
FAST GC
SPEEDY GC

Optimization Goals

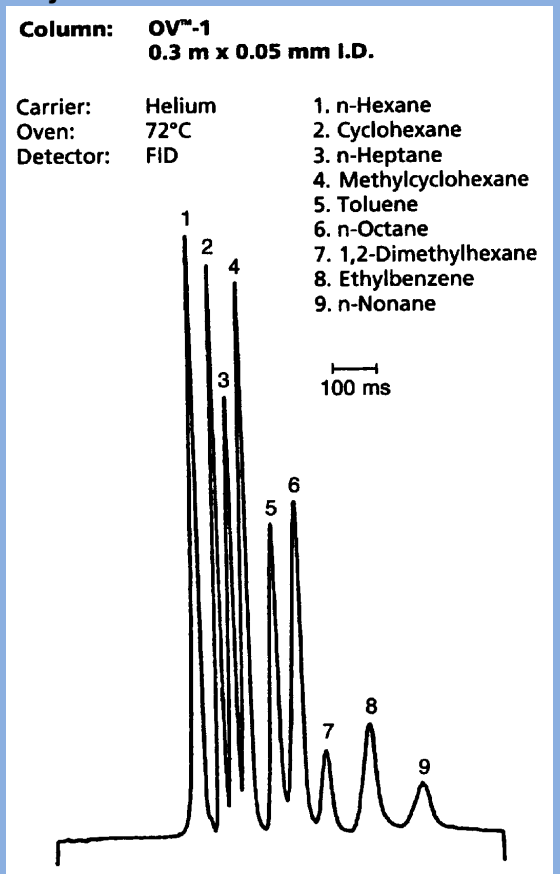
- Primary: Minimize time for the separation of a given number of peaks

- Secondary Maximize the number of peaks separated by a given column

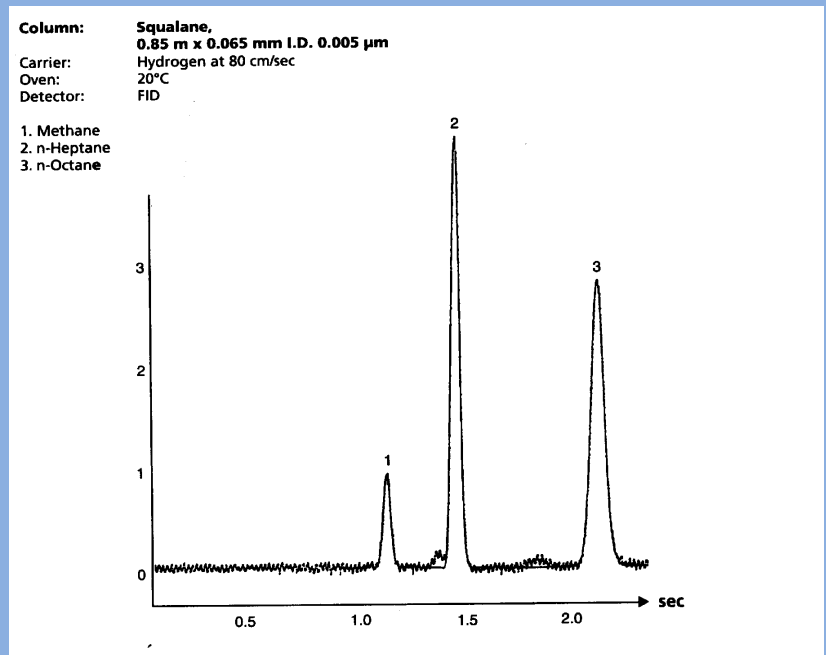
HOW CAN THESE GOALS BE ACHIEVED?

Extreme Systems

Ultra-fast chromatogram (< 1 sec) using cold trap injection device.



Ultra-fast chromatogram (2 sec) obtained by using a fluidic logic gate injector



Complex Inlet systems
Extremely short columns
Small internal diameter

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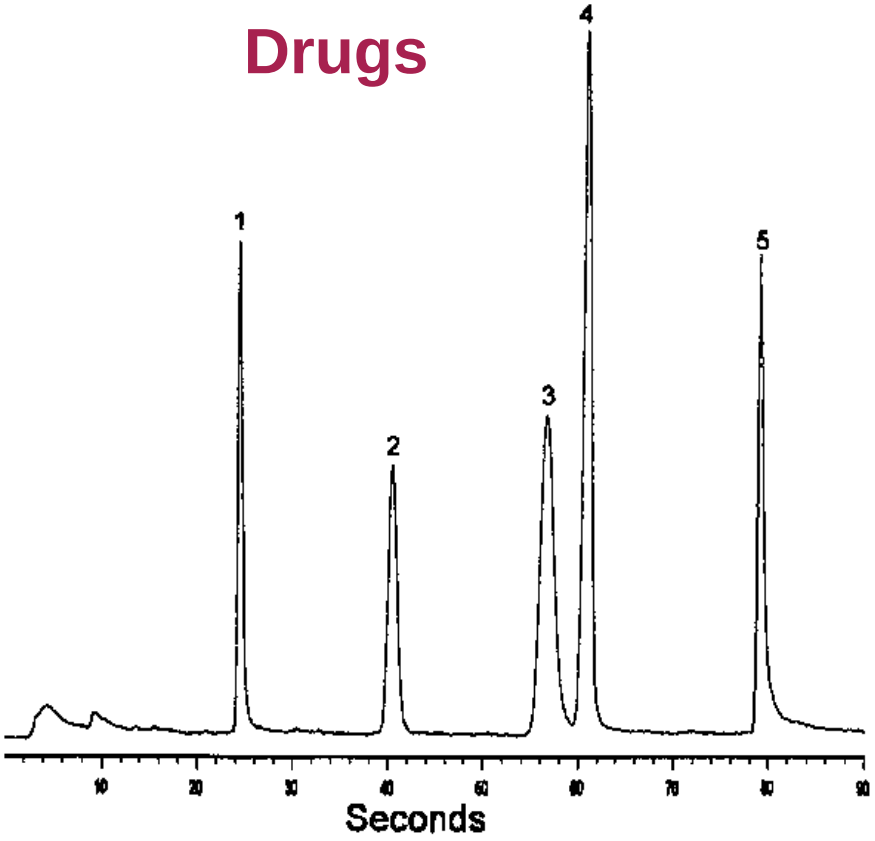
Fast Temperature Programming

- **Capillary GC column wrapped in a metal casing**
- **Resistively heats the capillary column**
- **Heats up to 20°C/sec**

THERMEDICS EZFLASH™



Column: TCX-DB17
6m, 0.25mm I.D., 0.50µm
Carrier: Hydrogen @ 32 psig
Detector: TEA in Nitrogen Mode, 850°C
Injector: 300°C
Oven: 80°C for 10 seconds
80-100°C at 120°C/min.
100-160°C at 180°C/min
160-280°C at 300°C/min.
280-300°C at 60°C/min.

Compounds:
1. Meperidine
2. Methadone
3. Benzotropine
4. Clomipramine
5. Verapamil

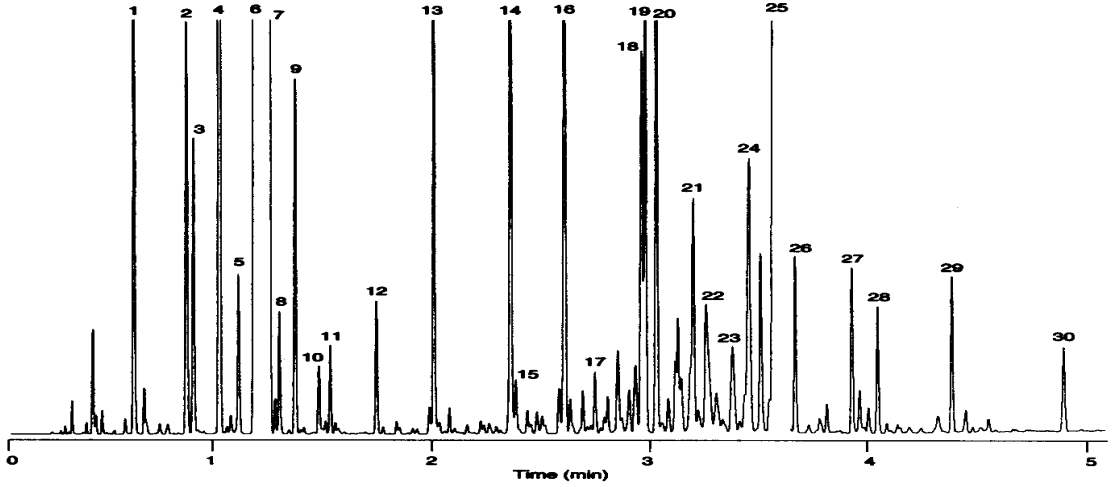
Short Microbore Columns

Western Spearmint Oil DB-Wax

10 m x 100 µm I.D., 02µm
µ = 55 cm/sec H2

Inlet: Split 275:1, 275°C
FID:350°C

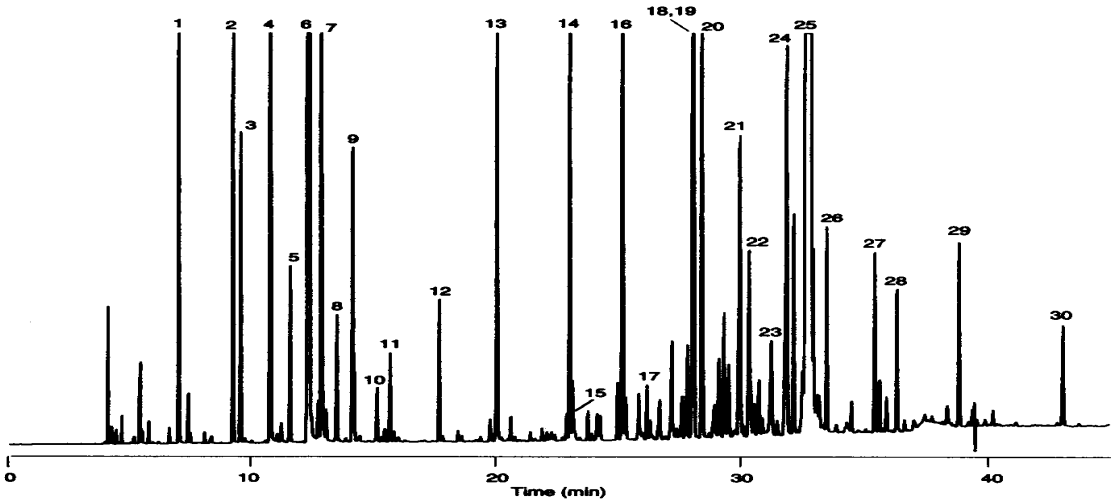
70°C for 0.5 min
30°/min to 120°C
20°/min to
200°C for 2 min



60m x 250 µm I.D., 025µm
µ = 25 cm/sec He

Inlet: Split 150:1, 270°C
FID:270°C

75°C for 8 min
4°/min to 200°C
200°C for 5 min



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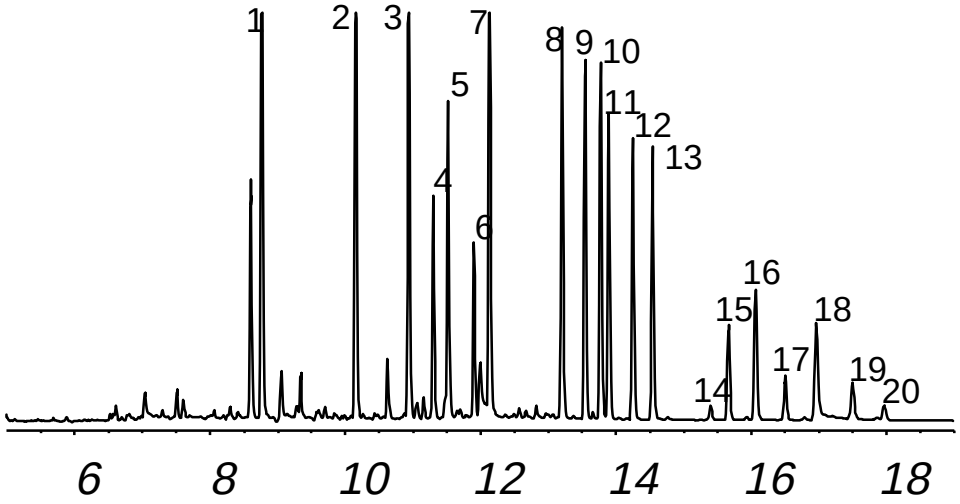
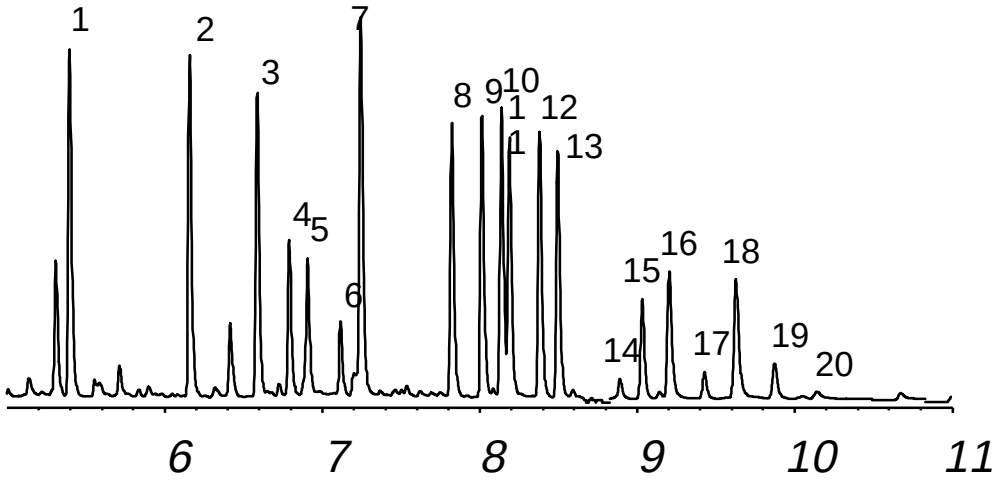
Small Dimension Changes

Pesticides DB-17ms



30 m x 0.25 mm I.D., 0.25 µm
Hydrogen at 45 cm/sec
Splitless
ECD
Oven program:
50°C for 0.5 min,
50-150°C at 25°/min,
150-260°c at 12°/min,
260-320°C at 15°/min



20 m x 0.18 mm I.D., 0.18 µm
Hydrogen at 45 cm/sec
Splitless
ECD
Oven program:
50°C for 0.5 min,
50-150°C at 35°/min,
150-260°C at 22°/min,
260-320°C at 25°/min

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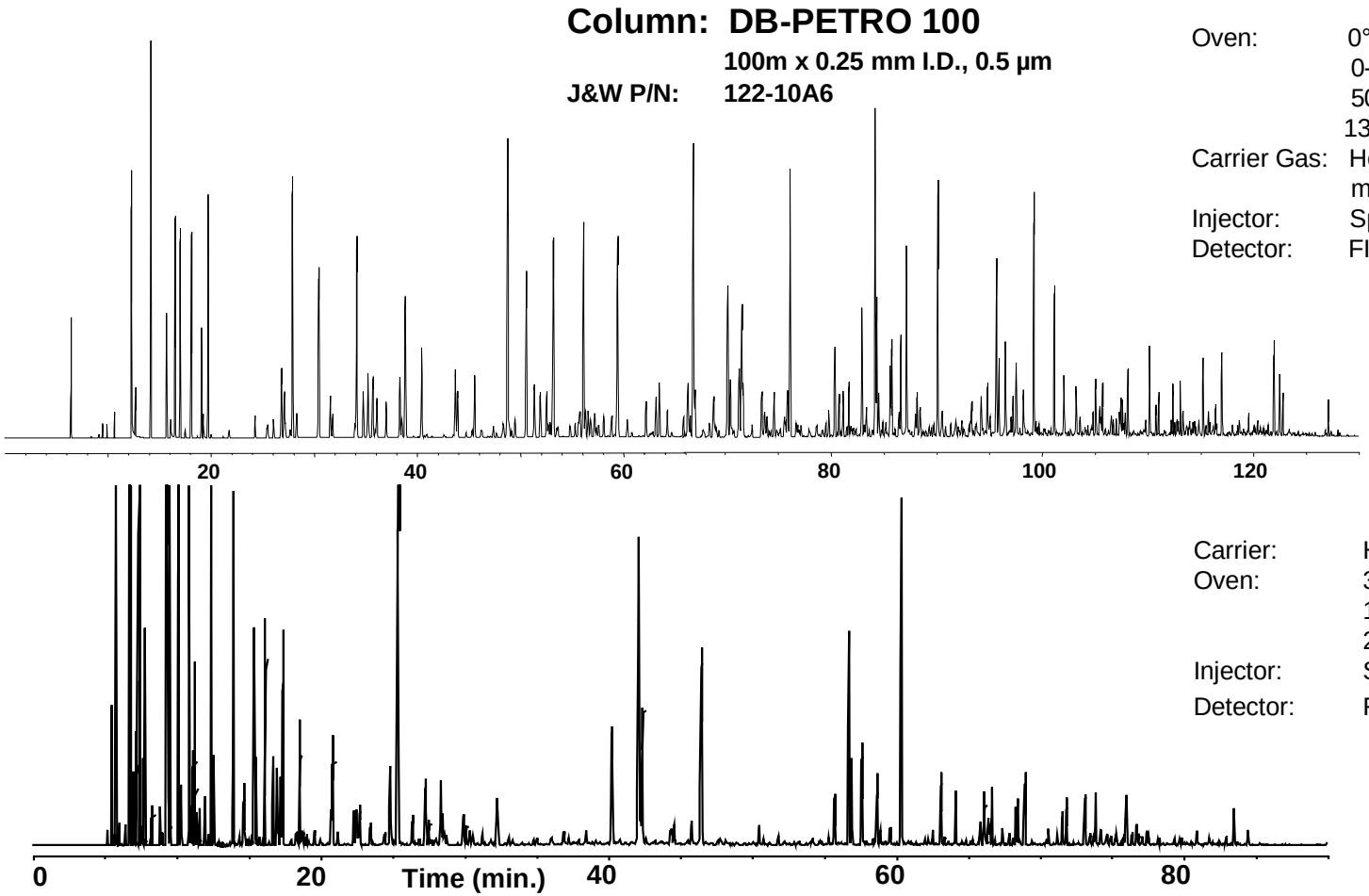
SAME GC SYSTEM
Optimize the Method



Questions to Ask

- **What information do you need from your analysis?**
- **Do you have more baseline than you need between your peaks?**
- **Do you need to resolve all of the components?**

Factors Affecting Resolution

$$R_s = [(N)^{1/2}/4]\,[k/(k+1)]\,[(\alpha-1)/\alpha)]$$

- Efficiency: N = theoretical plates
- Retention: k = retention factor
- Selectivity: α = separation factor

Variables for Shortening Run Times

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

Stationary phase and temperature changes do not affect the Kc of all solutes equally

Distribution Constant

$$K_C = \frac{\text{conc. of solute in stationary phase}}{\text{conc. of solute in mobile phase}}$$

- Change in K_c affects retention
- Co-elution if solute K_c's are equal
- K_c is determined by:

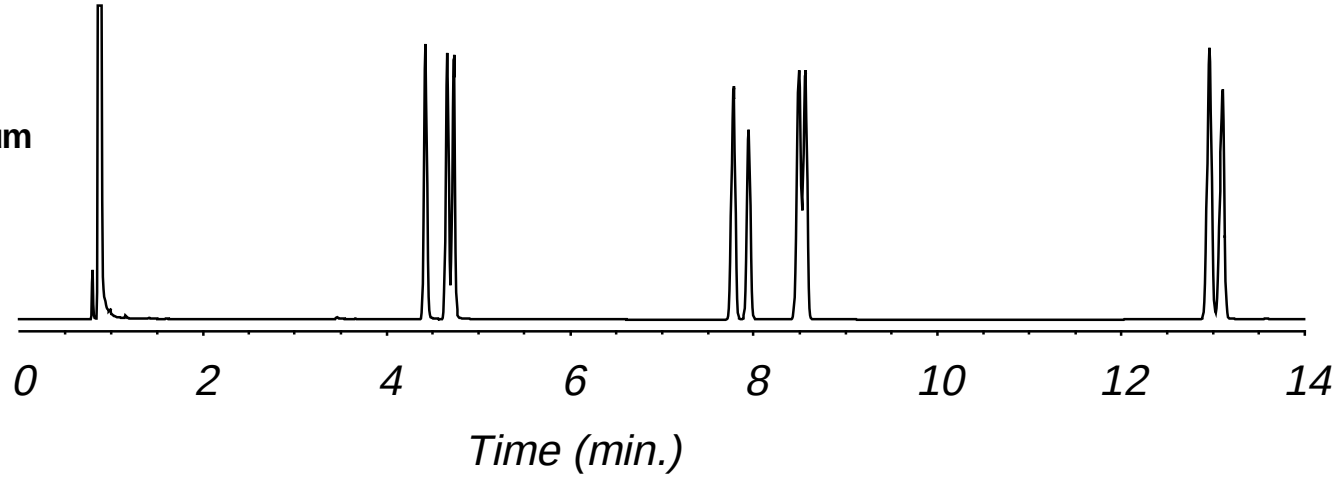
  solute

  stationary phase

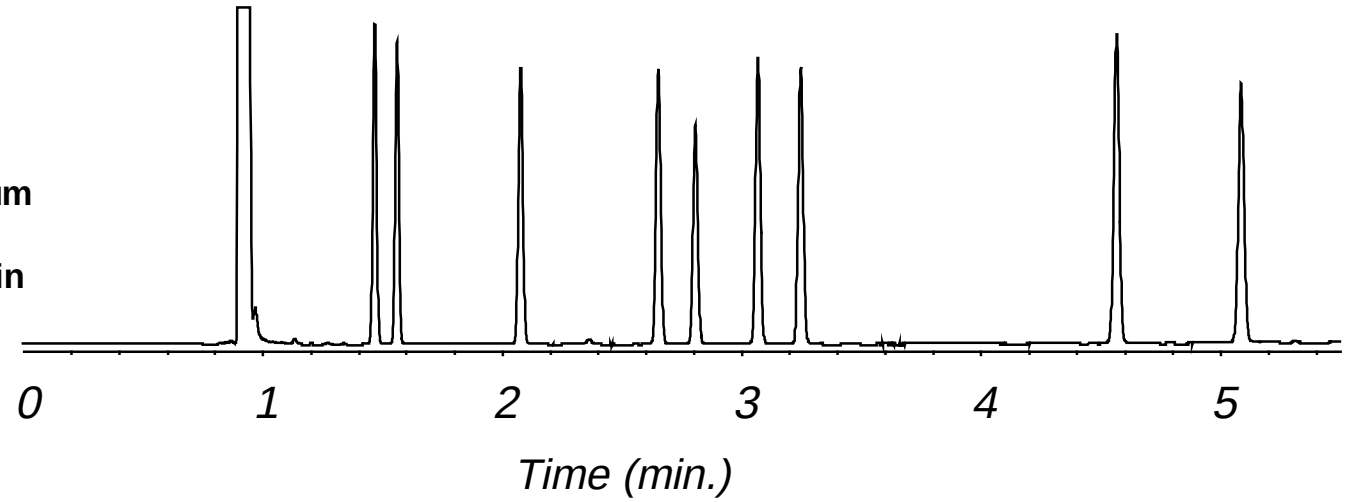
  temperature

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



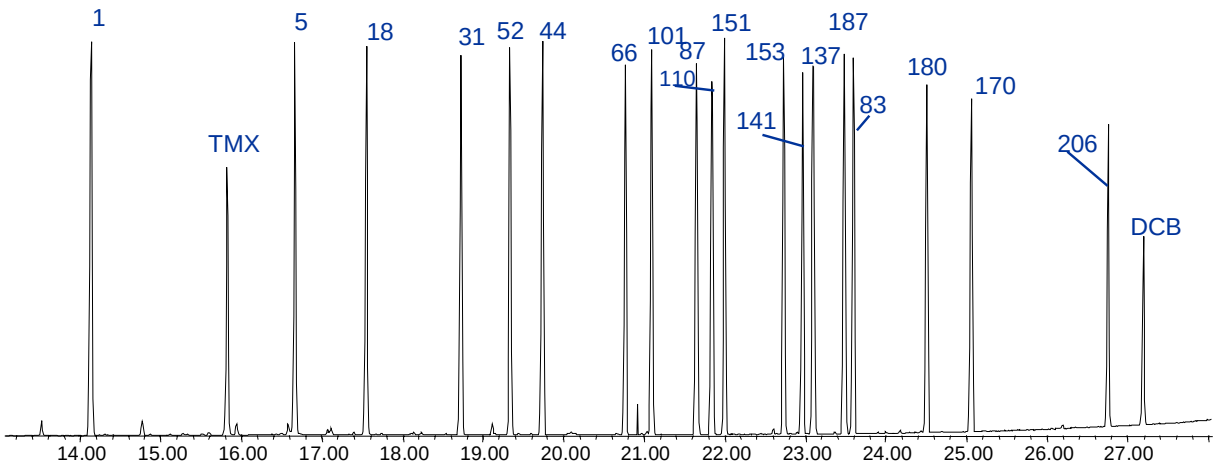
DB-Wax
15m, 0.32mm, 0.25µm
Oven:
80-190°C at 20°C/min



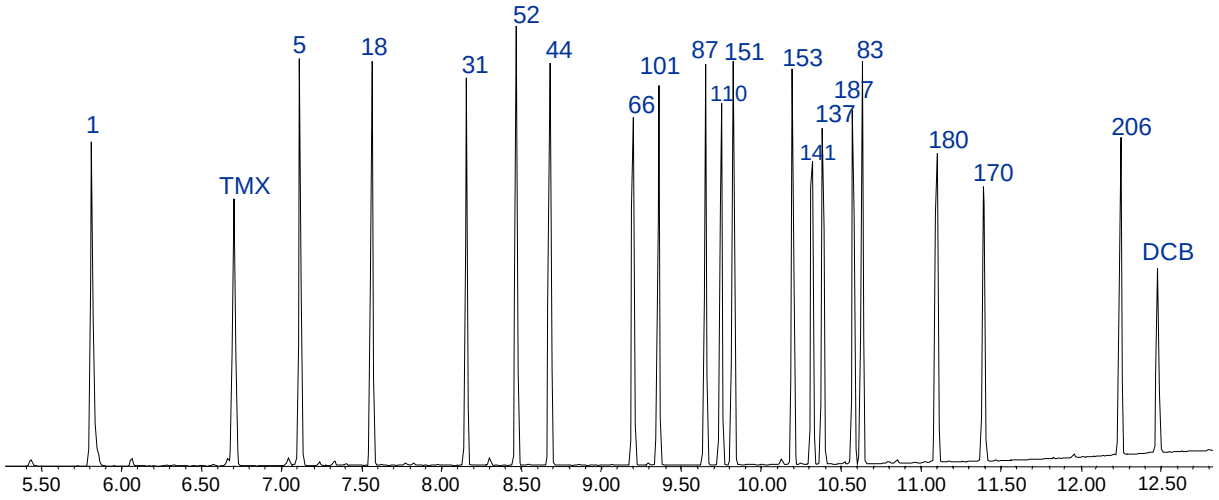
Temperature Programming

EPA Method 8082 A
Column: DB-XLB
30 m x 0.25 mm I.D., 0.25 µm
J&W P/N: 122-1232

Oven:	**50°C for 0.5 min** **50° to 340°C at 10°/min** **340°C for 5 min**
Carrier:	Helium, 36 cm/s @ 100°C constant flow mode
Injection:	275°C, Splitless 0.5 min
Detector:	HP 5973 MSD, 300°C transfer line temp, full scan of m/z 50-500
Sample:	1.0 µL of a 20 ng/µL mixture



Oven:	**100°C for 0.8 min** **100° to 340°C at 20°/min** **340°C for 2.2 min**
Carrier:	Helium, 36 cm/sec at 100°C constant flow mode
Injection:	275°C, Splitless 0.8 min
Detector:	HP 5973 MSD, 300°C transfer line temp, full scan of m/z 50-500
Sample:	1.0 µL of a 20 ng/µL mixture



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K_c and Temperature

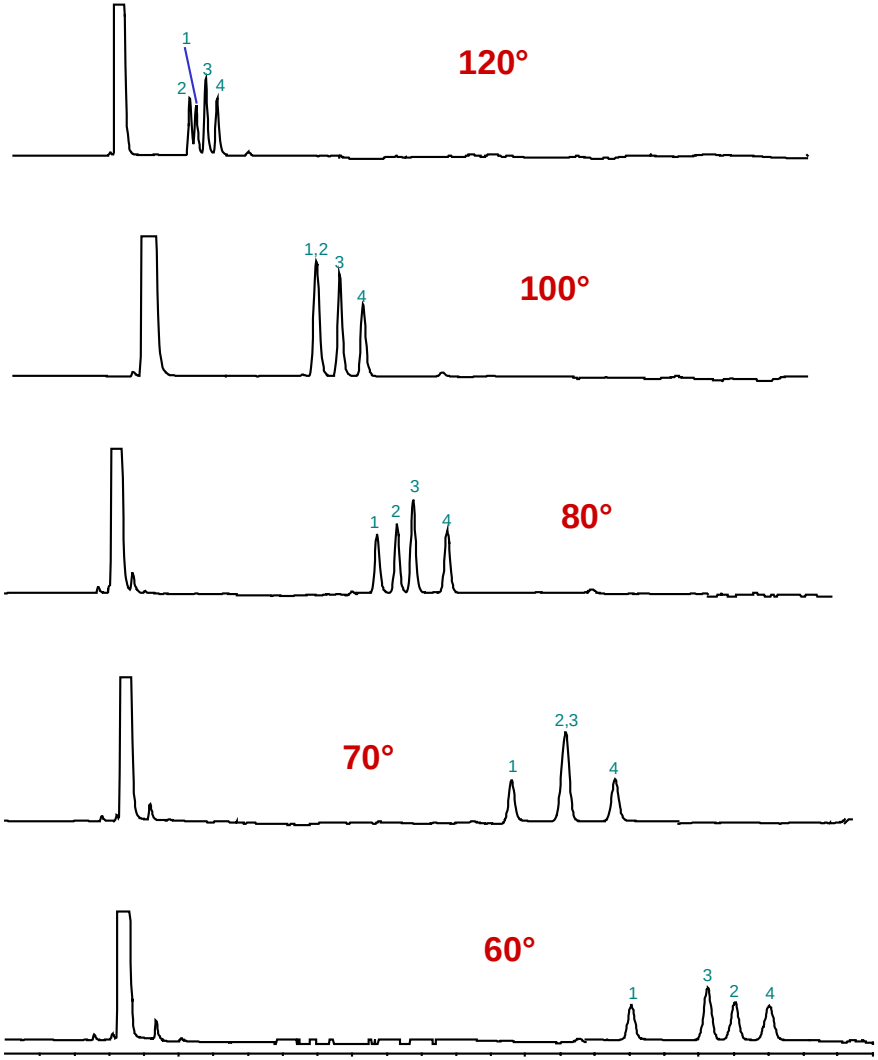
$$K_C = \frac{\text{conc. of solute in stationary phase}}{\text{conc. of solute in mobile phase}}$$

- K_C decreases with an increase in temperature
- Each solute's K_C may change at it's own rate

DB-WAX

Temperature Dependence

1. α-Terpinene
2. Dodecane
3. Limonene
4. 1,8-Cineole



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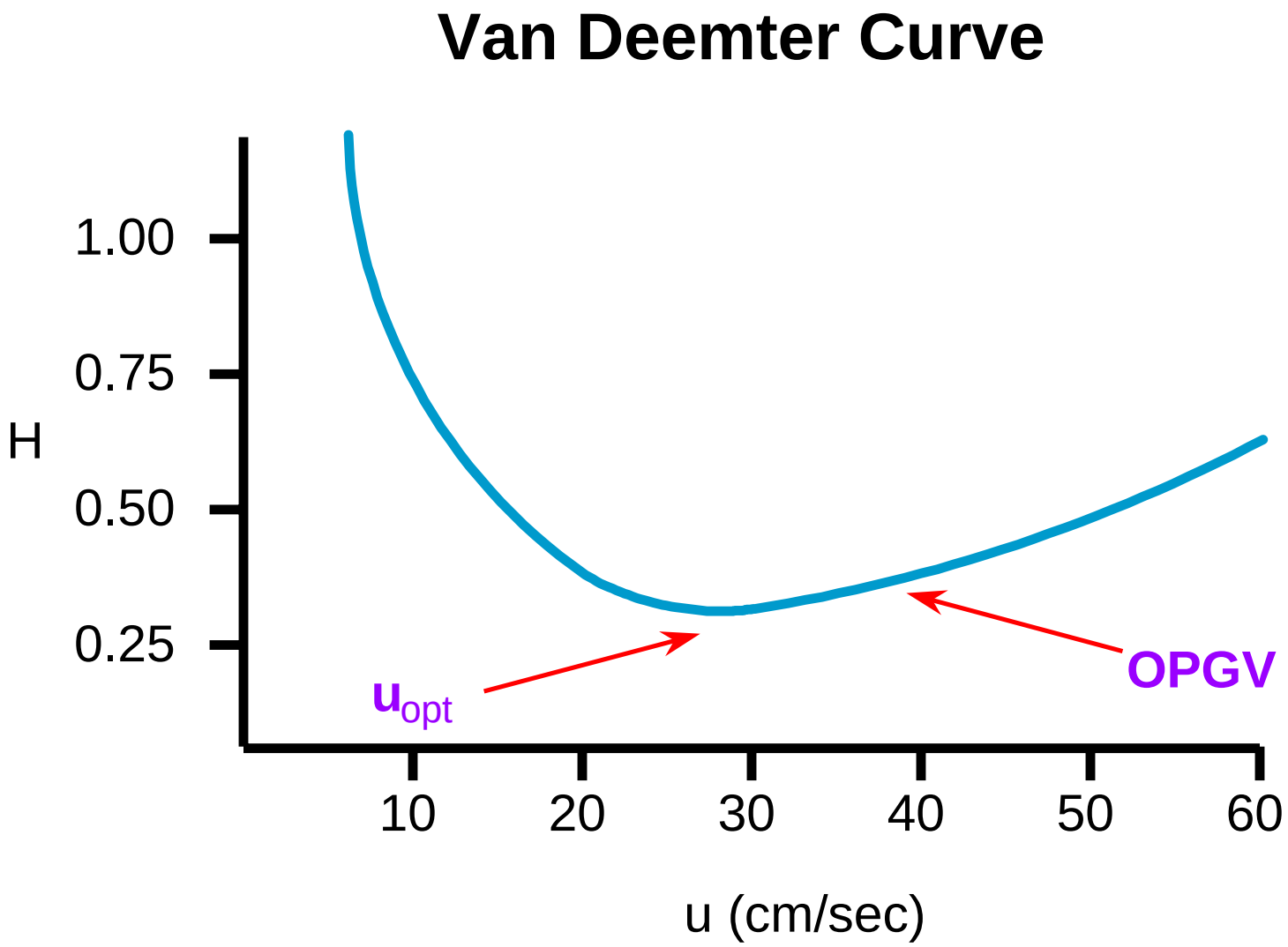
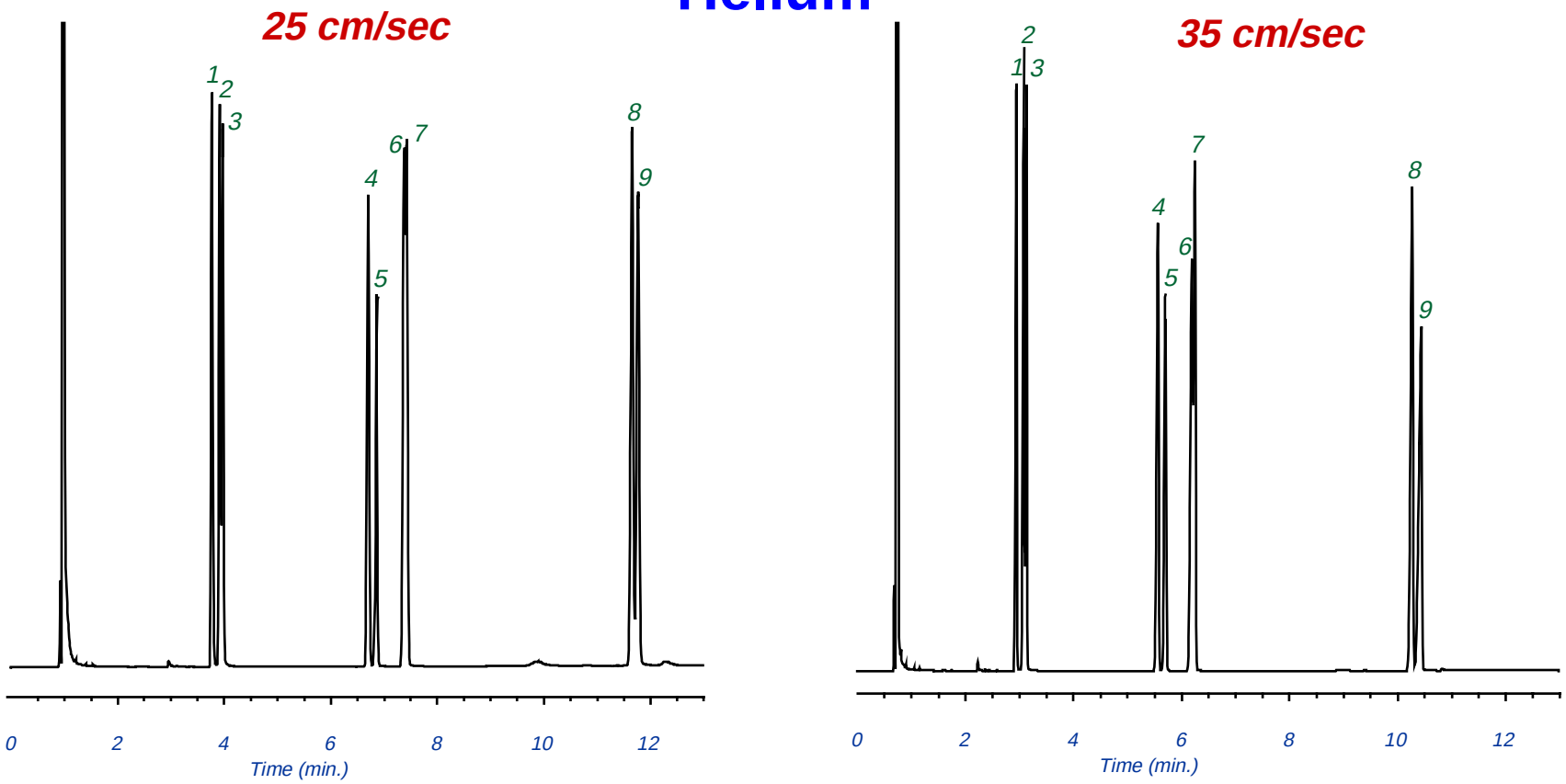
Carrier Gas
Type and Linear Velocity

Carrier Gas


Van Deemter Curve
1.00
0.75
H
0.50
0.25
u_{opt}
OPGV
10
20
30
40
50
60
u (cm/sec)

Carrier Gas
Linear Velocity

Helium



DB-1, 15 m x 0.25 mm I.D., 0.25 µm
50°C for 2 min, 50-110°C at 5°/min

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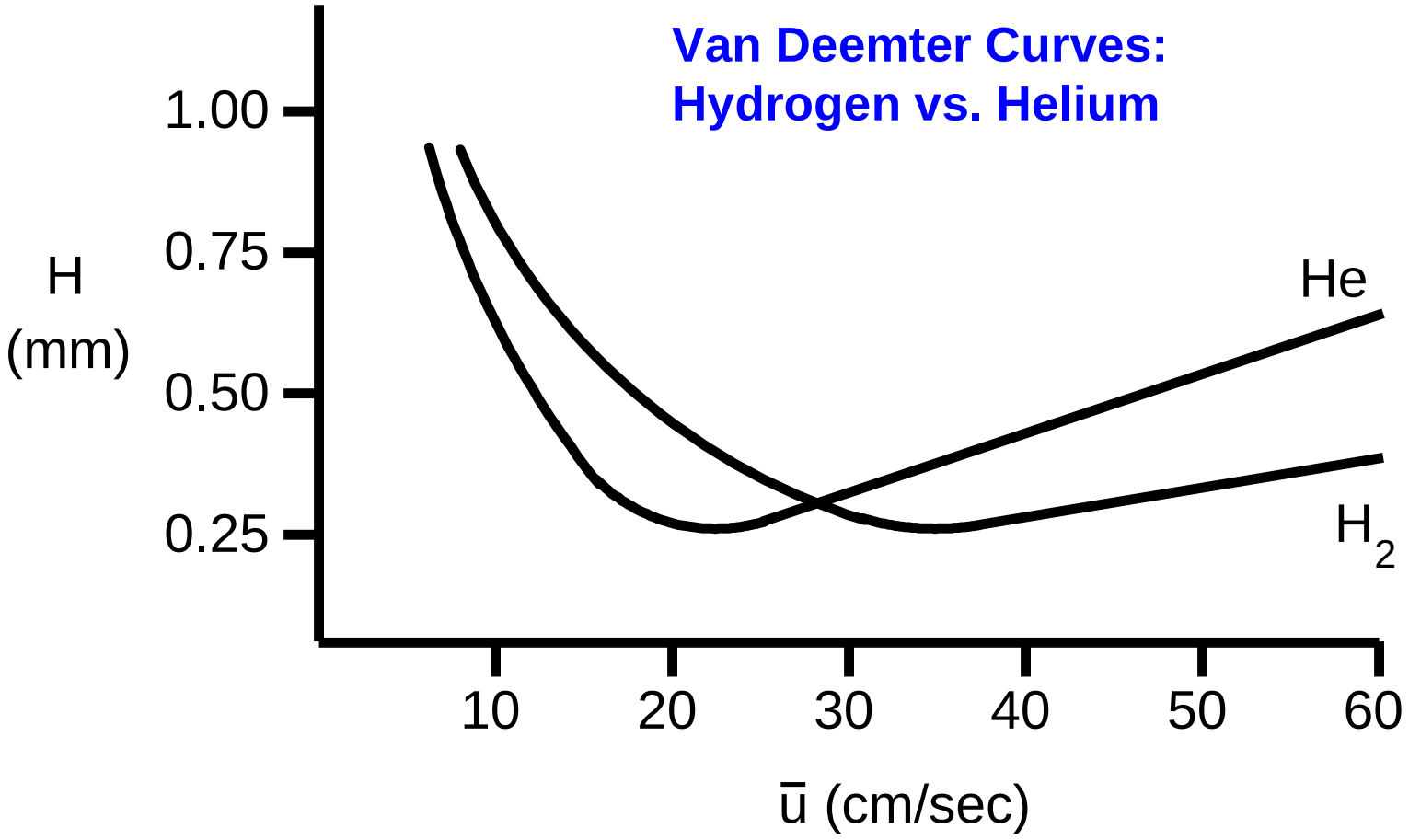
Carrier Gas
Type

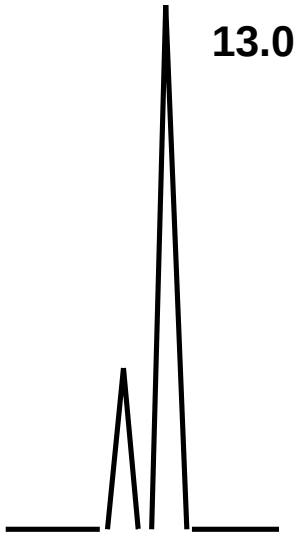


Hydrogen vs. Helium

SE-52
15m x 0.25mm
150°C isothermal

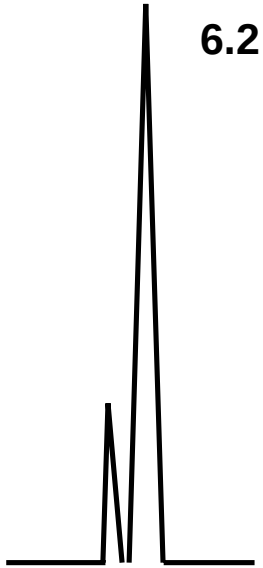
Compounds:
C17
Pristane

Helium
23.2 cm/sec



R = 1.67

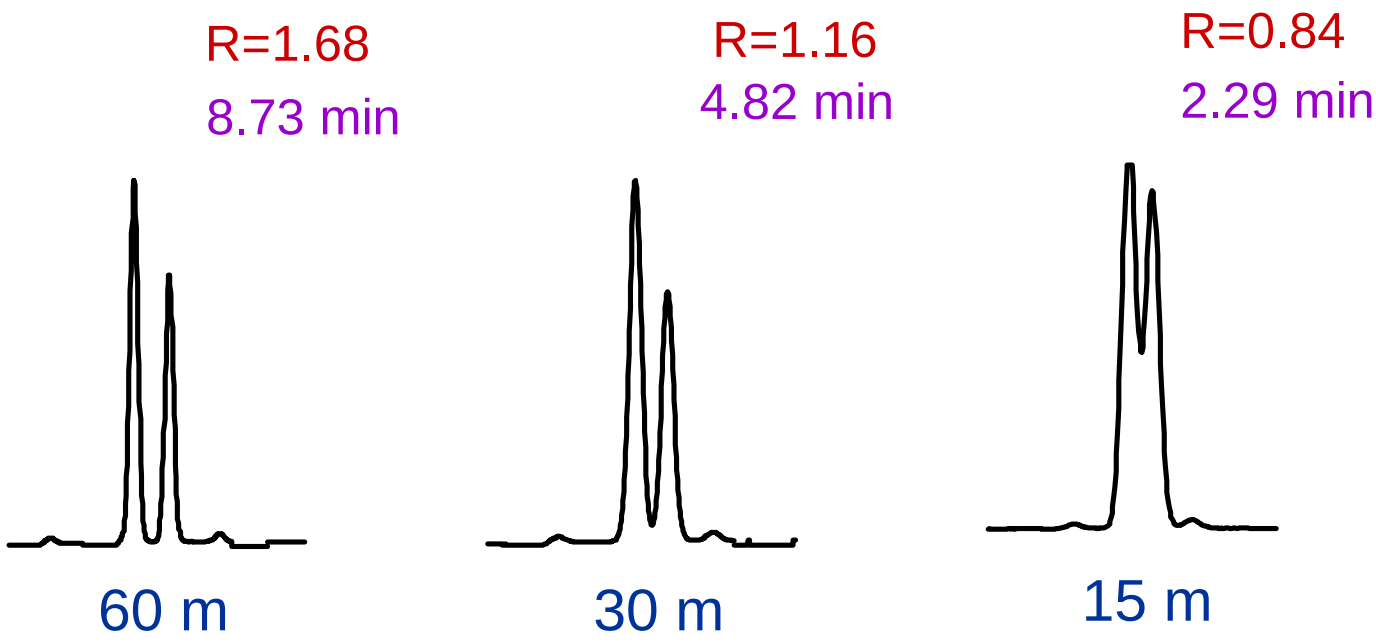
Hydrogen
48 cm/sec



R = 1.65

Optimize Column Dimensions

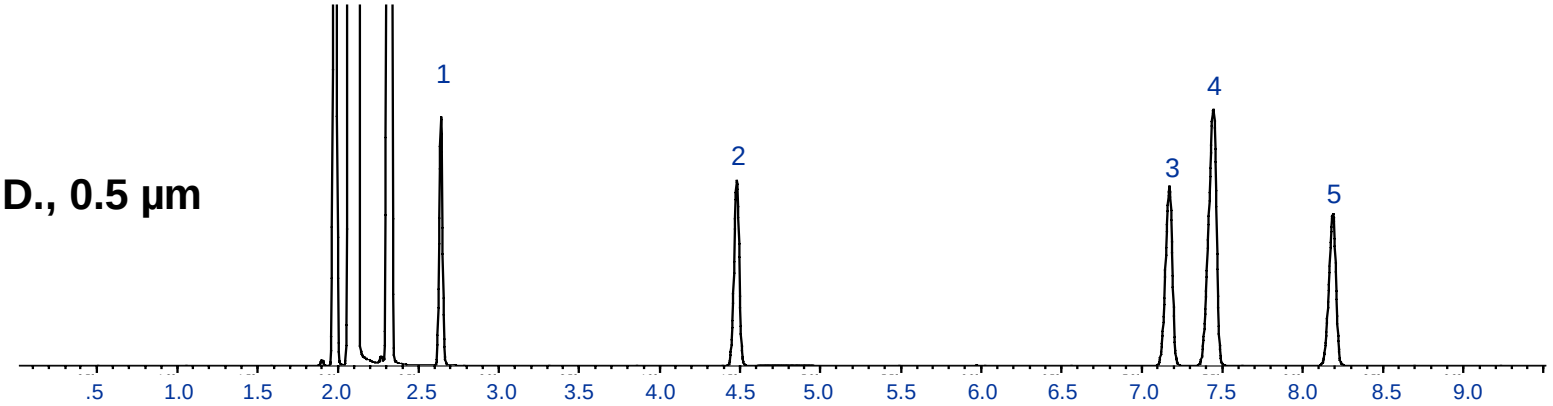
Column Length
Resolution and Retention
210°C isothermal



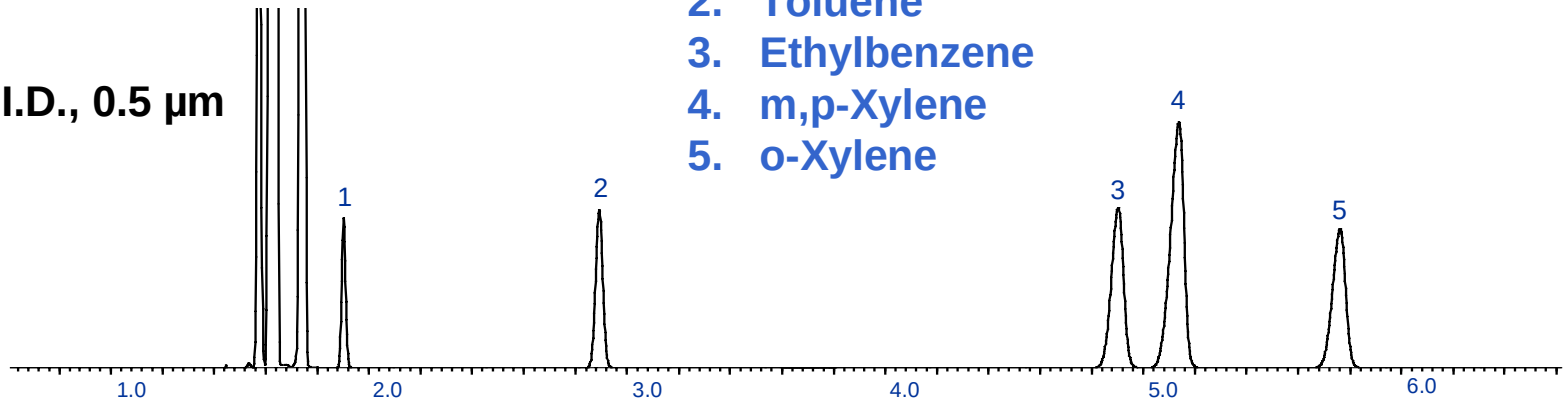
Resolution is proportional to square root of length
Isothermal: Retention is proportional to length
Temperature program: 1/3-1/2 of isothermal values

Decreasing Column Length

DB-5
30 m
0.53 mm I.D., 0.5 µm



DB-5
15 m
0.53 mm I.D., 0.5 µm

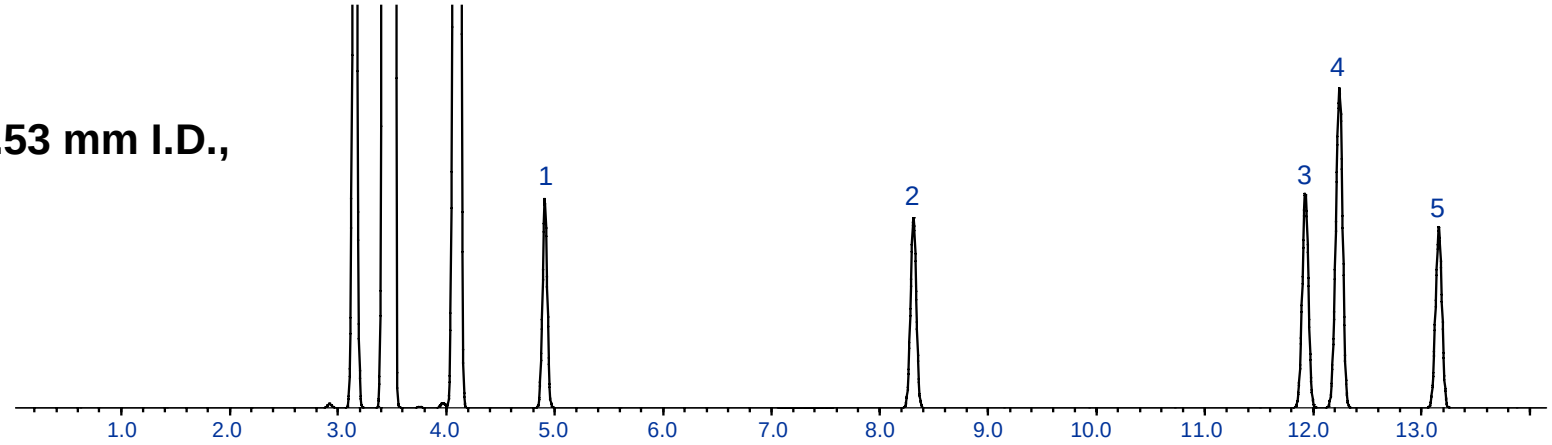


1. **Benzene**
2. **Toluene**
3. **Ethylbenzene**
4. **m,p-Xylene**
5. **o-Xylene**

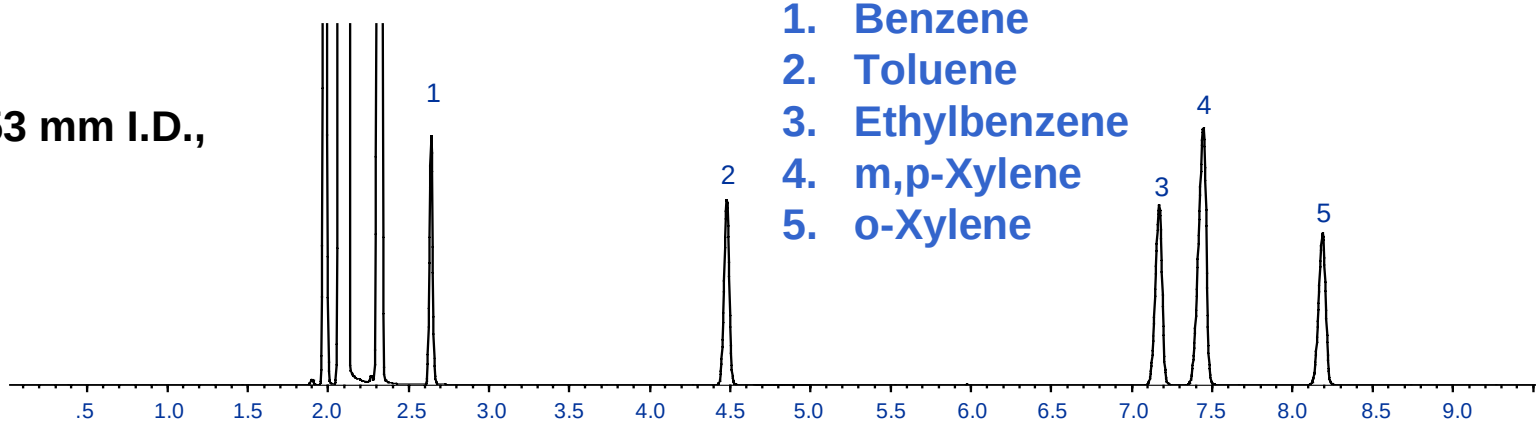
BTEX
Carrier: **Helium, 36 cm/sec at 40°c**
Oven : **40°C for 3 min, 5°/min to 100°C**

Decreasing Film Thickness

DB-5
30 m, 0.53 mm I.D.,
1.5 µm



DB-5
30 m, 0.53 mm I.D.,
0.5 µm



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

BTEX
Carrier: Helium, 36 cm/sec at 40°c
Oven : 40°C for 3 min, 5°/min to 100°C

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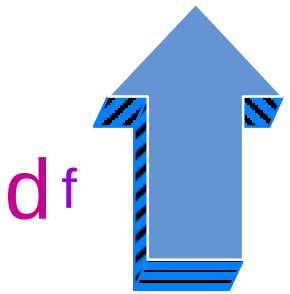
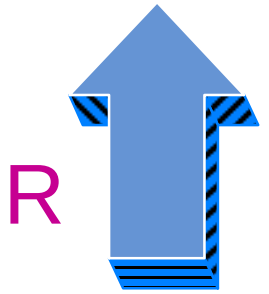
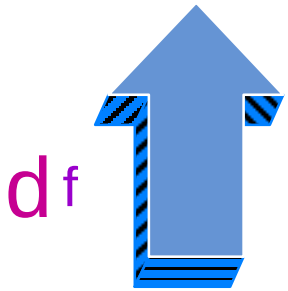
Effect of Film Thickness on Resolution

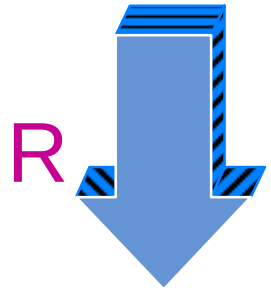
When solute k < 5





When solute k > 5





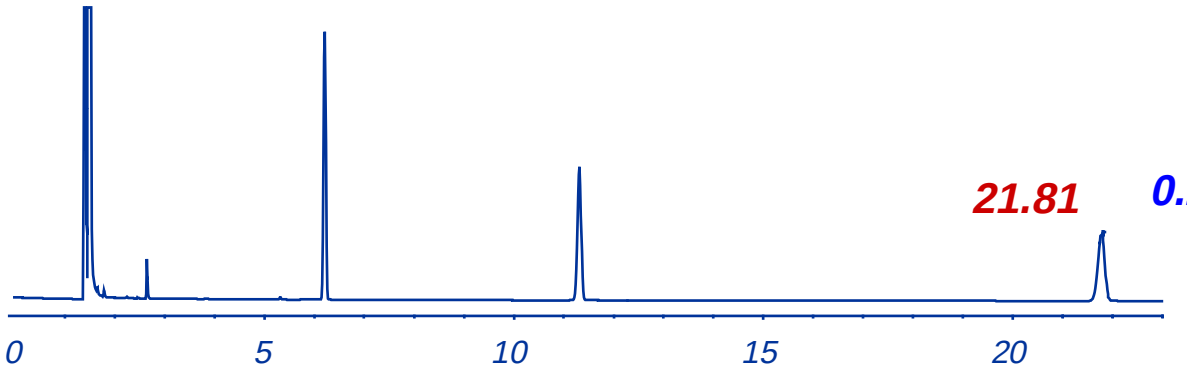
Column Diameter

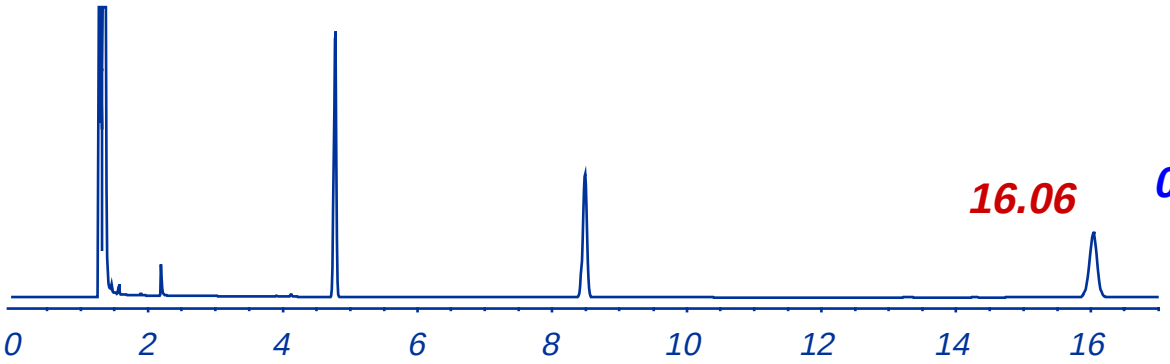
Retention: Same film thickness

I.D. (mm)	Retention Change (k)
0.18	1.39
0.20	1.25
0.25	1.00
0.32	0.78
0.53	0.47

Normalized to 0.25mm

Decrease Diameter
Effect on Retention





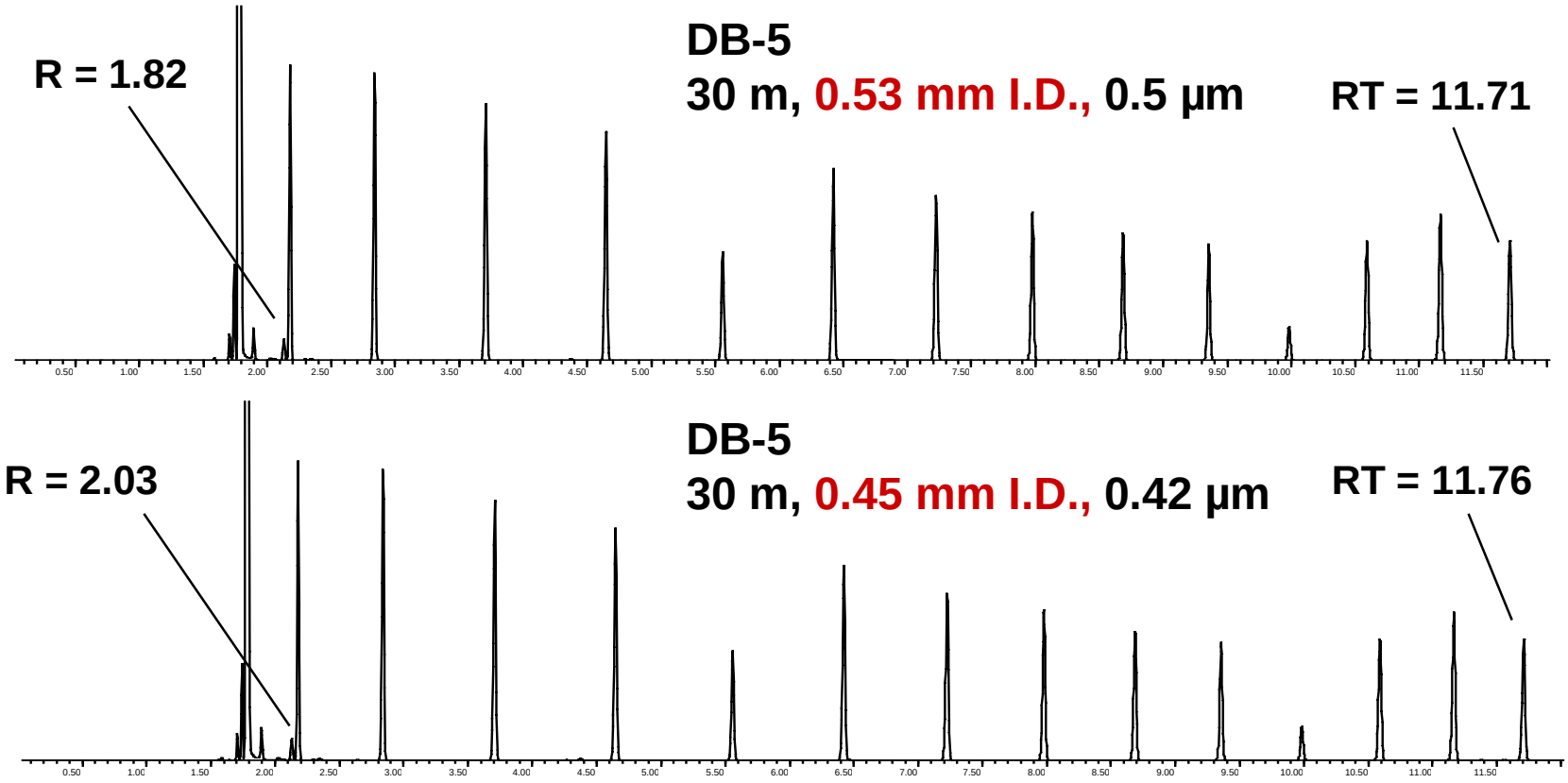
DB-1, 30 m, 0.25 um
80°C isothermal, He at 37 cm/sec
C10, C11, C12

Distribution Constant
K_C

$$K_C = k\beta$$

$$k = \frac{t_r'}{t_m} \qquad \beta = \frac{r}{2d_f}$$

Decrease Diameter
Phase Ratio Held Constant



C_7 - C_{20}
Carrier: Helium, 36 cm/sec at 40°C
Oven : 60°C for 1 min, 20°/min to 300°C

Column Diameter
Theoretical Efficiency

I.D. (mm)	N/m
0.10	11905
0.18	6666
0.20	5941
0.25	4762
0.32	3717
0.53	2242

k = 5

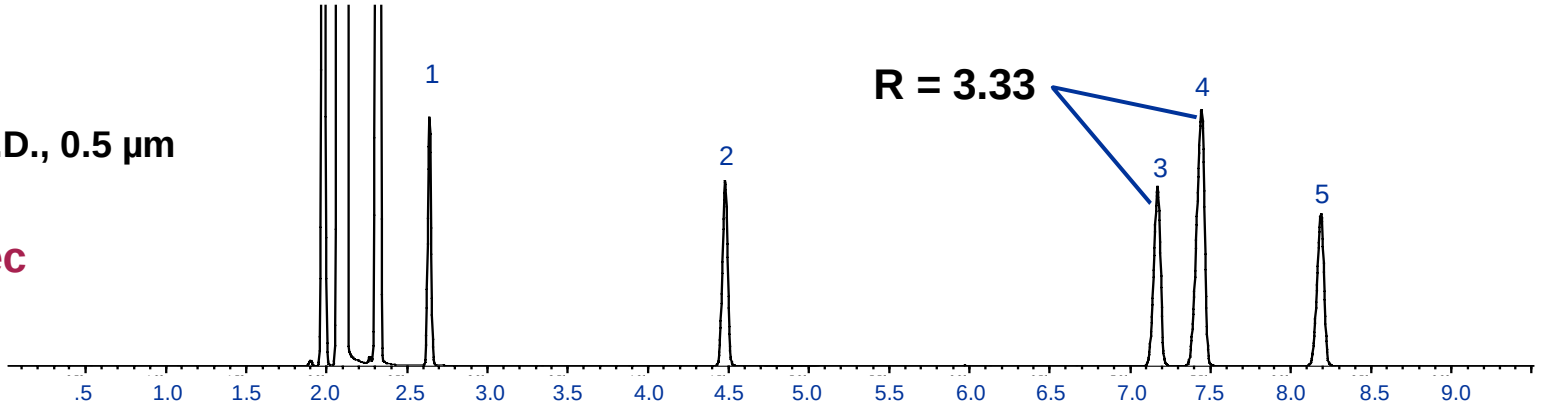
Decrease Diameter

- **Small changes**
  - **examples: 0.53 mm to 0.45 mm**
    0.25 mm to 0.20 mm
- **Extreme changes**
  - **Using short microbore columns: ≤ 0.1mm**

Decrease Diameter
Adjust Linear Velocity

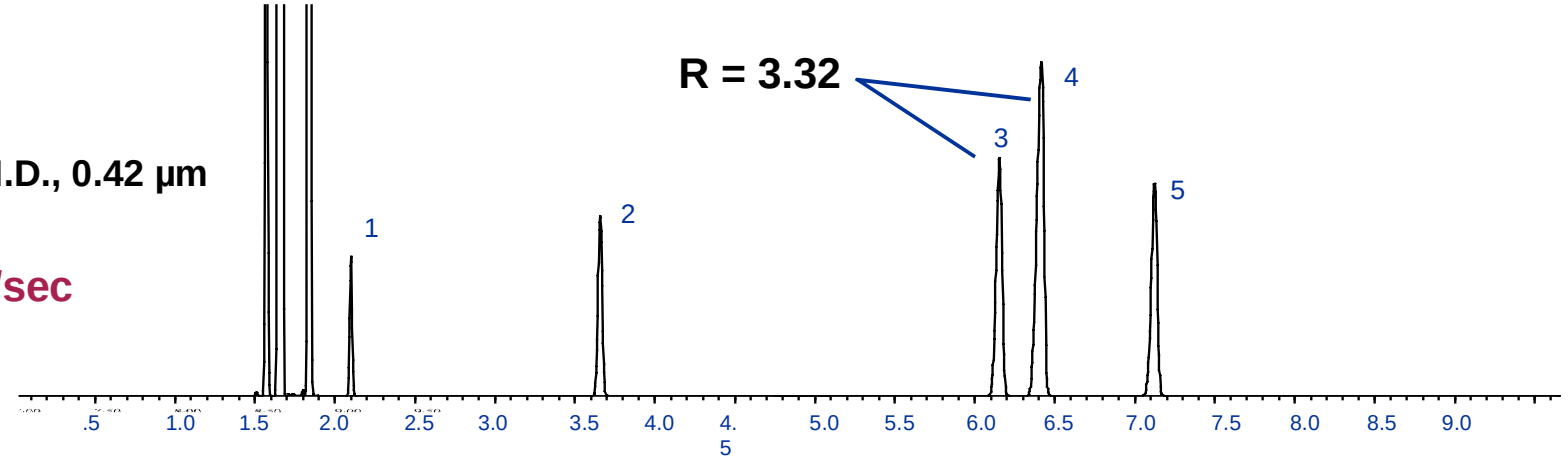
DB-5
30 m,
0.53 mm I.D., 0.5 µm

36 cm/sec



DB-5
30 m,
0.45 mm I.D., 0.42 µm

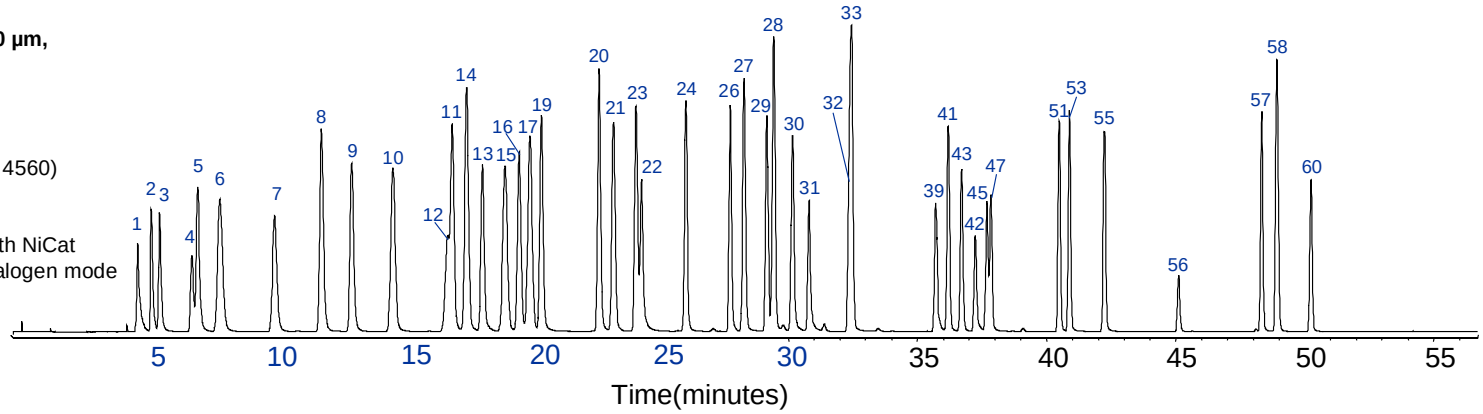
45.9 cm/sec



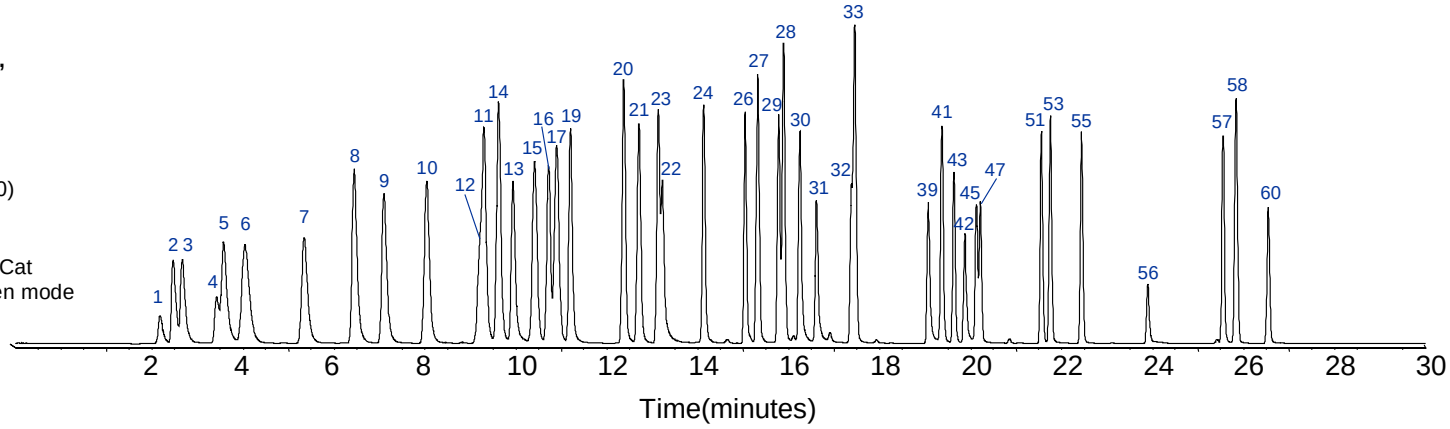
BTEX
Carrier: Helium
Oven : 40°C for 3 min, 5°/min to 100°C

Decrease Diameter
Small Change - adjust conditions

Column: **DB-502.2**
105m x 0.53mm x 3.0 µm,
Oven: 35°C for 10 min,
35-200°C at 4°C/min
200°C for 5 min
Carrier: Helium at 10 ml/min
Injector: Purge and trap (O.I.A 4560)
Trap: Tenax/Silica gel/CMS
Desorb: 200°C for 0.6 min
Detector B: ELCD (O.I.A 4420) with NiCat reaction tube in the halogen mode



Column: **DB-502.2**
75m x 0.45mm x 2.55 µm,
Oven: 35°C for 6 min,
35-200°C at 8°C/min
200°C for 3.5 min
Carrier: Helium at 10 ml/min
Injector: Purge and trap (O.I.A 4560)
Trap: Tenax/Silica gel/CMS
Desorb: 200°C for 0.6 min
Detector B: ELCD (O.I.A 4420) with NiCat reaction tube in the halogen mode



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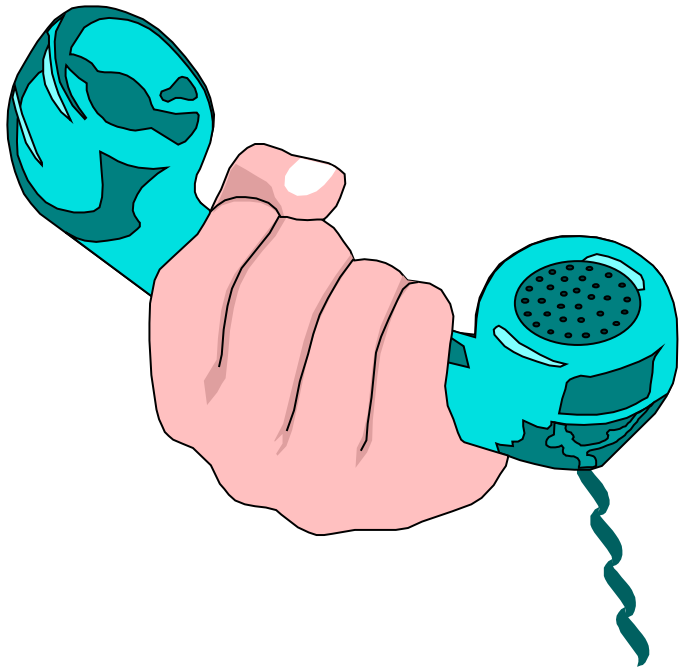
Extreme Diameter Changes

- **Shorten column length to decrease run time**
- **Increase plates/meter by decreasing column diameter**
- **For similar retention and selectivity keep the stationary phase and phase ratio**

  ($\beta = r/d_f$) the same.

Break

- For Questions and Answers
- Press *1 on Your Phone to
- Ask a Question



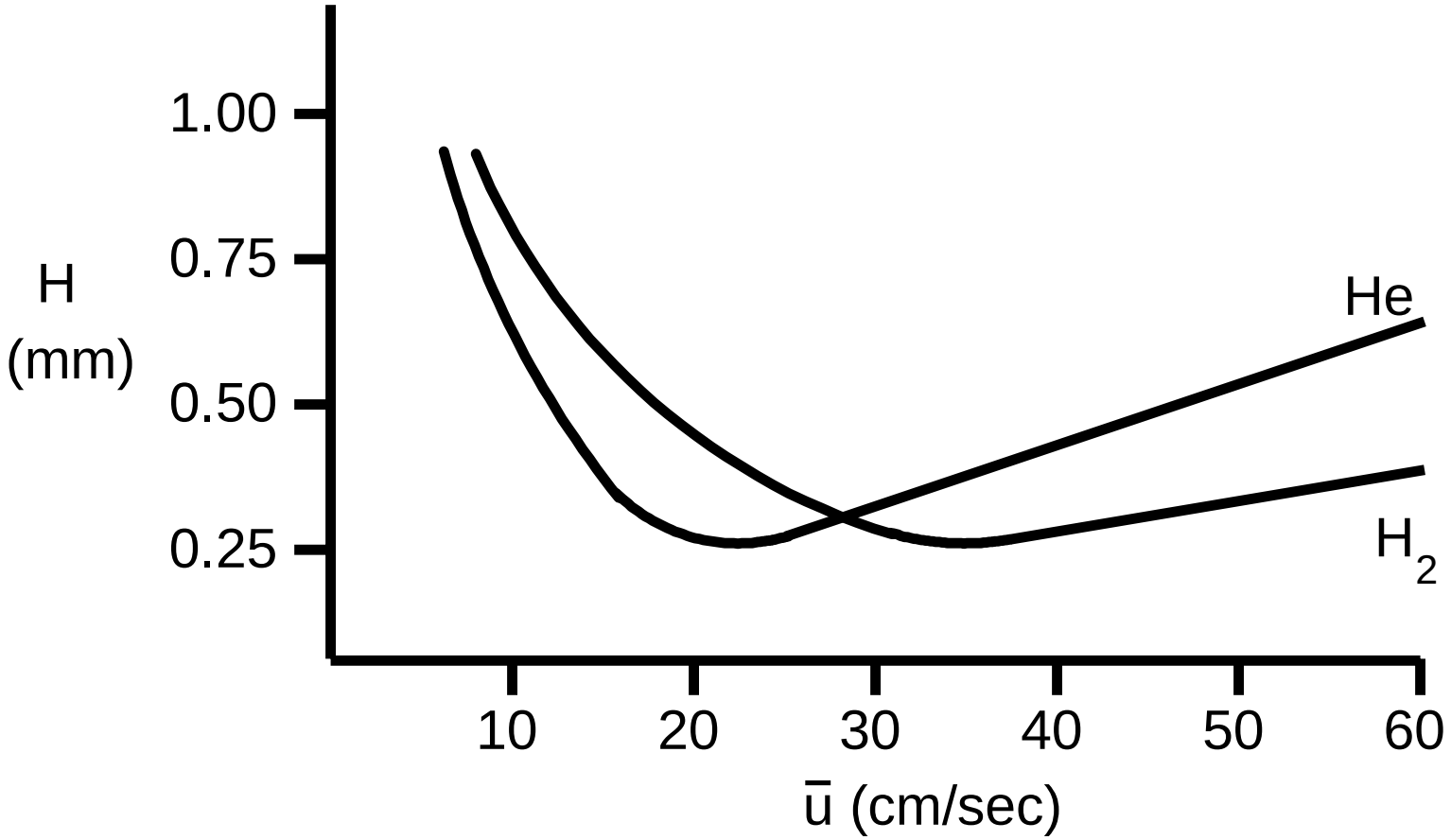
Considerations of using 0.1mm ID Columns

- Carrier Gas
- Temperature Program
- Injection efficiency
- Data system and detectors
- Working Range

Carrier Gas

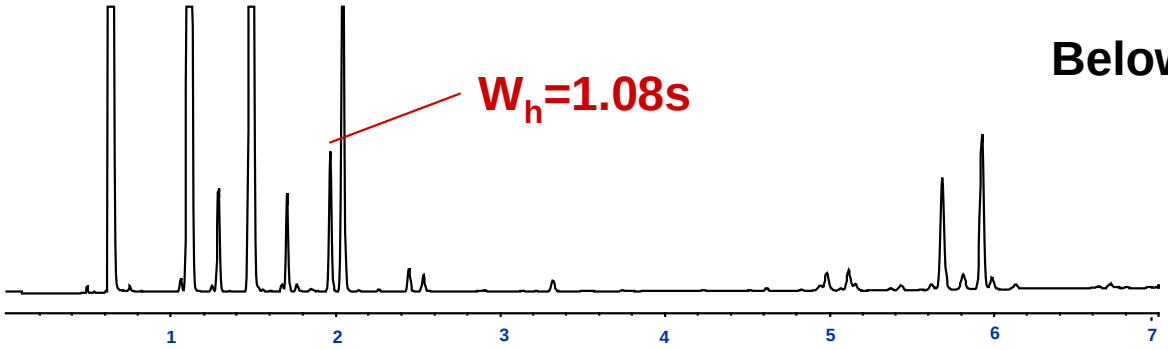
- **Hydrogen is the accepted carrier gas for fast GC analysis**
- **Fast optimal linear velocity**
- **Lower head pressure requirements**

Van Deemter Curves:
Hydrogen vs. Helium



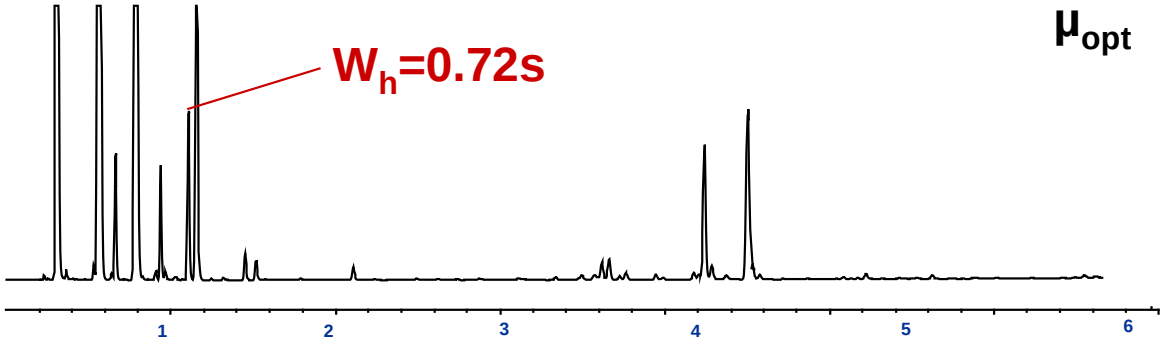
Turpentine
DB-WAX

10 m x 0.10 mm, 0.2 µm
70° to 180°C at 15°/min
H_2, 30 cm/sec at 70°



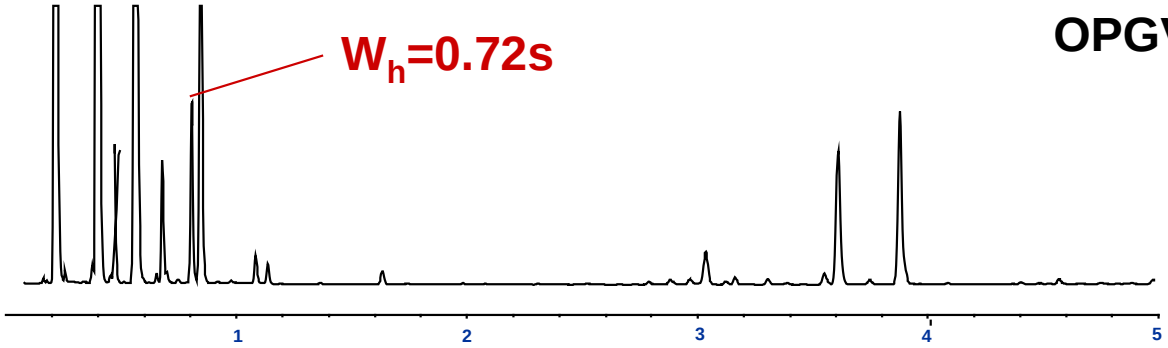
Below μ_{opt}

10 m x 0.10 mm, 0.2 µm
70° to 180°C at 15°/min
H_2, 65 cm/sec at 70°



μ_{opt}

10 m x 0.10 mm, 0.2 µm
70° to 180°C at 15°/min
H_2, 95 cm/sec at 70°



OPGV

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Pressure Considerations: 0.10 ID

Hydrogen

10m	Velocity	Split Ratio	Split Flow	Column Flow	Head Pressure
μ_{opt}	65	300	204	0.68	33.5
OPGV	94	300	387	1.29	50.1

Helium

10m	Velocity	Split Ratio	Split Flow	Column Flow	Head Pressure
μ_{opt}	41	300	153	0.51	43.3
OPGV	59	300	394	0.98	64.5

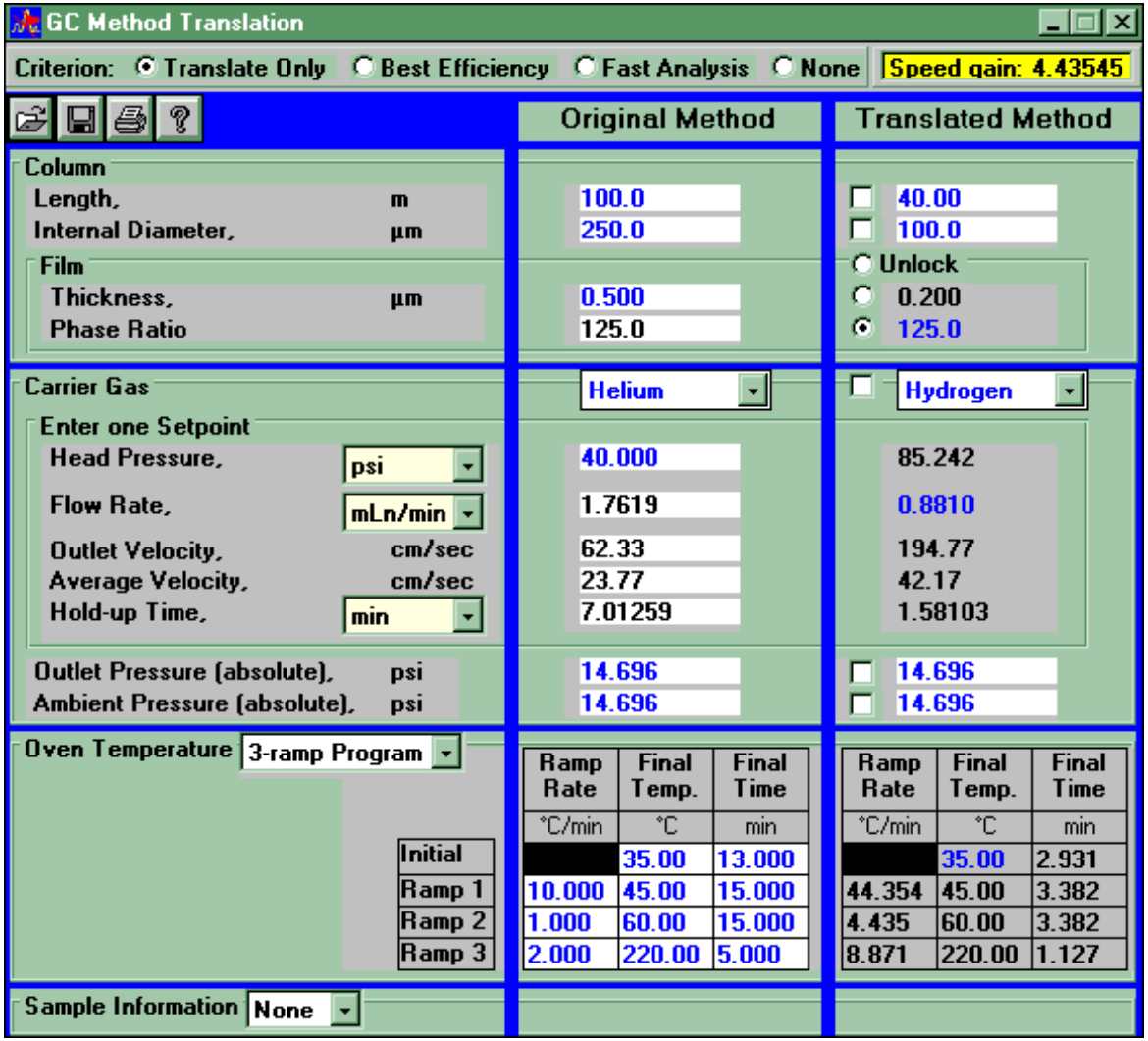
K_c and Temperature

- K_c of analytes must be maintained
- Temperature programs must be accurately scaled to maintain relative analyte retention

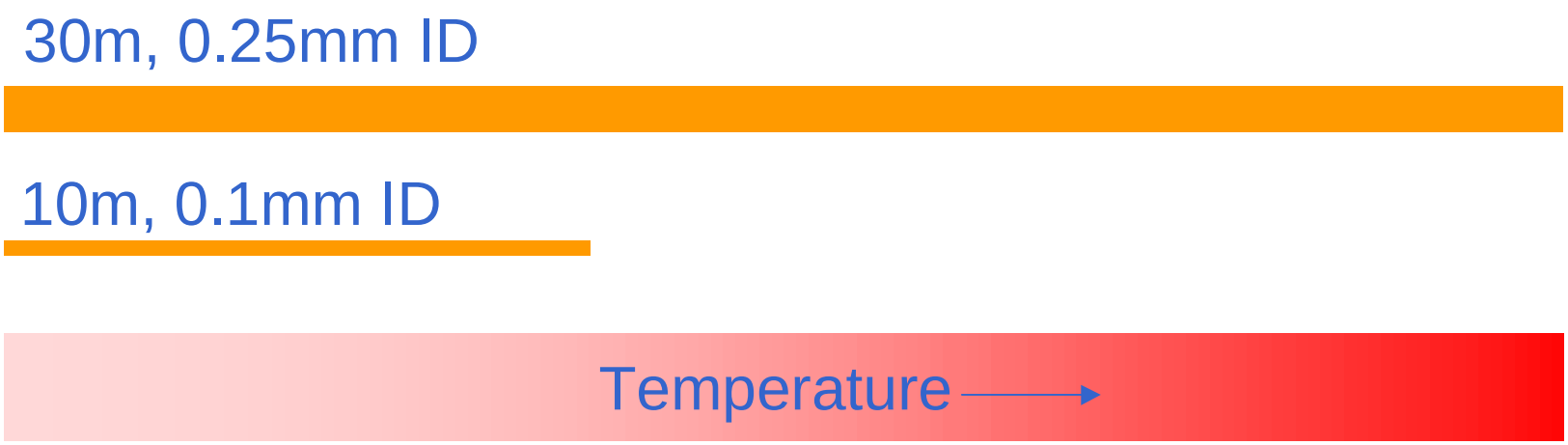
Method Translation Software

- **Tool allowing GC methods to be translated to different conditions & maintain selectivity/resolution**
  - new column configuration
  - different carrier gas
  - faster separation
- **Translates:**
  - inlet Pressure, temp program, hold times
- **Benefits**
  - reduces methods development time
  - help assess if GC method compatible with HW

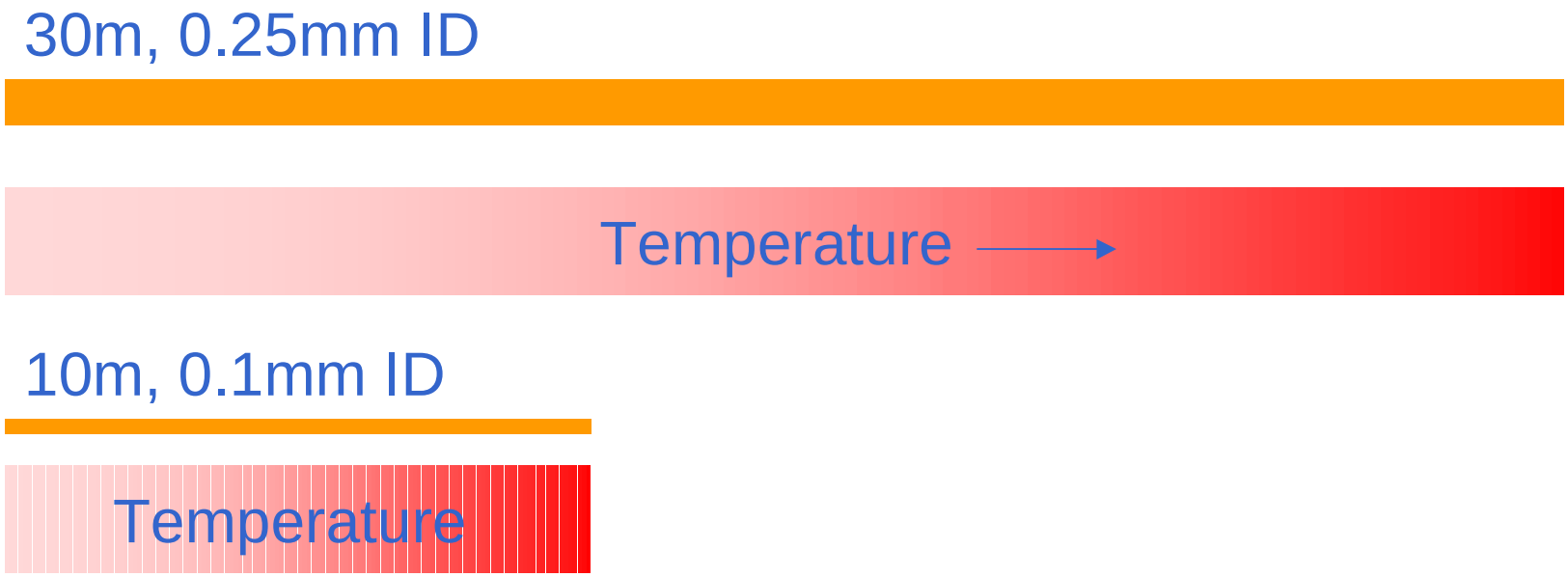
Method Translation Software



Temperature Programming



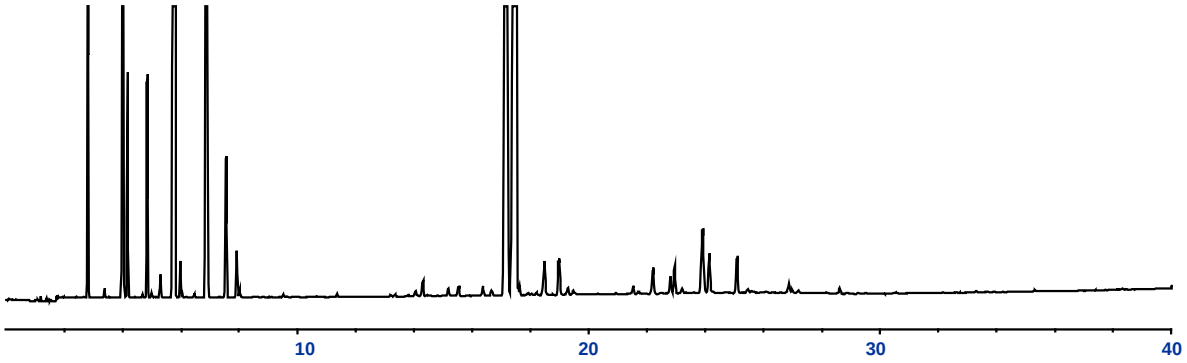
Temperature Programming



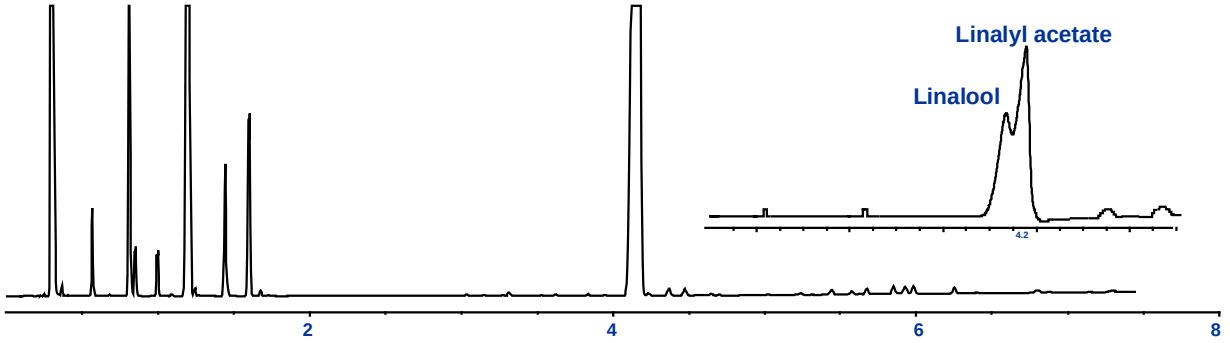
Temperature program must be modified to give same temperature of elution (i.e. faster ramps, shorter hold times)

Bergamot Oil
DB-WAX

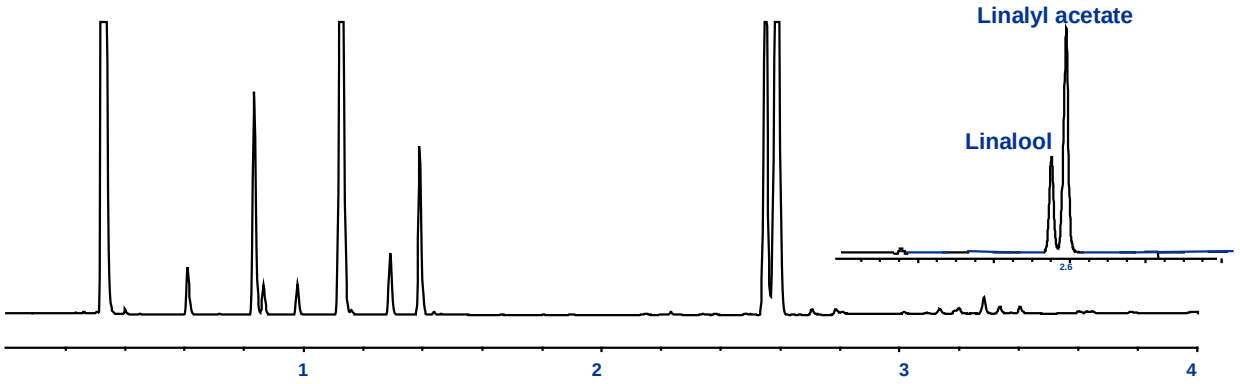
30 m x 0.25 mm, 0.5 µm
70° to 200°C at 3°/min
H_2, 45 cm/sec at 70°



10 m x 0.10 mm, 0.2 µm
70° to 200°C at 10.4°/min
H_2, 65 cm/sec at 70°



10 m x 0.10 mm, 0.2 µm
60° to 180°C at 30°/min
H_2, 65 cm/sec at 60°



Injector Efficiency

Injector Efficiency

Narrow columns generate narrow peaks

Injection band must be narrow to take advantage of the column efficiency

Injection Techniques

Split: high split ratio

Cold trapping

Data System Requirements

Narrow columns generate narrow peaks

0.5-5 second peak widths are common

Requires fast sample rate of detectors and data system

Detectors

FID - works well

NPD - works well

ECD - increase makeup, but very large dead volume

TCD - large volume issue

MS - scan speed

Area Counts Reduced

Scan rates- 50Hz for GC

12-20/sec scans for MS

Check your system

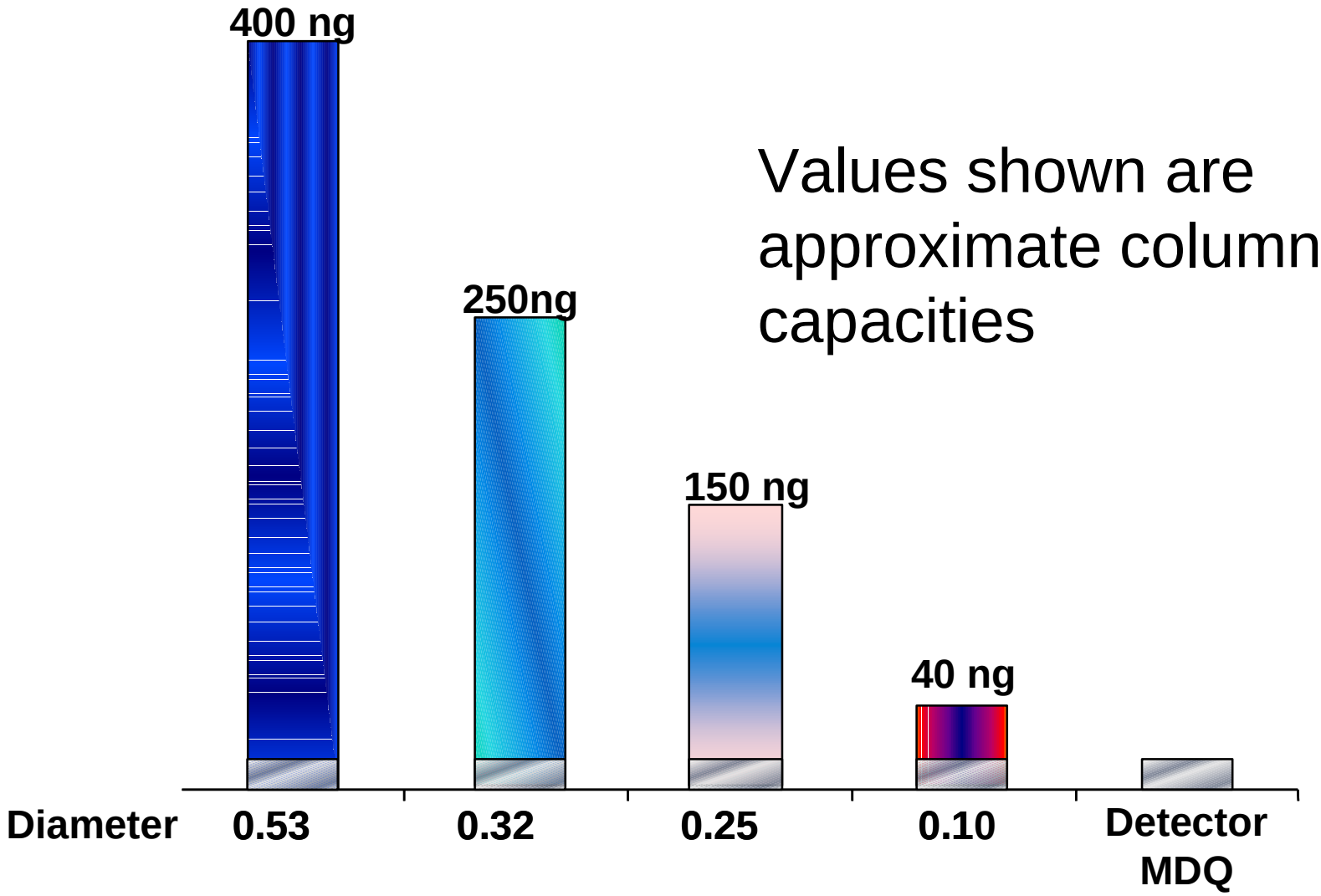
Working Range

$$W = Q_s \div Q_o$$

Q_s = maximum column capacity
Q_o = minimum amount that can be reliably detected

- Column capacity is proportional to column diameter
- Column diameter will have little effect on detector sensitivity

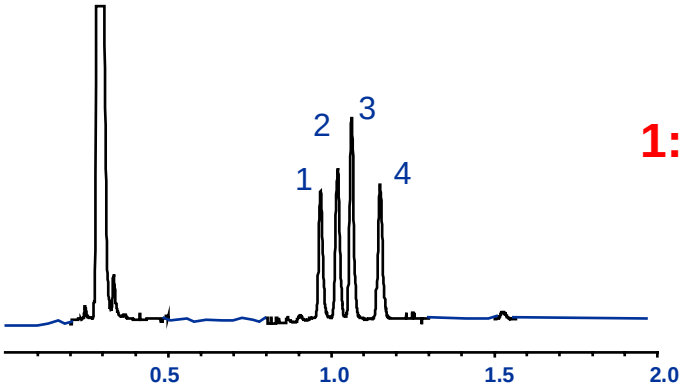
Working Range



Values shown are approximate column capacities

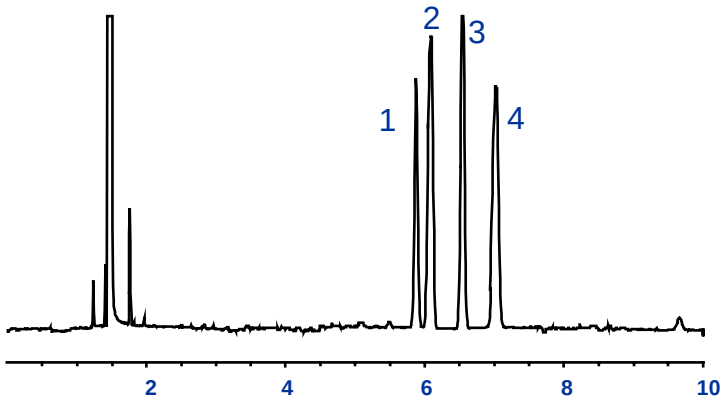
Capacity: Effect on Resolution

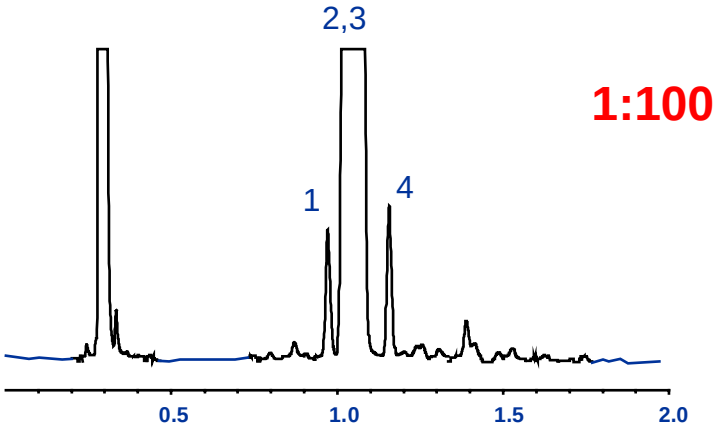
10 m x 0.1 mm, 0.2 µm



1:1:1:1

30 m x 0.25 mm, 0.5 µm





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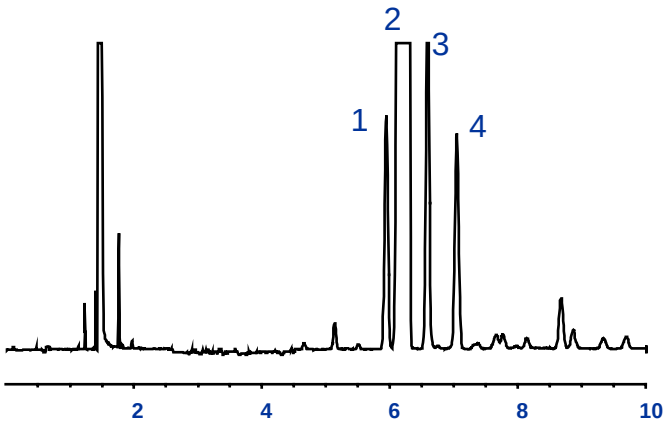
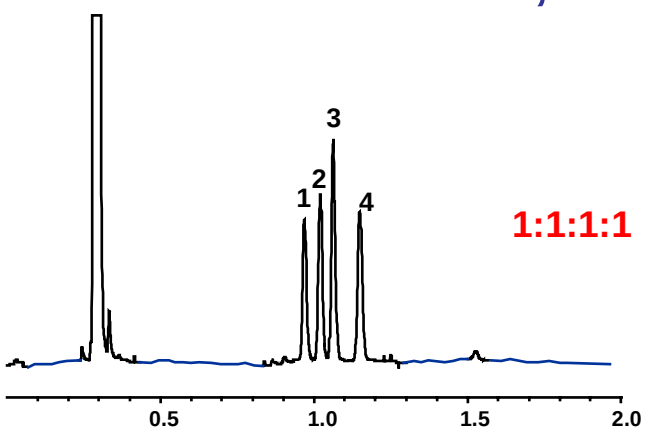


Is Dilution the Solution?

10 m x 0.1 mm, 0.2 µm



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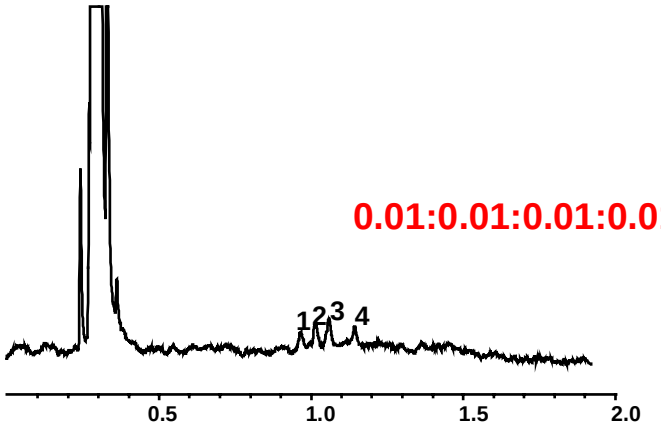
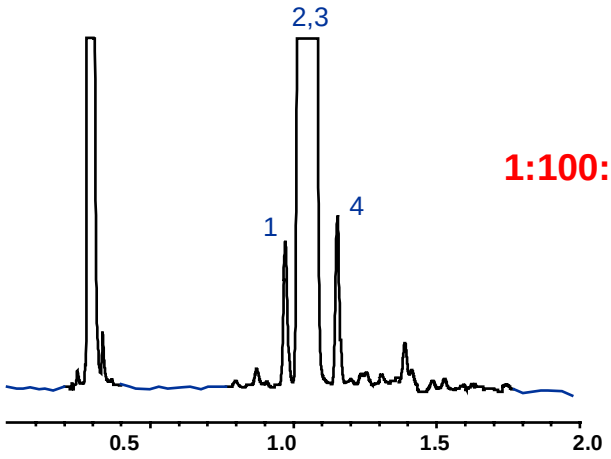


0.01:0.01:0.01:0.01



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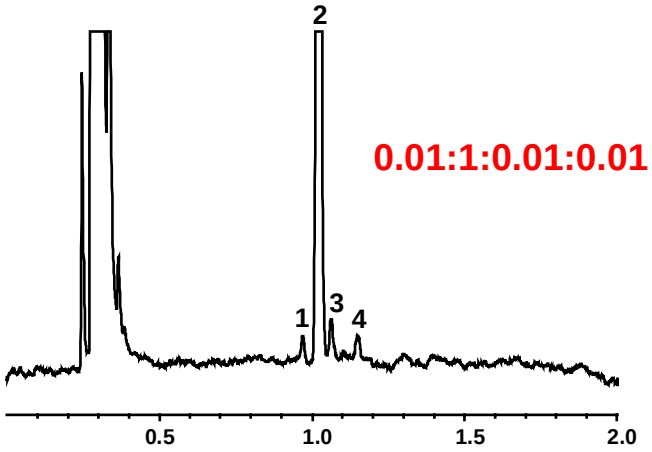

0.01:1:0.01:0.01

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Instrument Requirements

- High pressure capability
- Split Inlet
- Fast temperature ramping capability
- Fast detector scan or sample rate

Remember to ask the right questions

- **What information do you need from your analysis?**
- **Do you have more baseline than you need between your peaks?**
- **Do you need to resolve all of the components?**
- **Does your instrument have the necessary capabilities?**

Remember the Variables for Shortening Analysis Time

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

Wrap-up E-Seminar Questions

Thank you for attending Agilent e-Seminars. Our e-Seminar schedule is expanding every week. Please check our website frequently at:

www.agilent.com