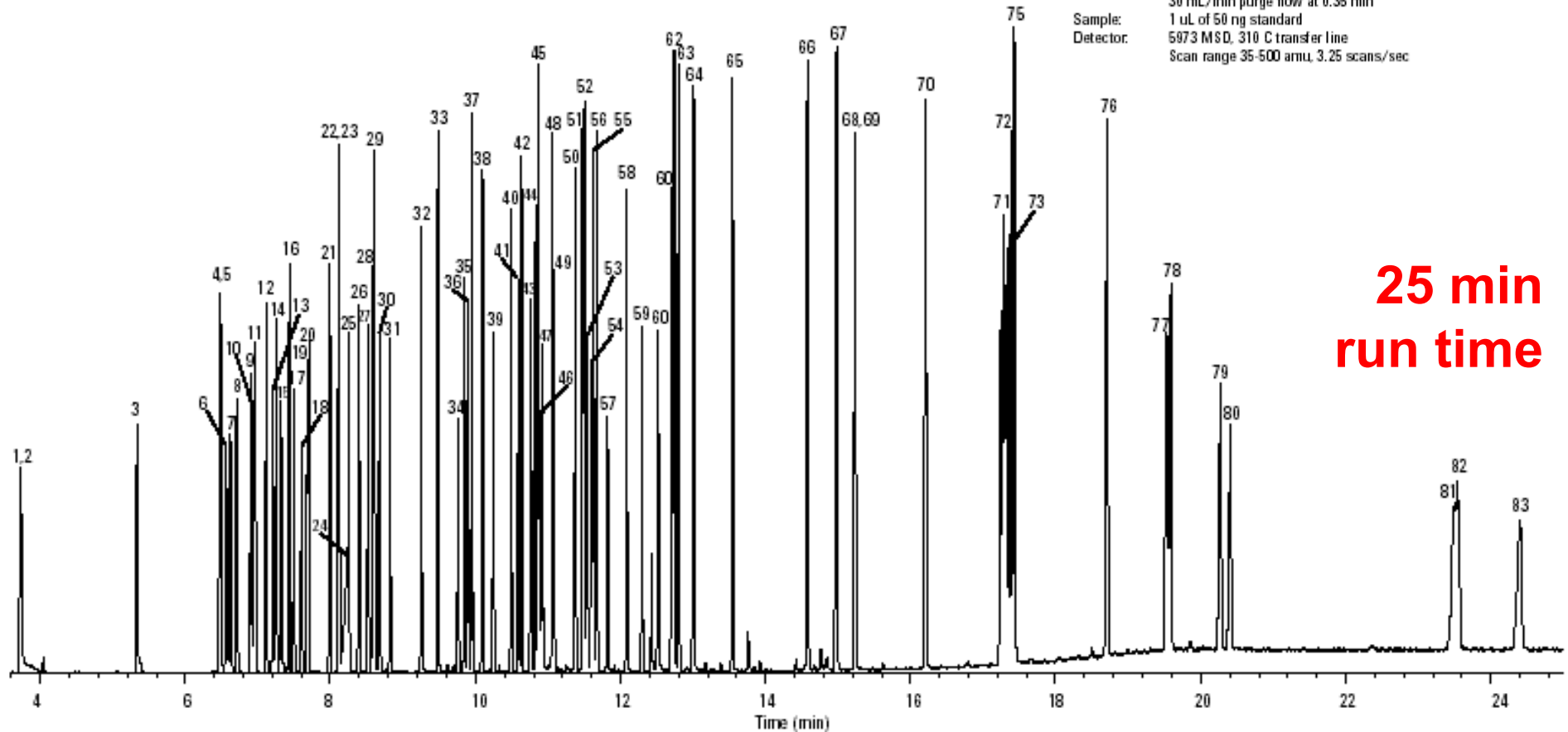
Examples.....

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 µm
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 µL of 50 ng standard
Detector: 5973 MSD, 310 C transfer line
Scan range 35-500 amu, 3.25 scans/sec



**25 min
run time**

C1080

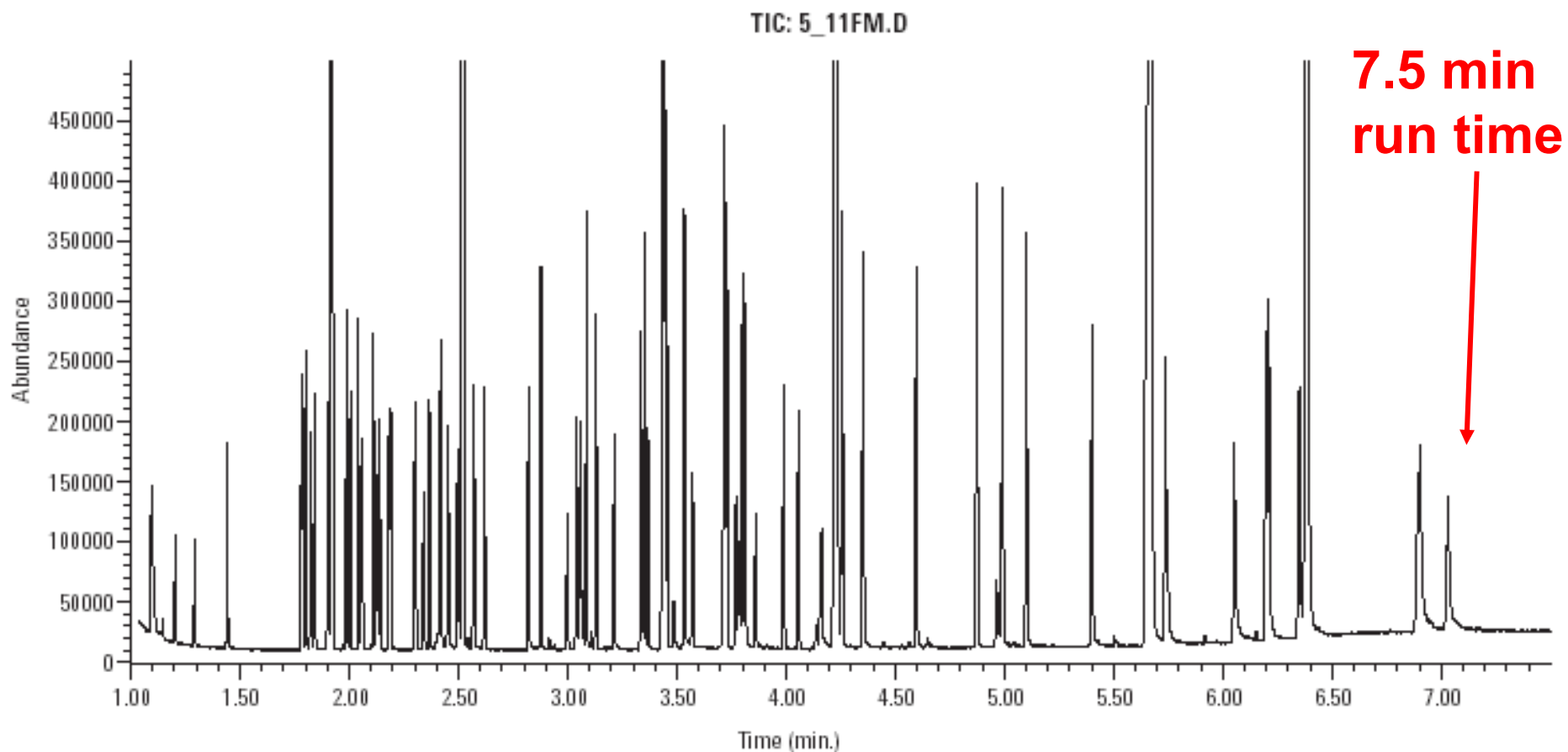


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Fast 8270 Semivolatile Analysis

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?

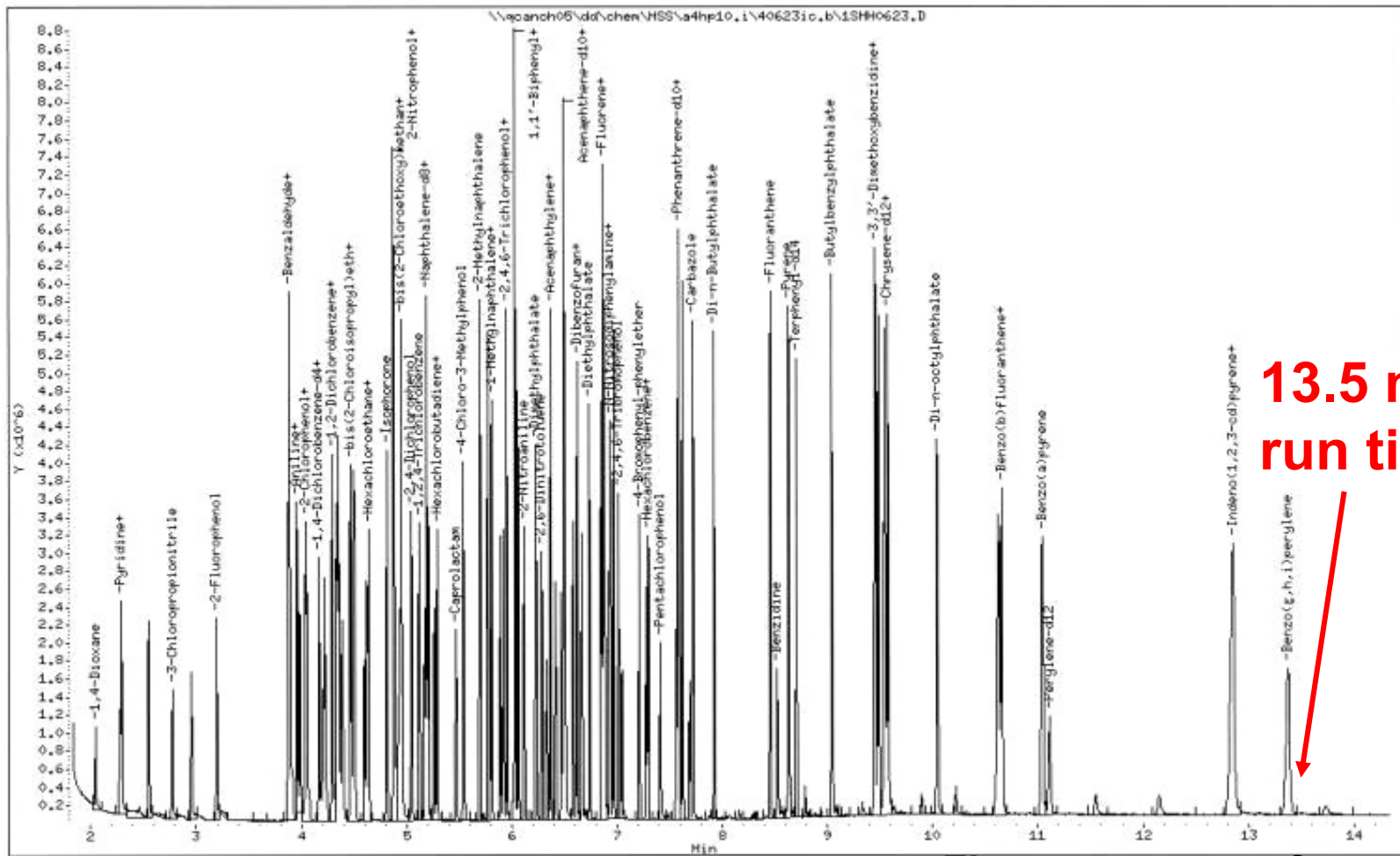
Environmental type samples = high contaminant residue potential. Smaller ID columns have reduced capacity for matrix contaminants due to lower surface area, shorter length (less forgiving).

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?**



What this method optimization means to your lab

**Before
Optimization**



19 Spls/day



19 Spls/day



19 Spls/day

**After
Optimization**



29 Spls/day



28 Spls/day

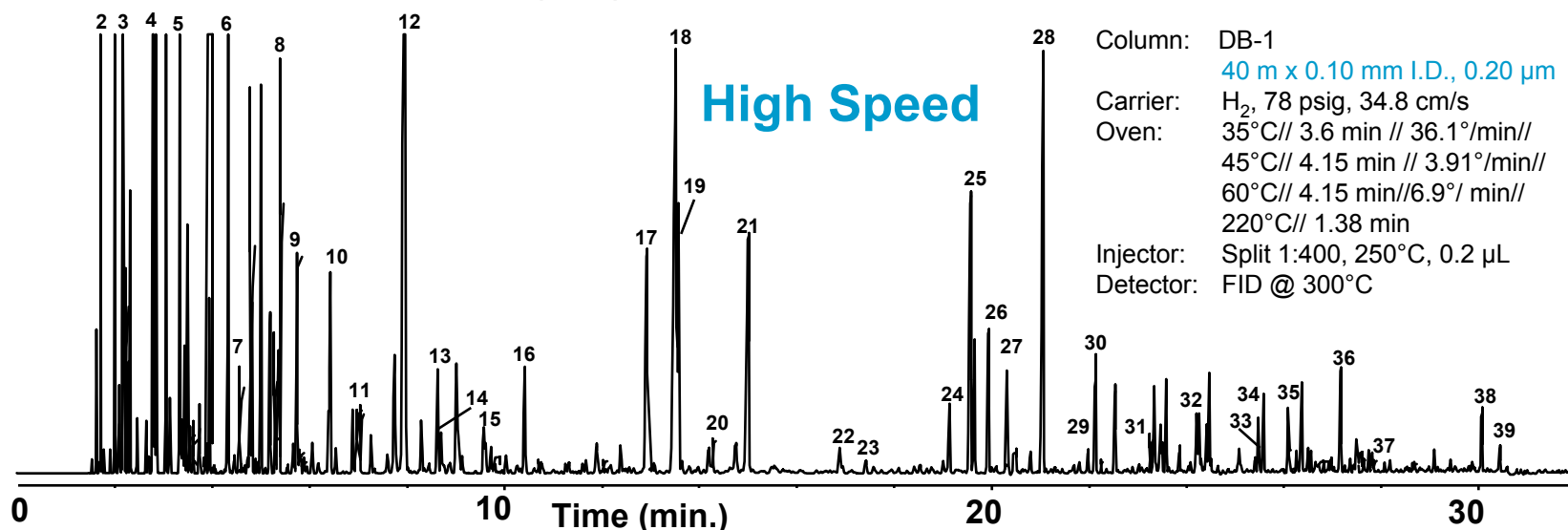
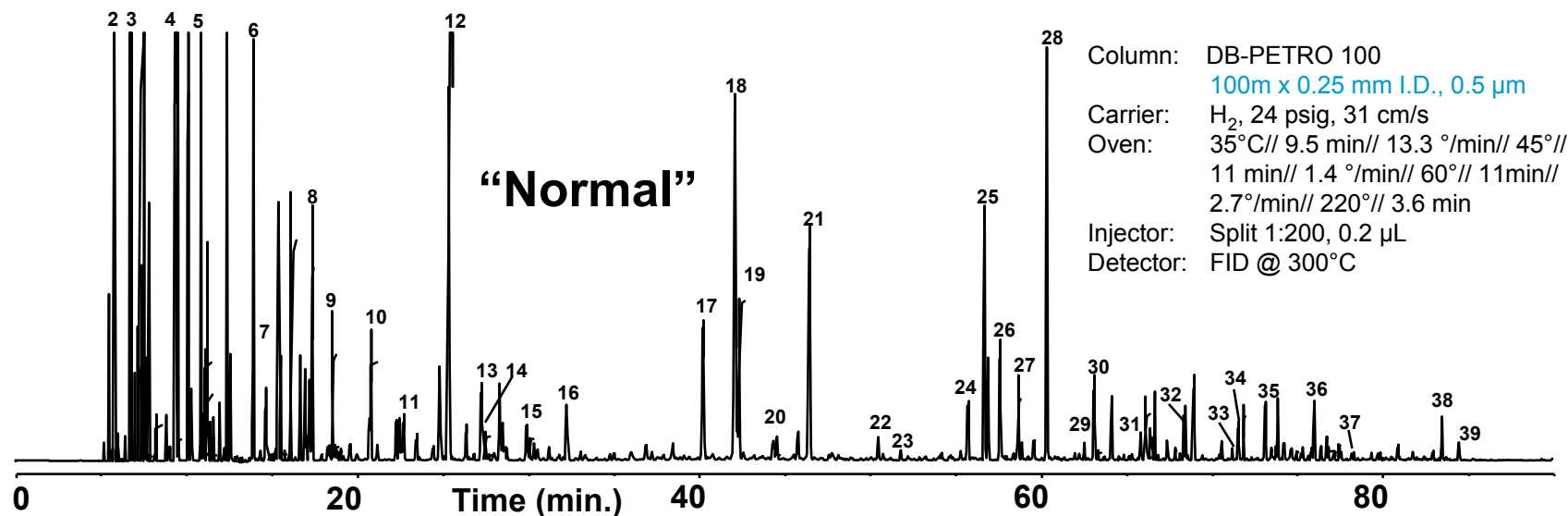


Idle



Regular Unleaded Gasoline

California Phase I



OK, Test Time

Fusel Oil Simple Standard

DB-624

25 m x .53 mm I.D. x 3.0 μ m

Inlet: 250°C, split

FID: 300°C

Carrier: H₂, 50 cm/sec

Oven: 40°C for 5 min.

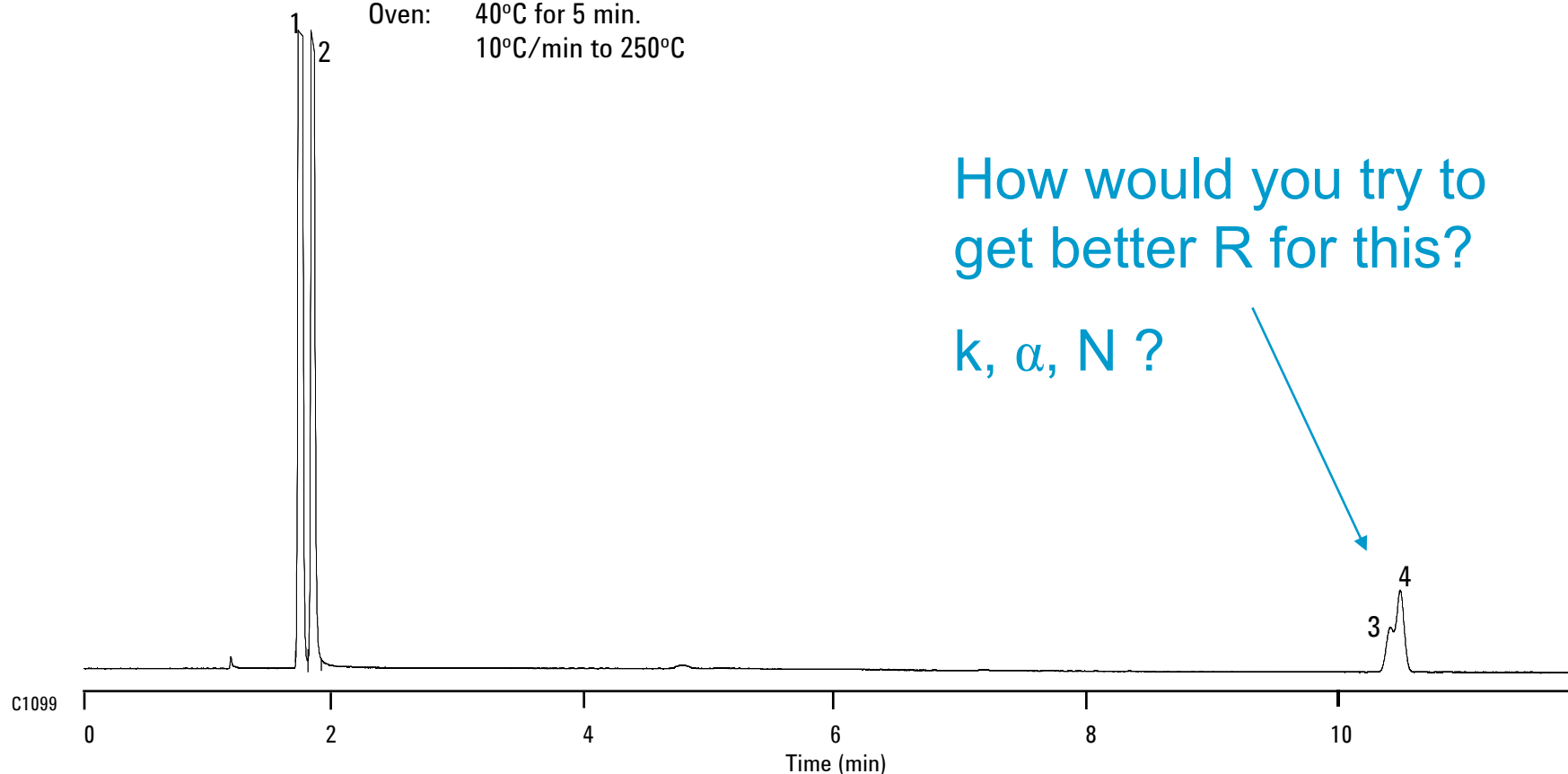
10°C/min to 250°C

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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Need Plates?...

Length AND Column Diameter

Column Dimensions

25 m x .53 mm x 3.0 μm

60 m x .25 mm x 1.4 μm

Theoretical Plates

34,530

181,860



Fusel Oil Standard

DB-624

60 m x .25 mm I.D. x 1.4 μ m

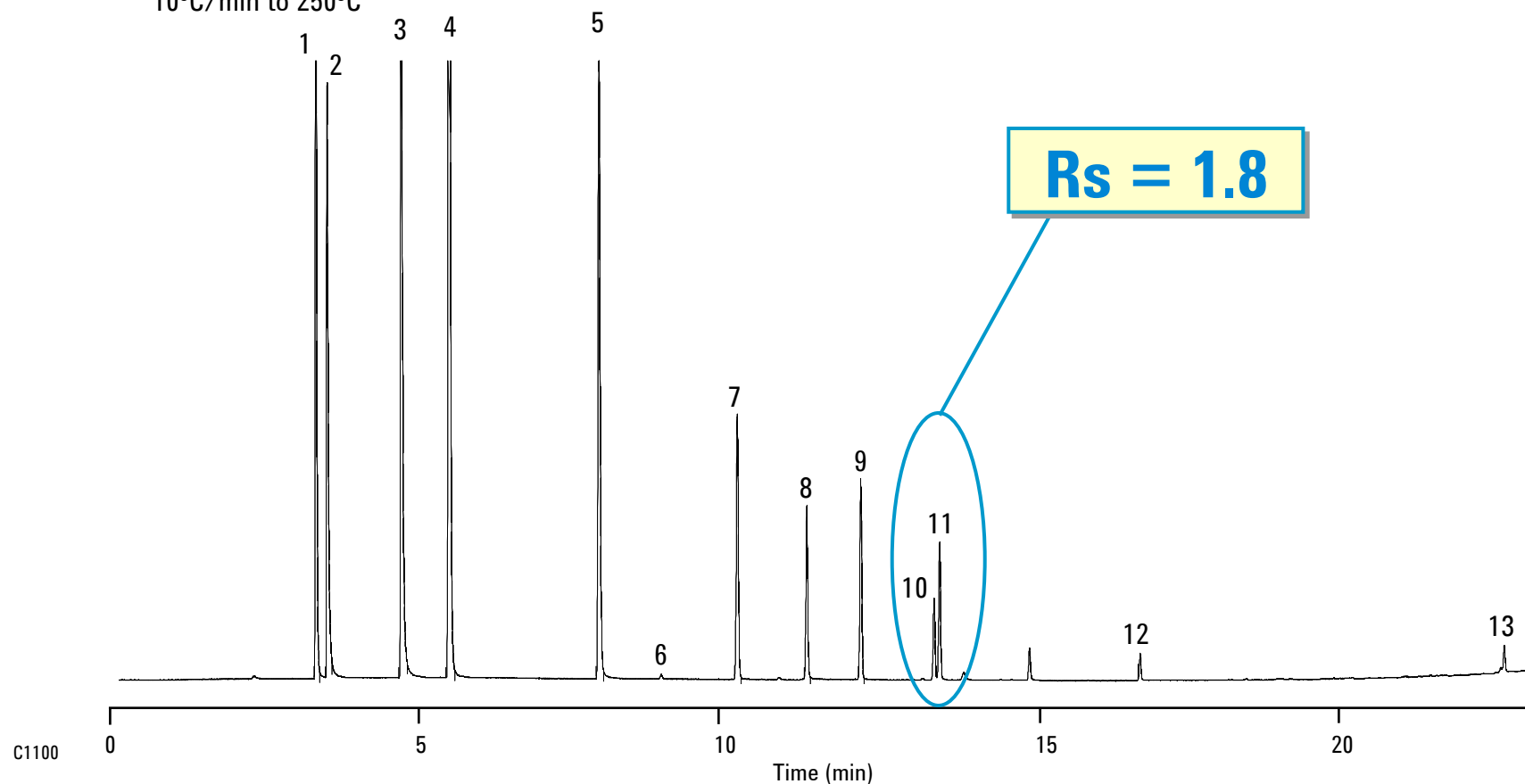
Inlet: 250°C, split

FID: 300°C

Carrier: H₂, 50 cm/sec

Oven: 40°C for 5 min.
10°C/min to 250°C

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



Method Translation Software Input Screen

GC Method Translation

Criterion: Translate Only Best Efficiency Fast Analysis None **Speed gain: 1.43108**

	Original Method	Translated Method																														
Column																																
Length, m	30	<input type="checkbox"/> 30																														
Internal Diameter, μm	250.0	<input type="checkbox"/> 250.0																														
Film		<input checked="" type="radio"/> Unlock																														
Thickness, μm	0.25	<input type="radio"/> 0.250																														
Phase Ratio	250.0	<input type="radio"/> 250.0																														
Carrier Gas	Helium	<input type="checkbox"/> Hydrogen																														
Enter one Setpoint																																
Head Pressure, psi	13.207	8.388																														
Flow Rate, mLn/min	1.2	1.5000																														
Outlet Velocity, cm/sec	42.28	52.85																														
Average Velocity, cm/sec	28.27	40.45																														
Hold-up Time, min	1.76893	1.23608																														
Outlet Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																														
Ambient Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																														
Oven Temperature 3-ramp Program																																
	<table border="1"> <thead> <tr> <th>Ramp Rate</th> <th>Final Temp.</th> <th>Final Time</th> </tr> <tr> <th>$^{\circ}\text{C}/\text{min}$</th> <th>$^{\circ}\text{C}$</th> <th>min</th> </tr> </thead> <tbody> <tr> <td></td> <td>35</td> <td></td> </tr> <tr> <td></td> <td></td> <td></td> </tr> <tr> <td></td> <td></td> <td></td> </tr> </tbody> </table>	Ramp Rate	Final Temp.	Final Time	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min		35								<table border="1"> <thead> <tr> <th>Ramp Rate</th> <th>Final Temp.</th> <th>Final Time</th> </tr> <tr> <th>$^{\circ}\text{C}/\text{min}$</th> <th>$^{\circ}\text{C}$</th> <th>min</th> </tr> </thead> <tbody> <tr> <td></td> <td>35</td> <td></td> </tr> <tr> <td></td> <td></td> <td></td> </tr> <tr> <td></td> <td></td> <td></td> </tr> </tbody> </table>	Ramp Rate	Final Temp.	Final Time	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min		35							
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Available at www.chem.agilent.com/en-us/Support/Downloads/Pages/default.aspx
 or use the key words "GC Method Translator" in the quick search box at the
 Agilent.com website.

CARRIER GAS

Carries the solutes down the column

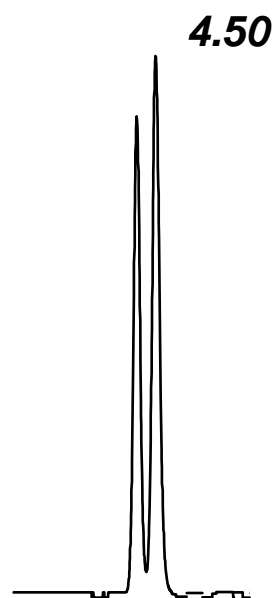
**Selection and velocity influences efficiency
and retention time**



RESOLUTION VS. LINEAR VELOCITY

Helium

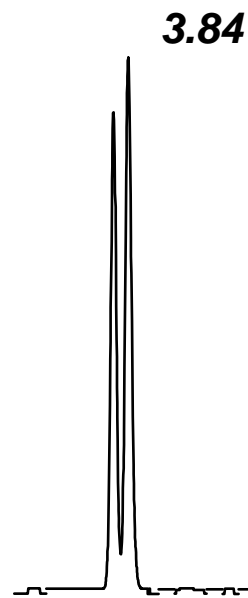
Resolution of 1.5 = baseline resolution



$R = 1.46$

30 cm/sec

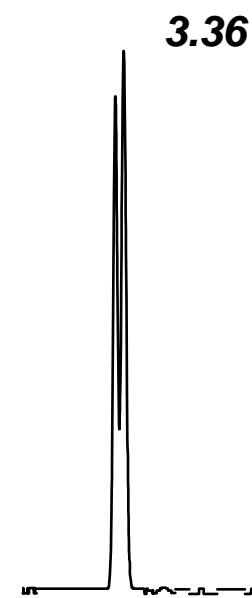
4.4 psig



$R = 1.31$

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

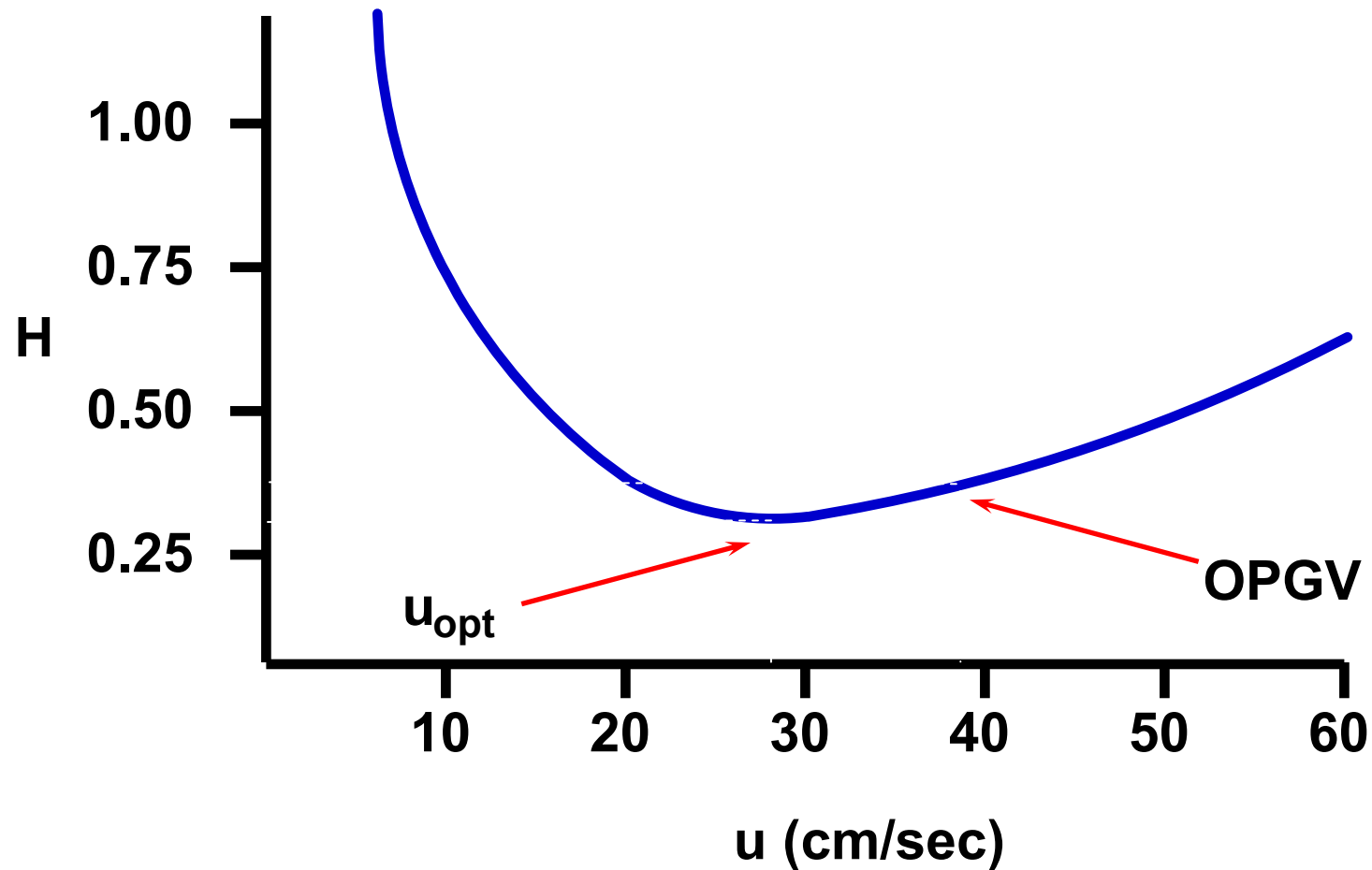


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VAN DEEMTER CURVE

OPGV = Optimum Practical Gas Velocity



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

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

Maximum efficiency

OPGV:

Optimal practical gas velocity

Maximum efficiency per unit time

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



COMMON CARRIER GASES

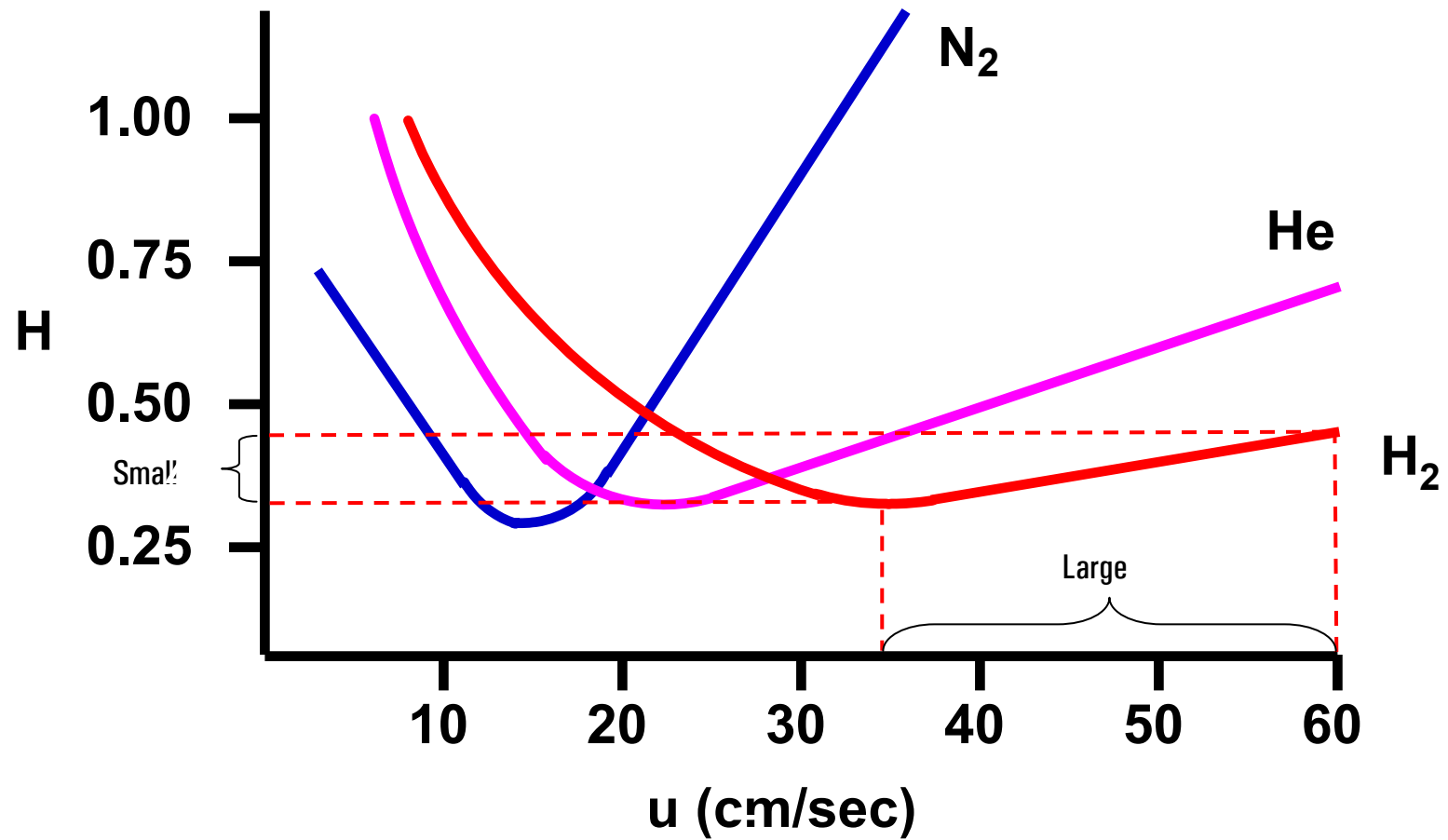
Nitrogen

Helium

Hydrogen



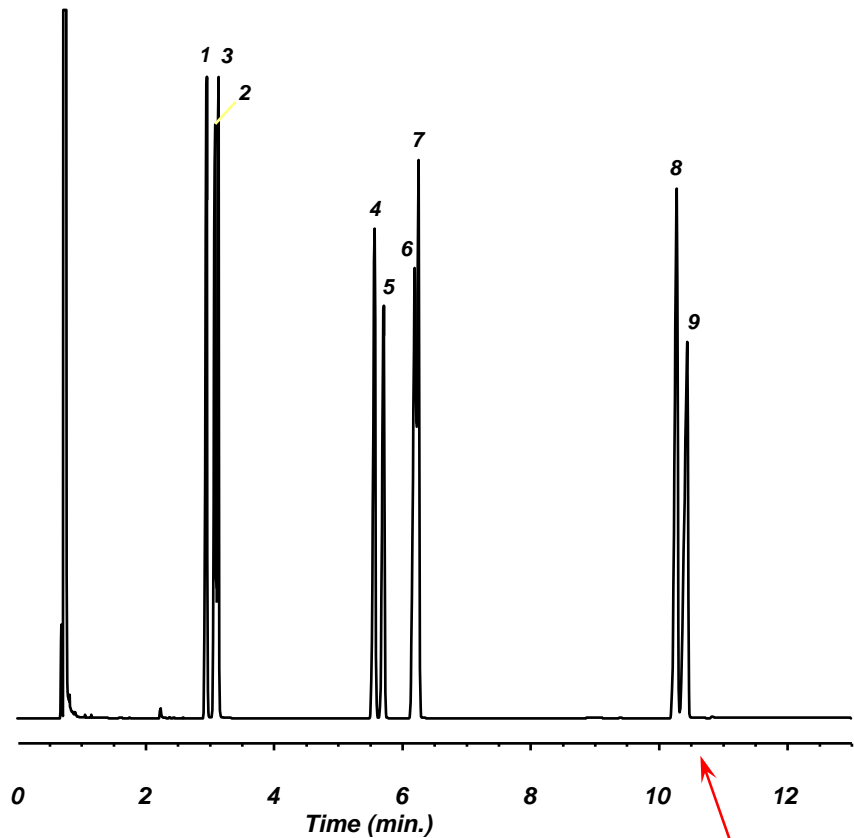
VAN DEEMTER CURVES



CARRIER GAS

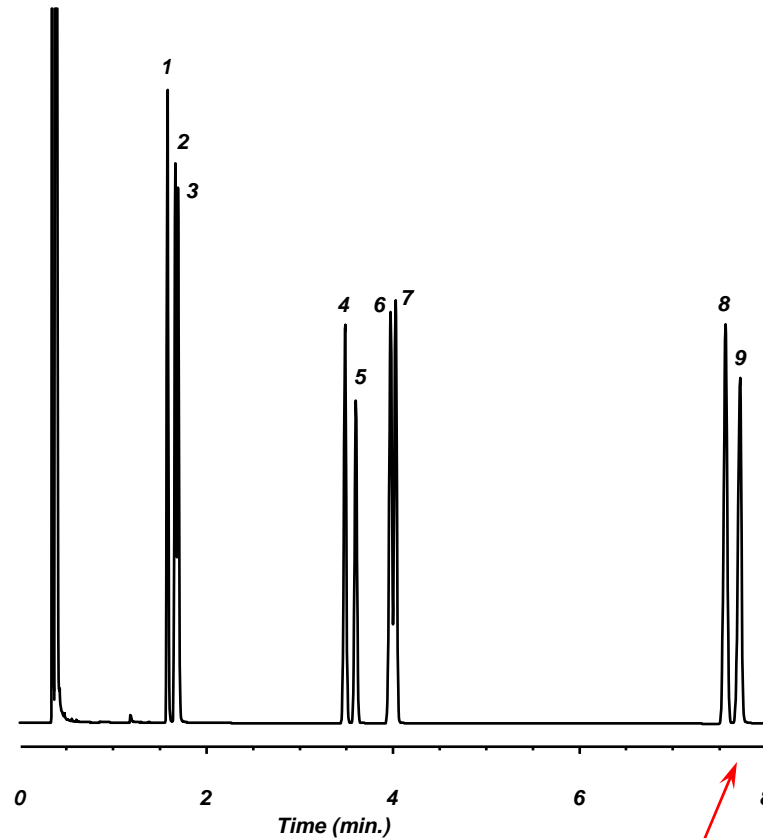
Helium vs. Hydrogen

Helium (35 cm/sec)



DB-1, 15 m x 0.25 mm i.d., 0.25 μ m 10.5 min
50°C for 2 min, 50-110°C at 20°/min

Hydrogen (73 cm/sec)



7.8 min



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
- ✓ Utilize Technical Support!

Thank you!

Agilent technical support can be reached at:

1-800-227-9770....4....1

Questions?



Wrap-up E-Seminar Questions

**Thank you for attending Agilent e-Seminars.
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Repeatable and Robust

June 20, 2008 – 1:00 p.m. EDT

Techniques, Tips and Tricks of Troubleshooting
Capillary GC Systems

July 15, 2008 – 2:00 p.m. EDT

