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Introduction to Capillary GC

11:00 a.m. EST
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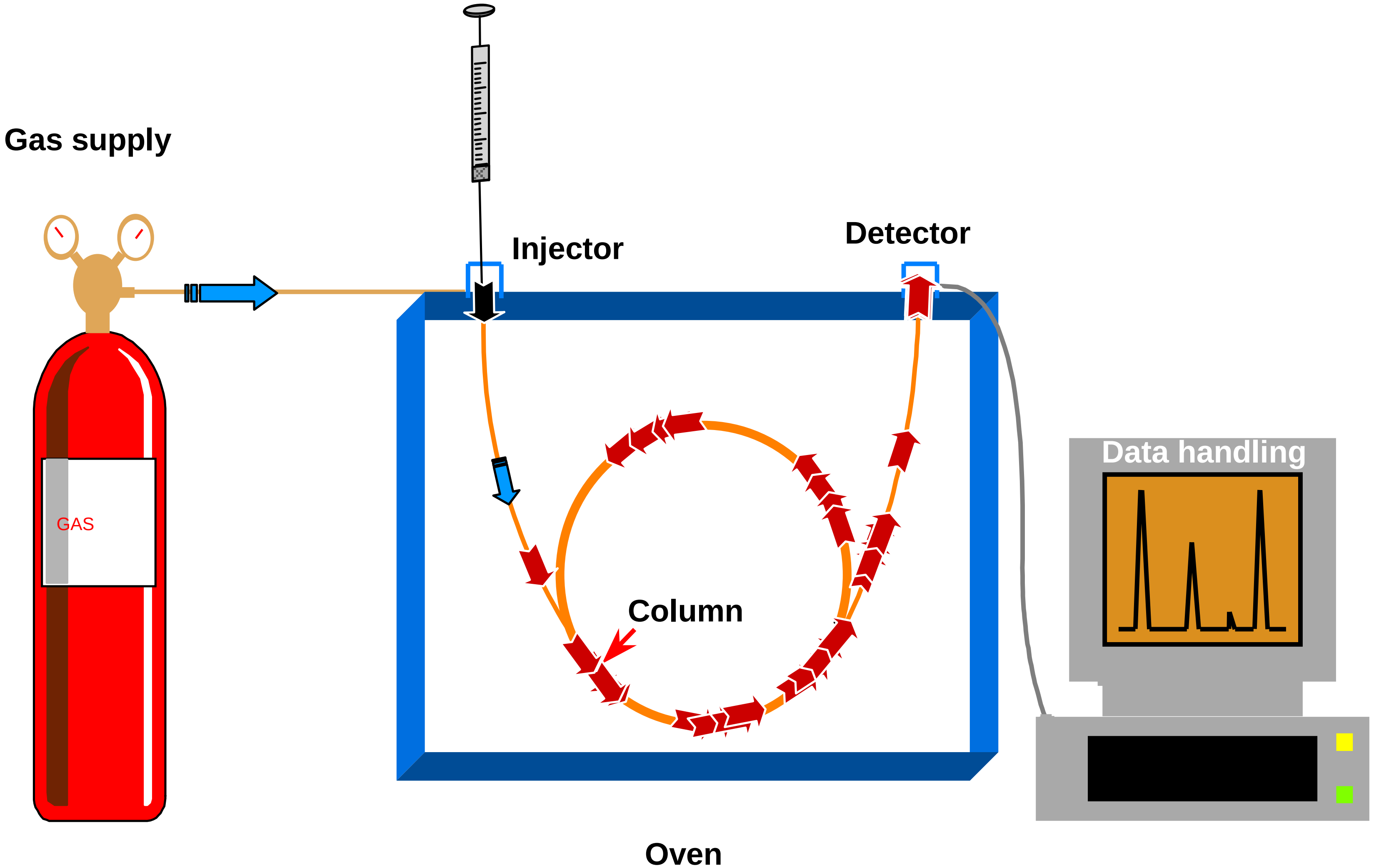
Introduction to Capillary GC

Typical GC System



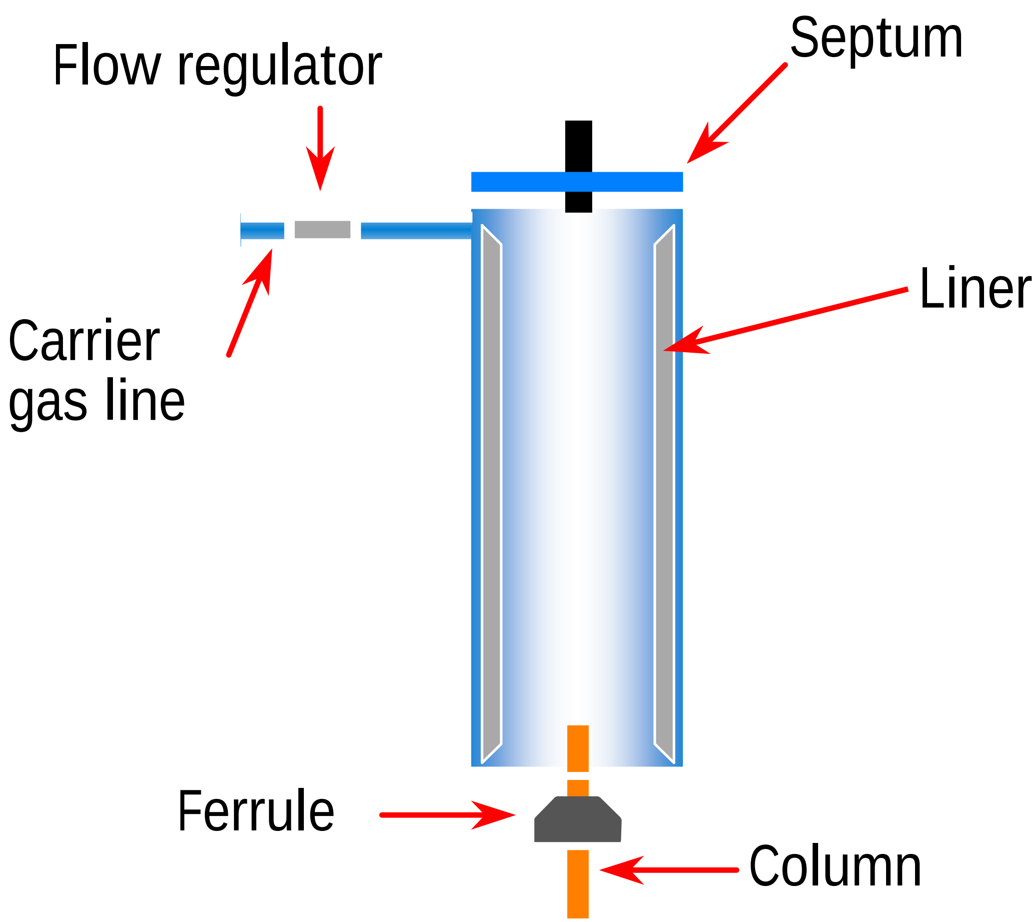
CARRIER GAS

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

SAMPLE INJECTION
Goals

- Introduce sample into the column
- Reproducible
- No efficiency losses
- Representative of sample

VAPORIZATION INJECTOR
General Design



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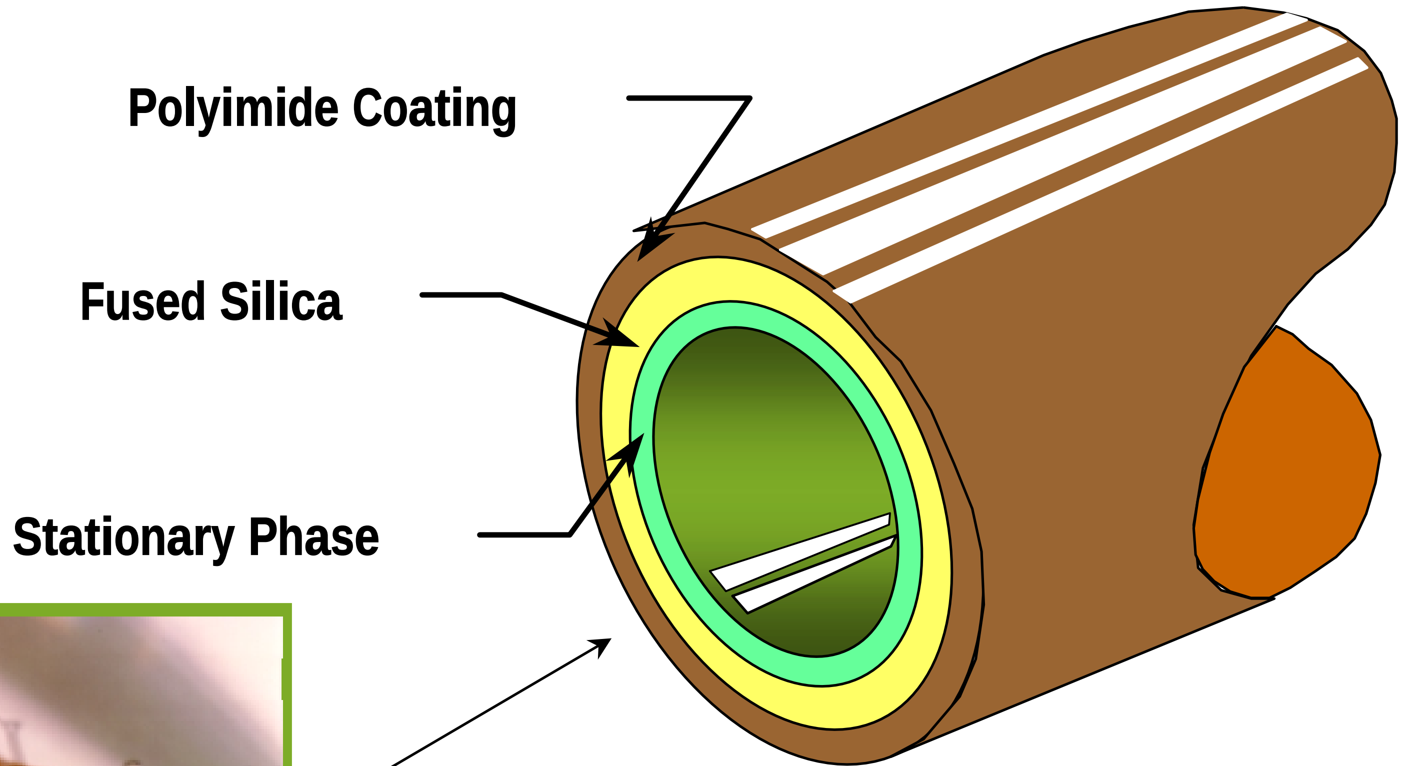
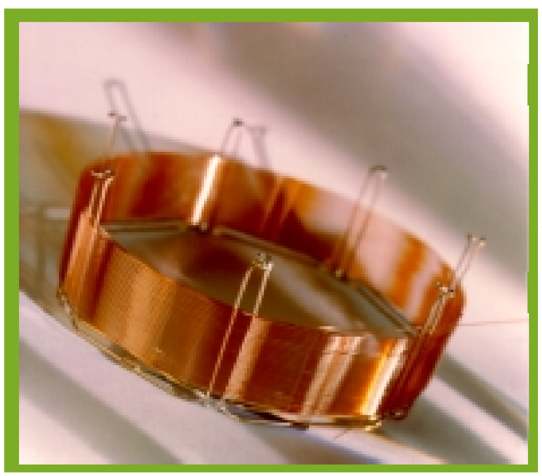
Typical Capillary Column





Expanded view of capillary tubing

DETECTORS
Purpose

- Responds to some property of the solutes
- Converts the interaction into a signal
- Immediate
- Predictable

DATA HANDLING

- Converts the detector signal into a chromatogram
    - Integrator
    - Software Program

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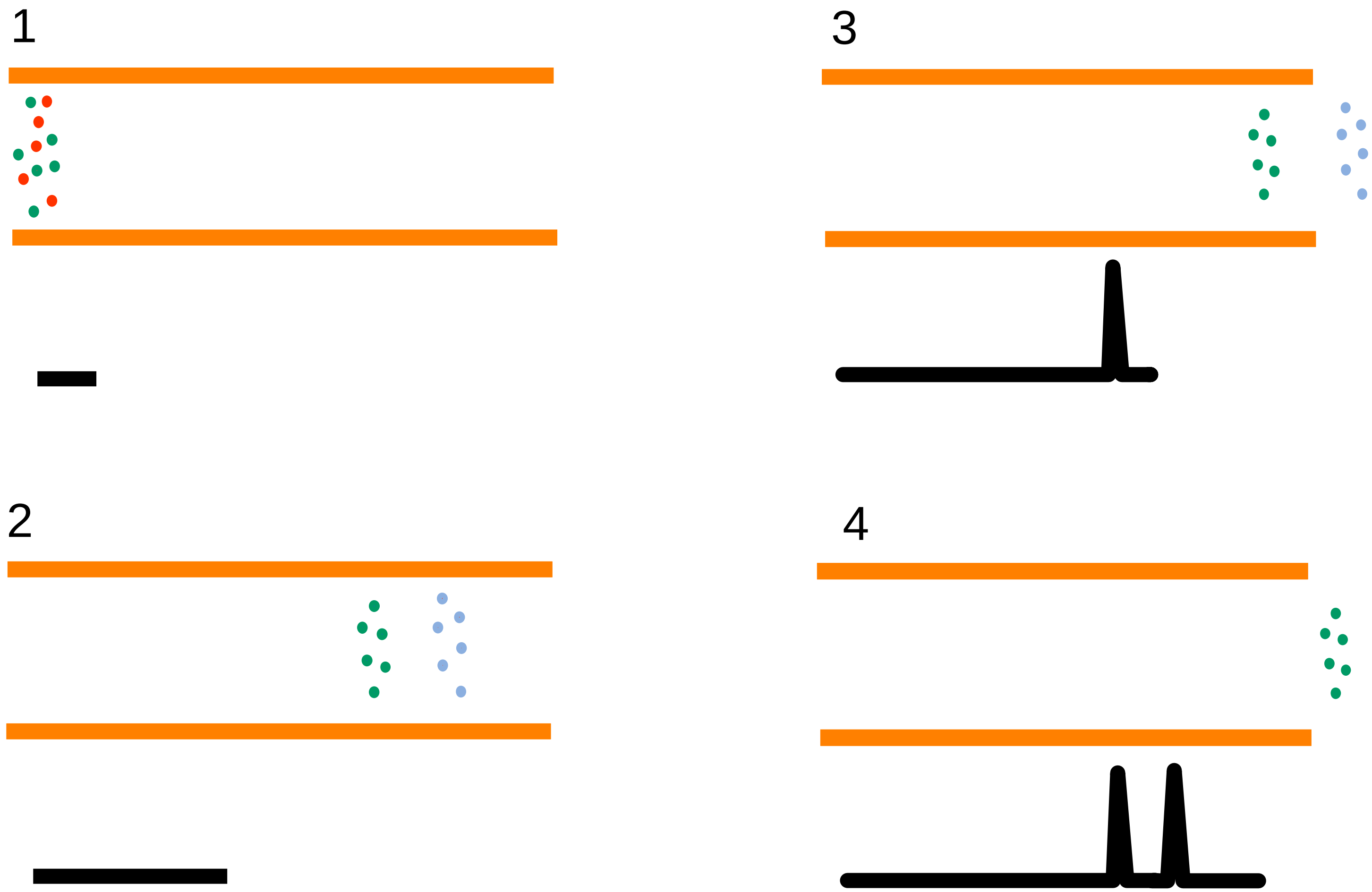
SEPARATION PROCESS



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COMPOUND REQUIREMENTS FOR GC

- Only 10-20% of all compounds are suitable for GC analysis
- The compounds must have:
  - Sufficient volatility
  - Thermal stability

TWO PHASES

Solute molecules distribute into the two phases

DISTRIBUTION CONSTANT (K_C)

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

K_C formerly written as K_D

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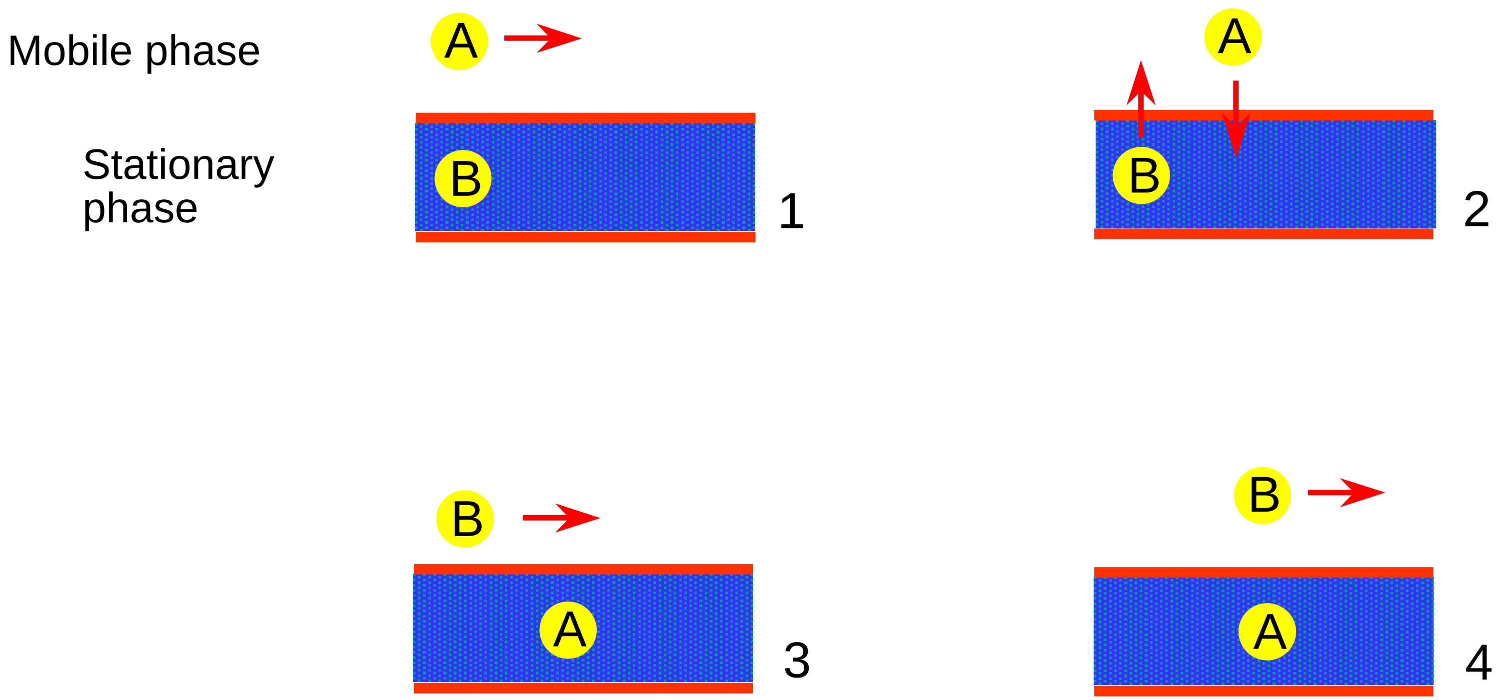
SOLUTE LOCATION

- In stationary phase = Not moving down the column
- In mobile phase = Moving down the column

SEPARATION PROCESS
Movement Down the Column



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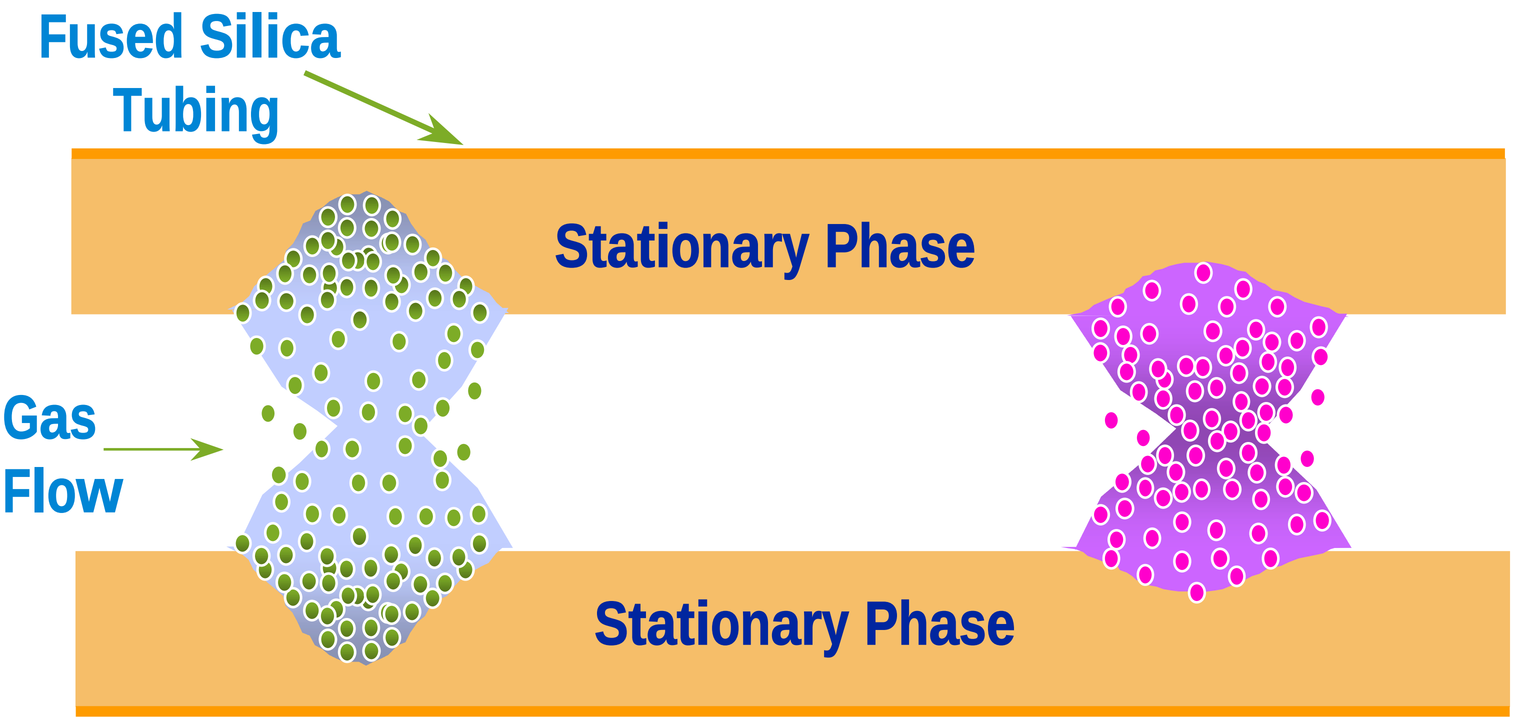
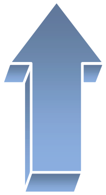
Break

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

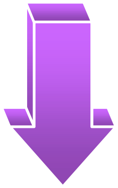
KC AND RETENTION



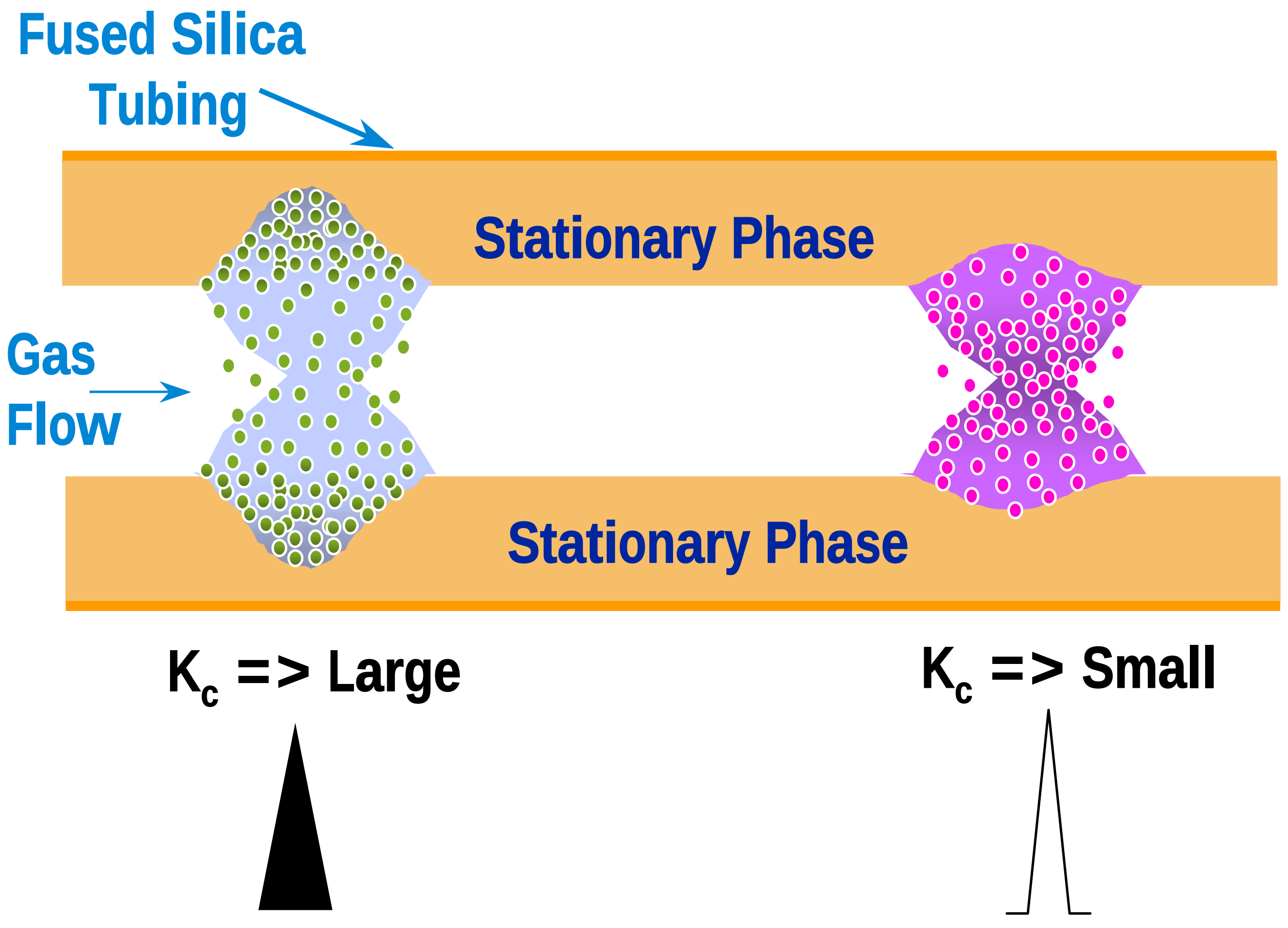
K_c => Large

retention 

K_c => Small

retention 

KC AND PEAK WIDTH
Time of Elution



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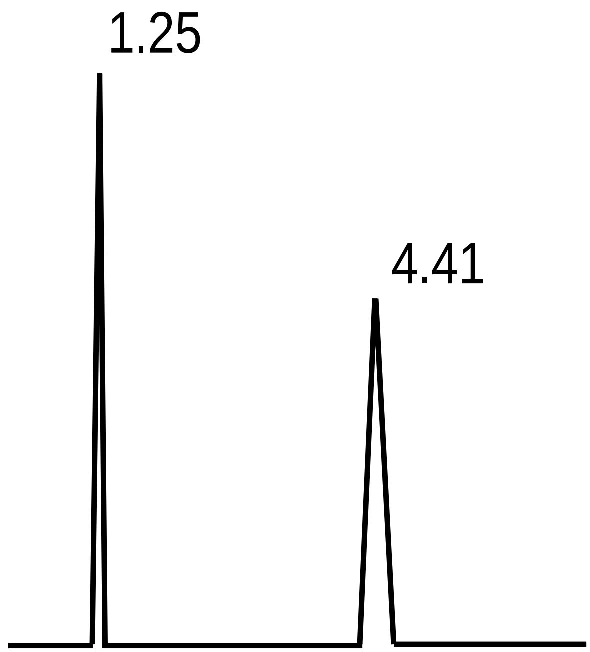
THREE PARAMETERS THAT AFFECT KC

- Solute:
  different solubilities in a stationary phase
- Stationary phase:
  different solubilities of a solute
- Temperature:
  K_C decreases as temperature increases

RETENTION TIME
tr



•Time for a solute to travel through the column

ADJUSTED RETENTION TIME
tr'

- Actual time the solute spends in the stationary phase
- $$t_r' = t_r - t_m$$

t_r = retention time
t_m = retention time of a non-retained solute

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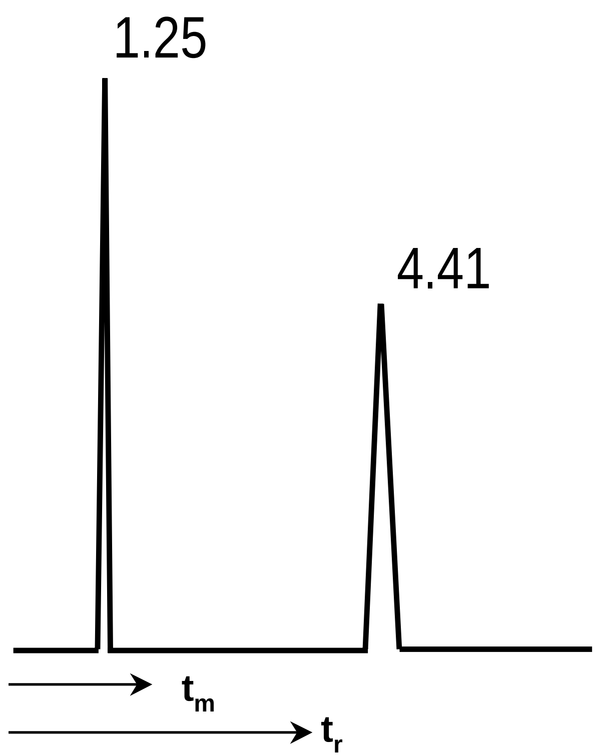
ADJUSTED RETENTION TIME
tr



t'_r = tr - tm

t'_r = 4.41 - 1.25

t'_r = 3.16 min = time spent in stationary phase

TIME IN THE MOBILE PHASE

- All solutes spend the same amount of time in the mobile phase

RETENTION FACTOR (k)

- Ratio of the time the solute spends in the stationary and mobile phases

$$k = \frac{t_r - t_m}{t_m}$$

t_r = retention time
t_m = retention time of non-retained compound
Formerly called partition ratio; k'

RETENTION FACTOR (k)

- Relative retention
- Linear
- Factors out carrier gas influence

PHASE RATIO
(β)

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

r = radius (µm)

d_f = film thickness (µm)

DISTRIBUTION CONSTANT (Kc)

$$K_c = k\beta$$

$$k = \frac{t_r'}{t_m}$$

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

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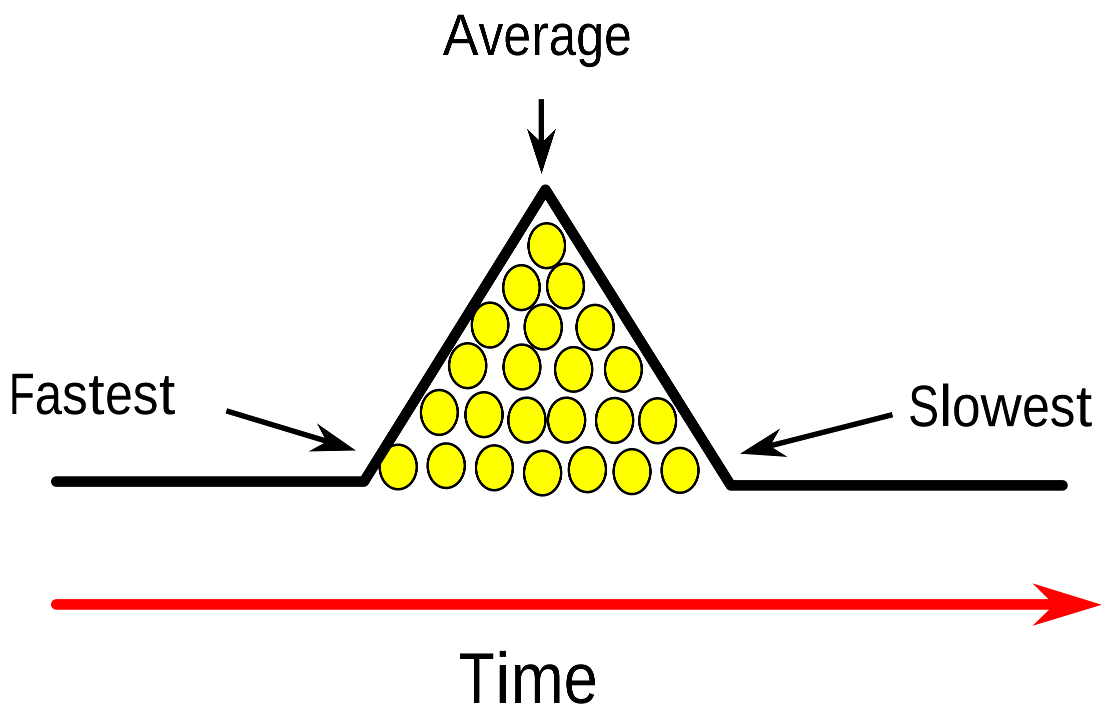
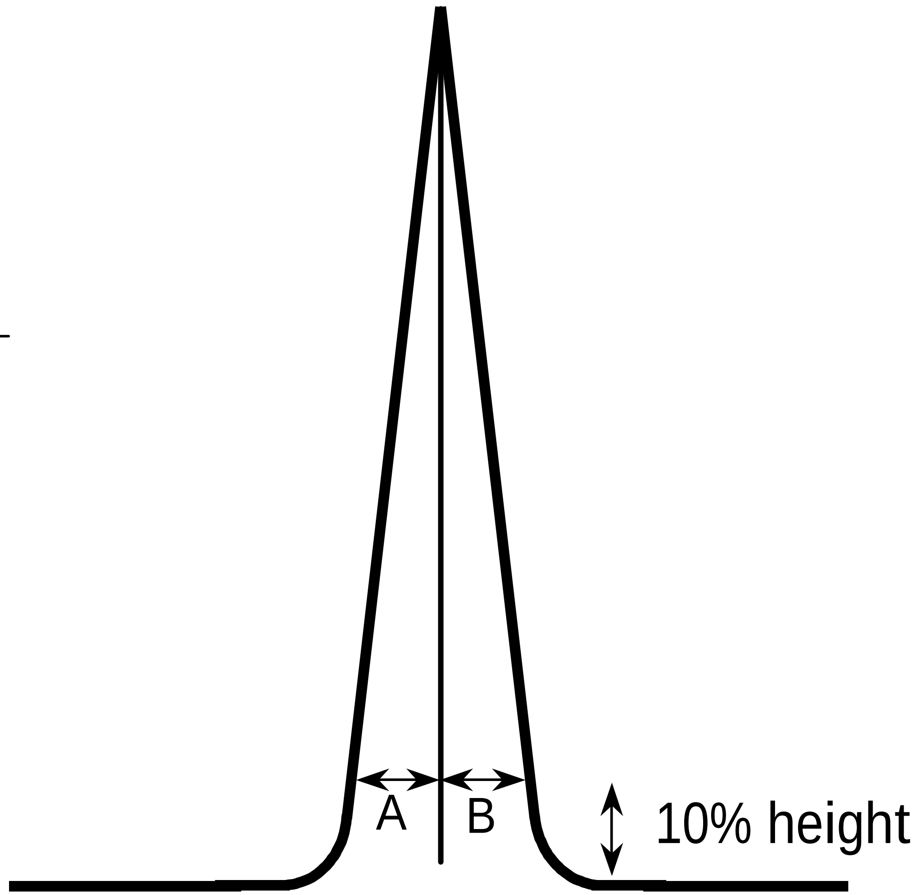
RANGE OF RETENTION



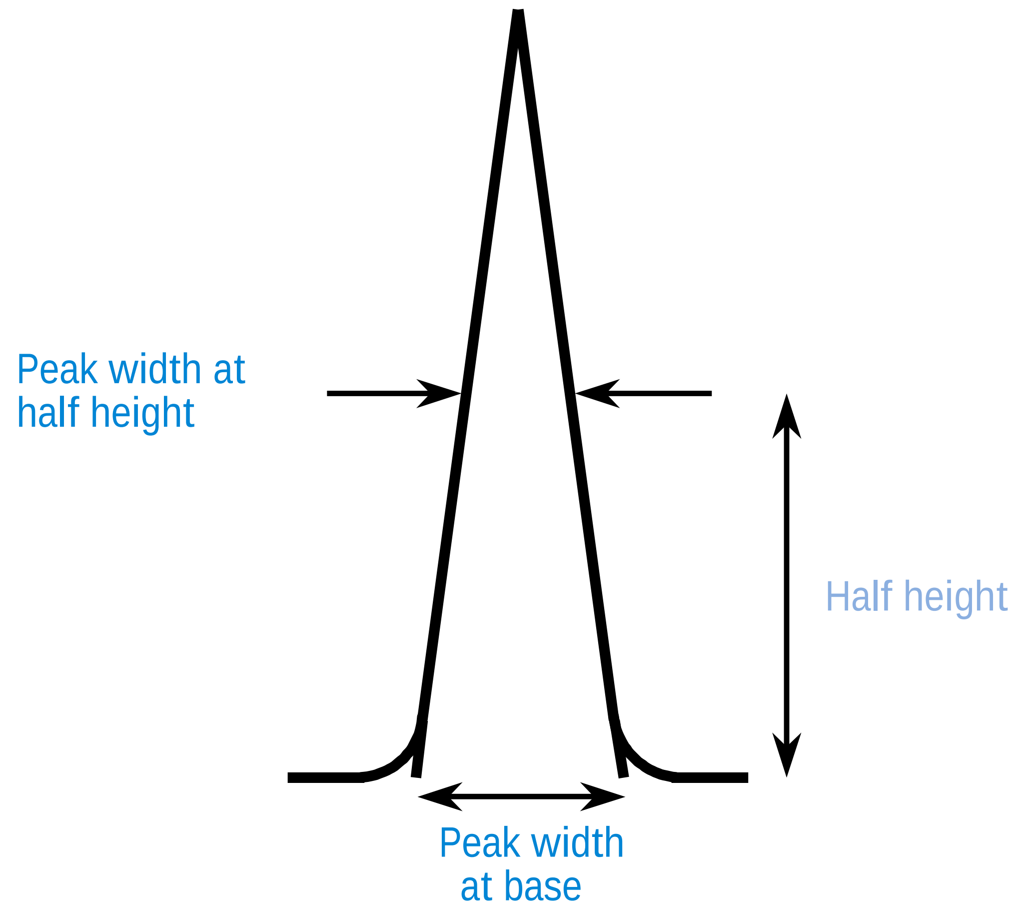
PEAK SYMMETRY

$$\text{Symmetry} = \frac{A}{B}$$

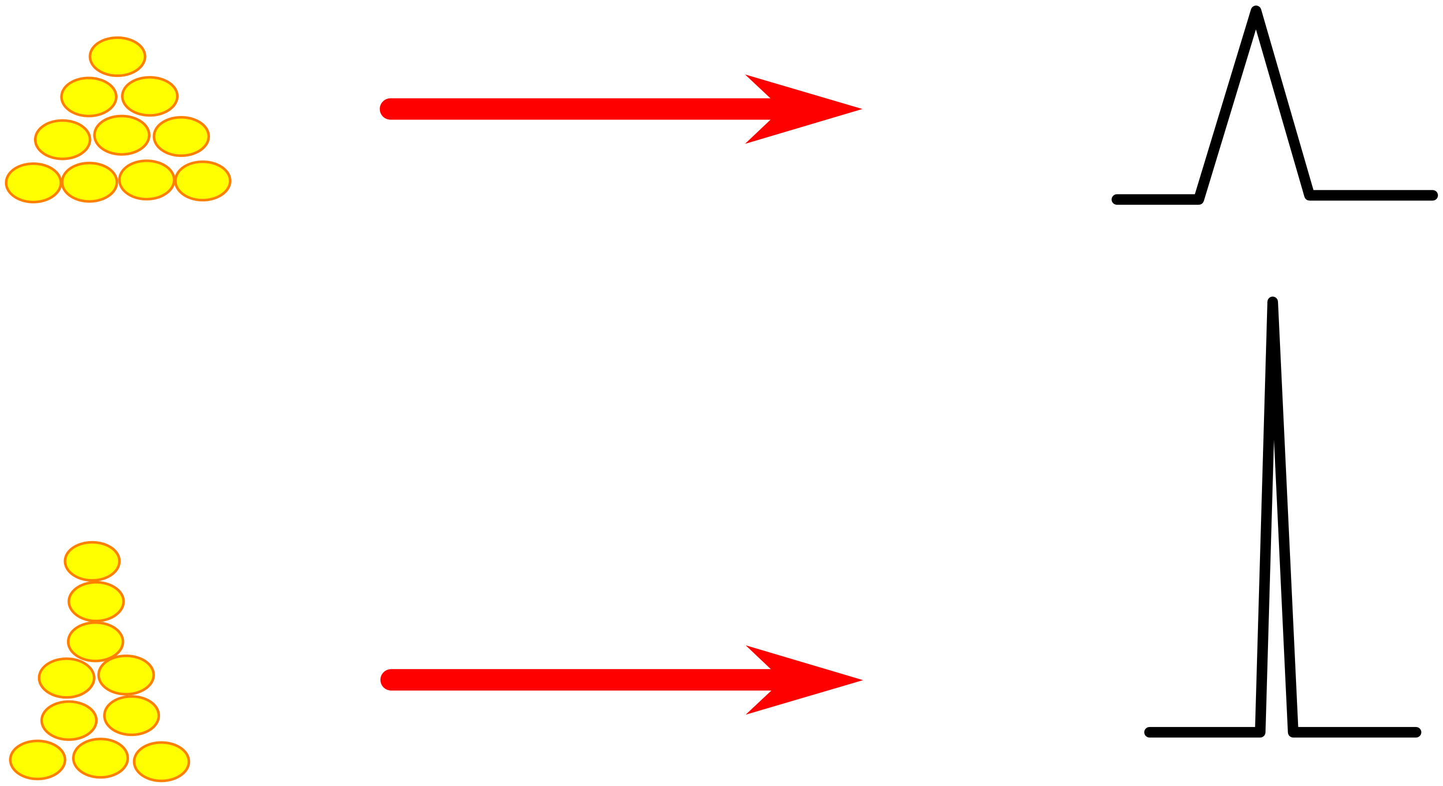


Tailing : Symmetry <1
Fronting : Symmetry >1

PEAK WIDTH



PEAK WIDTH



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EFFICIENCY
Theoretical Plates (N)

- Large number implies a better column
- Often a measure of column quality
- Relationship between retention time and width

THEORETICAL PLATES (N)

$$N = 5.545 \left(\frac{t_r}{W_h} \right)^2$$

t_r = retention time
W_h = peak width at half height (time)

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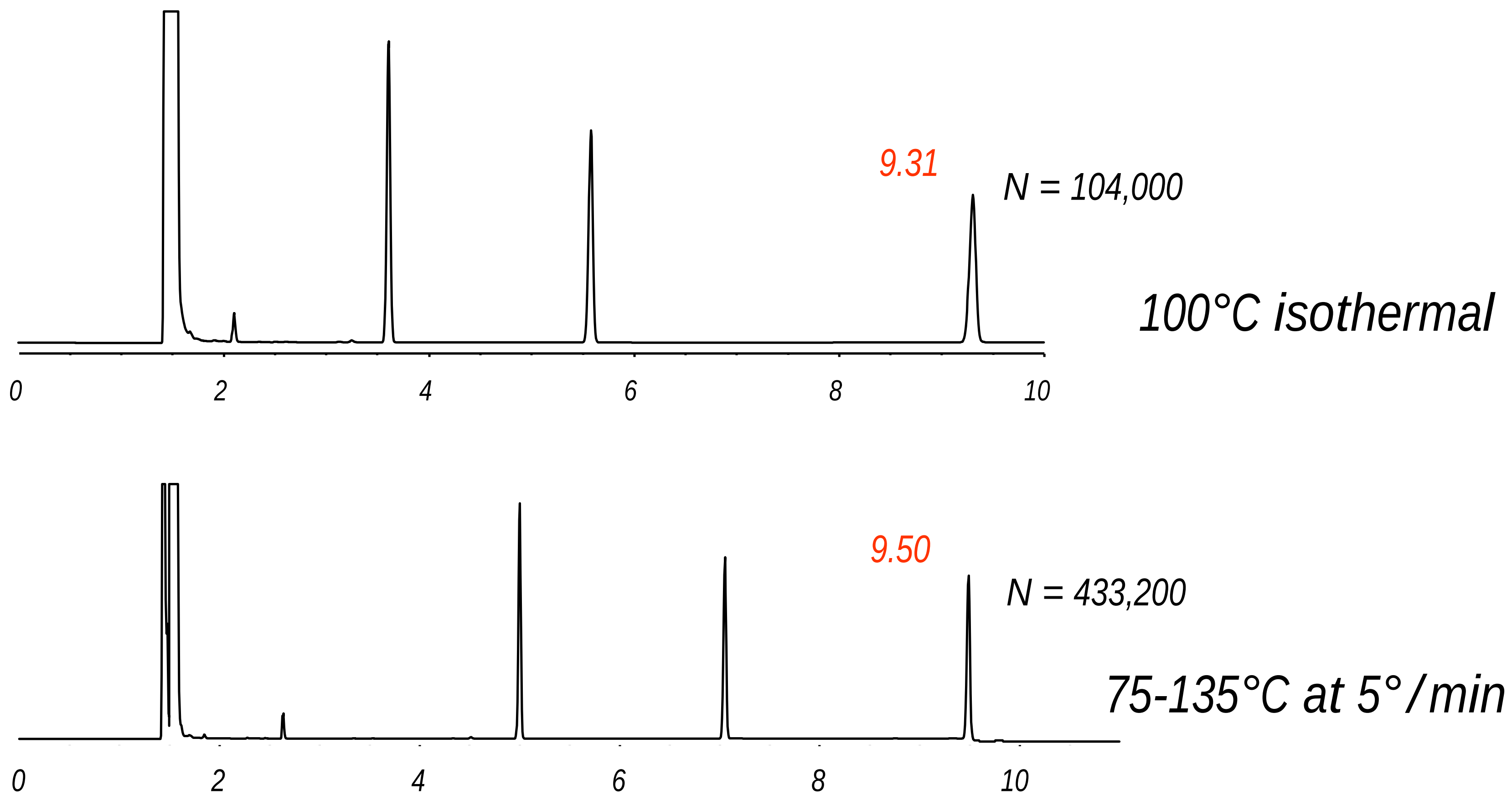
EFFICIENCY MEASUREMENT
Cautions

- Actually, measure of the GC system
- Condition dependent
- Use a peak with k>5

ISOTHERMAL VS. TEMPERATURE PROGRAMMING
Efficiency



DB-1, 30 m x 0.25 mm ID, 0.25 um
He at 37 cm/sec
C10, C11, C12

SEPARATION VS. RESOLUTION

- Separation: time between peaks

- Resolution: time between the peaks while considering peak widths

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SEPARATION FACTOR (α)

$$\alpha = \frac{k_2}{k_1}$$

•co-elution: $\alpha = 1$

k_2 = retention factor of 2nd peak
k_1 = retention factor of

RESOLUTION (Rs)

$$R_s = 1.18 \left(\frac{t_{r2} - t_{r1}}{W_{h1} + W_{h2}} \right)$$

t_r = retention time
W_h = peak width at half height (time)

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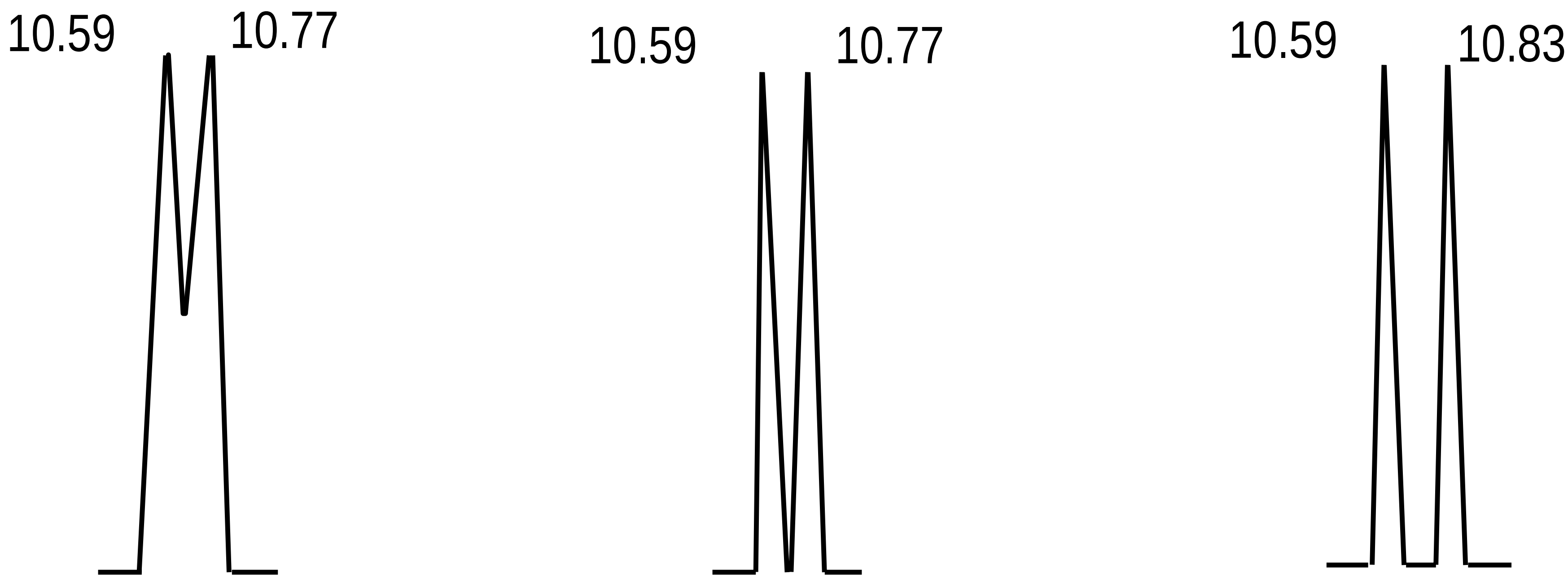
RESOLUTION
Baseline Resolution: Rs = 1.5



W_h = 0.105	W_h = 0.059	W_h = 0.059
R = 0.84	R = 1.50	R = 2.40
% = 50	% = 100	% = 100

Resolution

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

N = Theoretical plates

k = Retention factor

α = Separation factor

INFLUENCING RESOLUTION
Variables

N: column dimensions, carrier gas

α: stationary phase, temperature

k: stationary phase, temperature,
column dimensions

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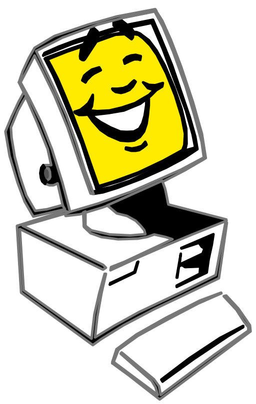

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...Instant Answers

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