Introduction to Capillary GC

GC Columns and Consumables

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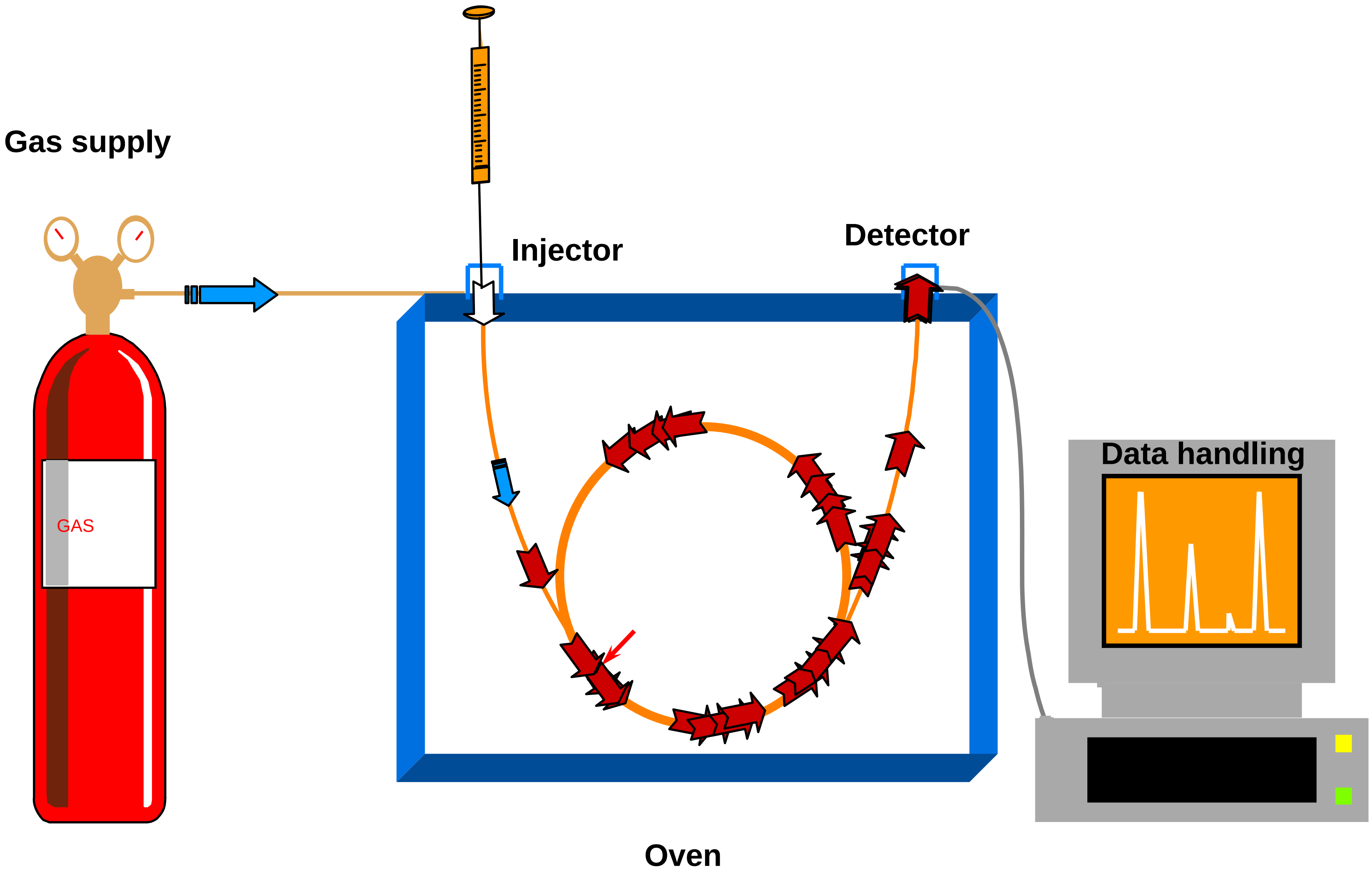
February 20, 2008

Introduction to Capillary GC

Typical GC System

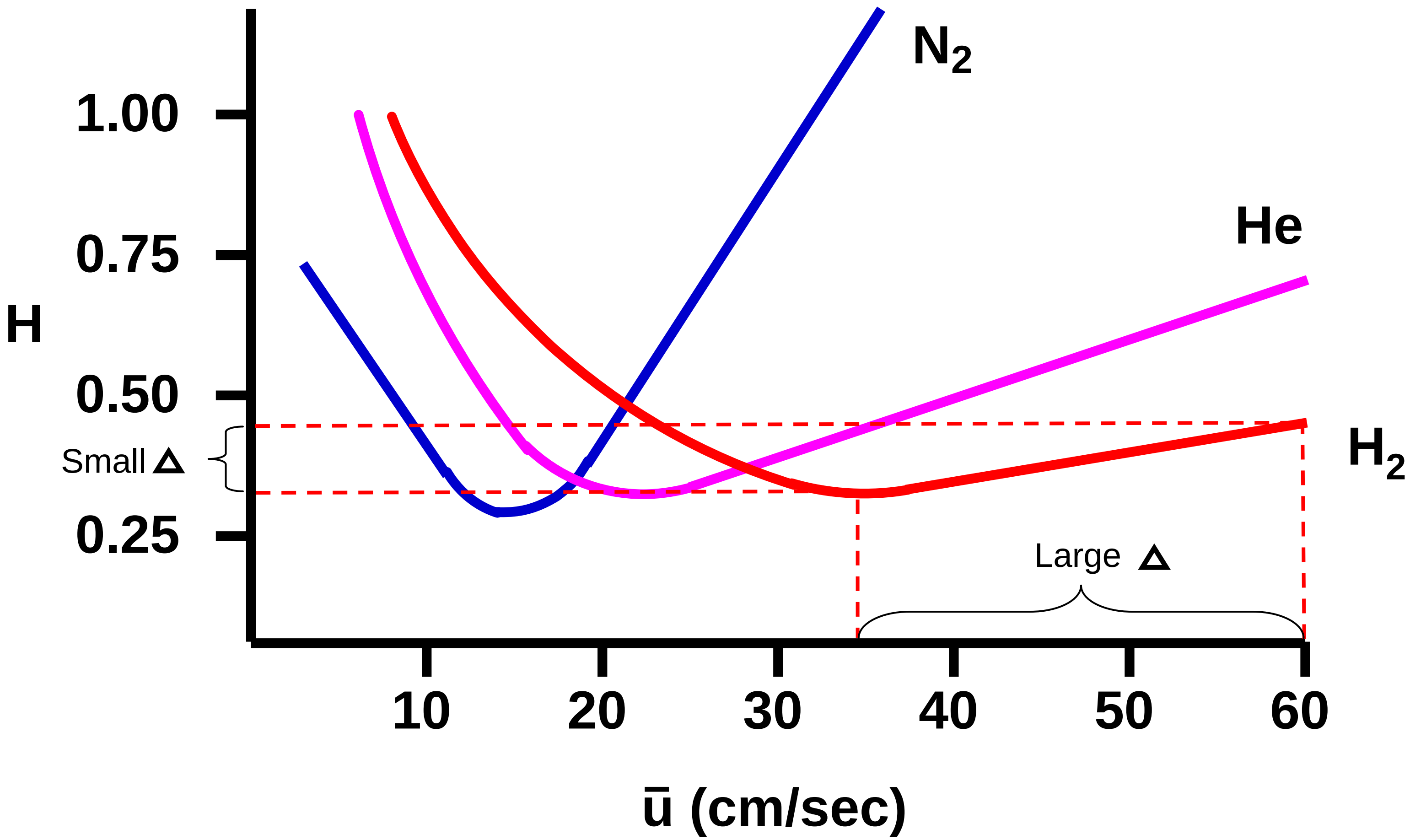


CARRIER GAS

Carries the solutes down the column

Selection and velocity influences efficiency and retention time

VAN DEEMTER CURVES



SAMPLE INJECTION

Goals:

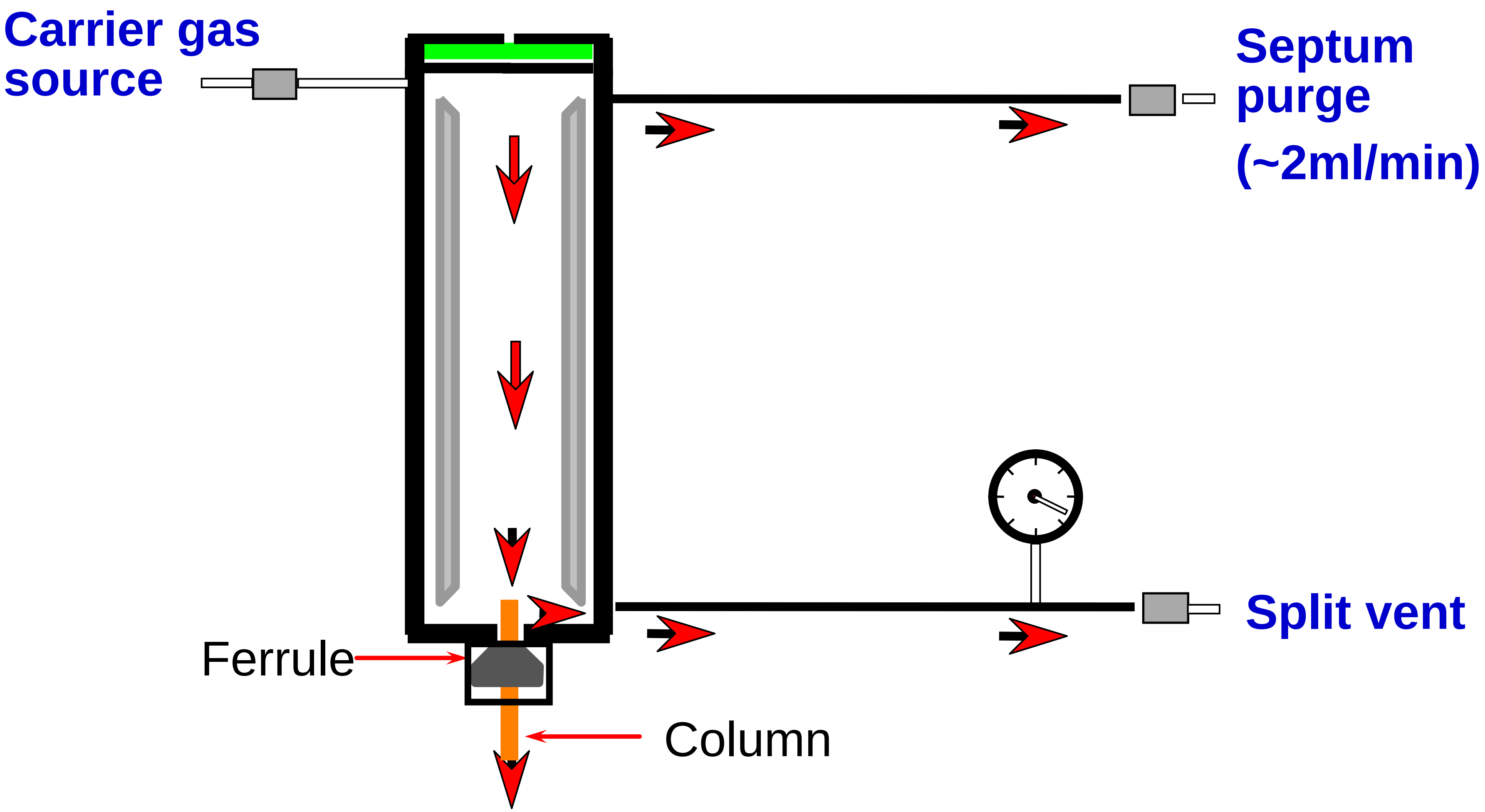
Introduce sample into the column

Reproducible

No efficiency losses

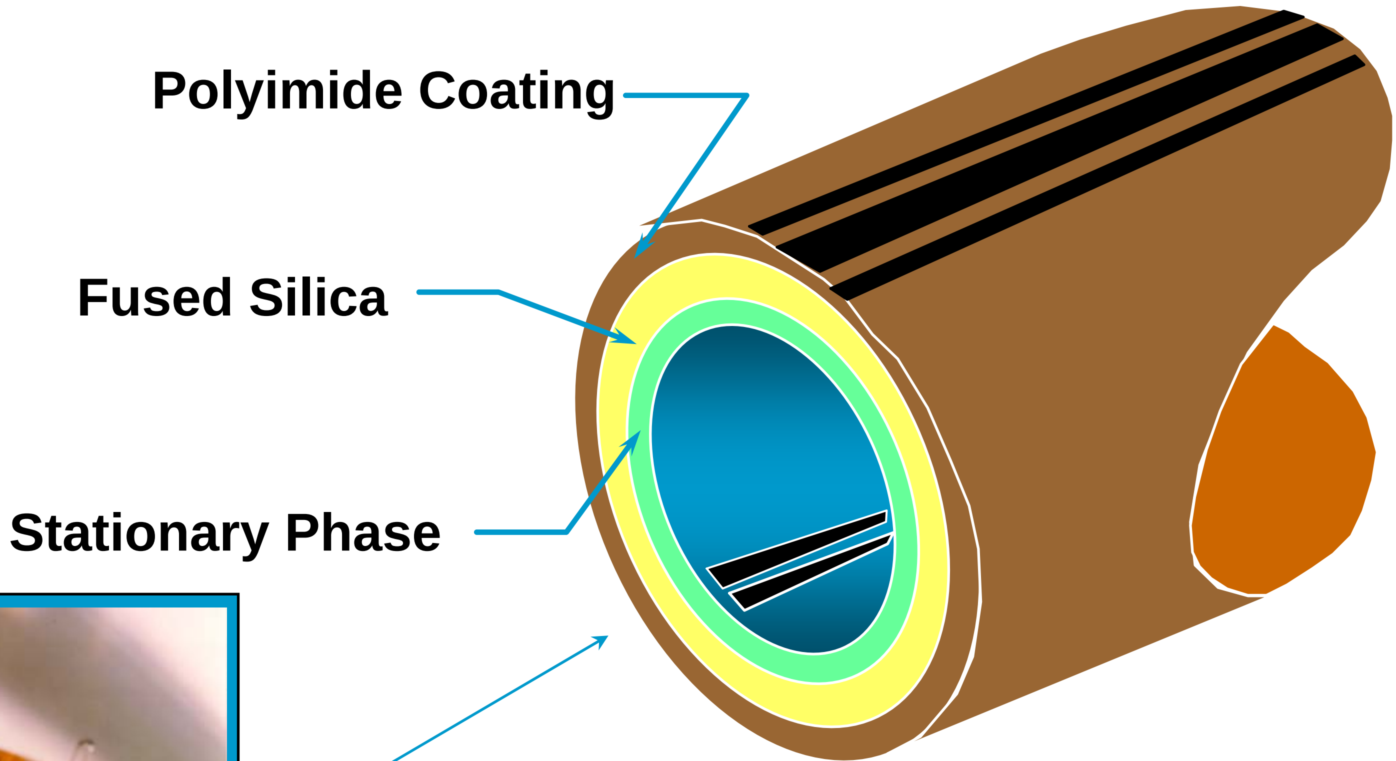
Representative of sample

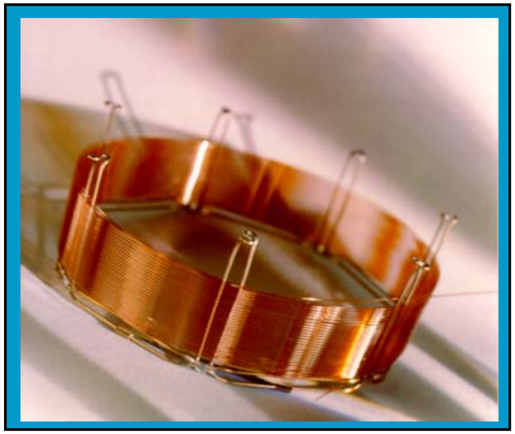
SPLIT/SPLITLESS INJECTOR



Flow through injector = Column flow + Split Vent Flow

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**

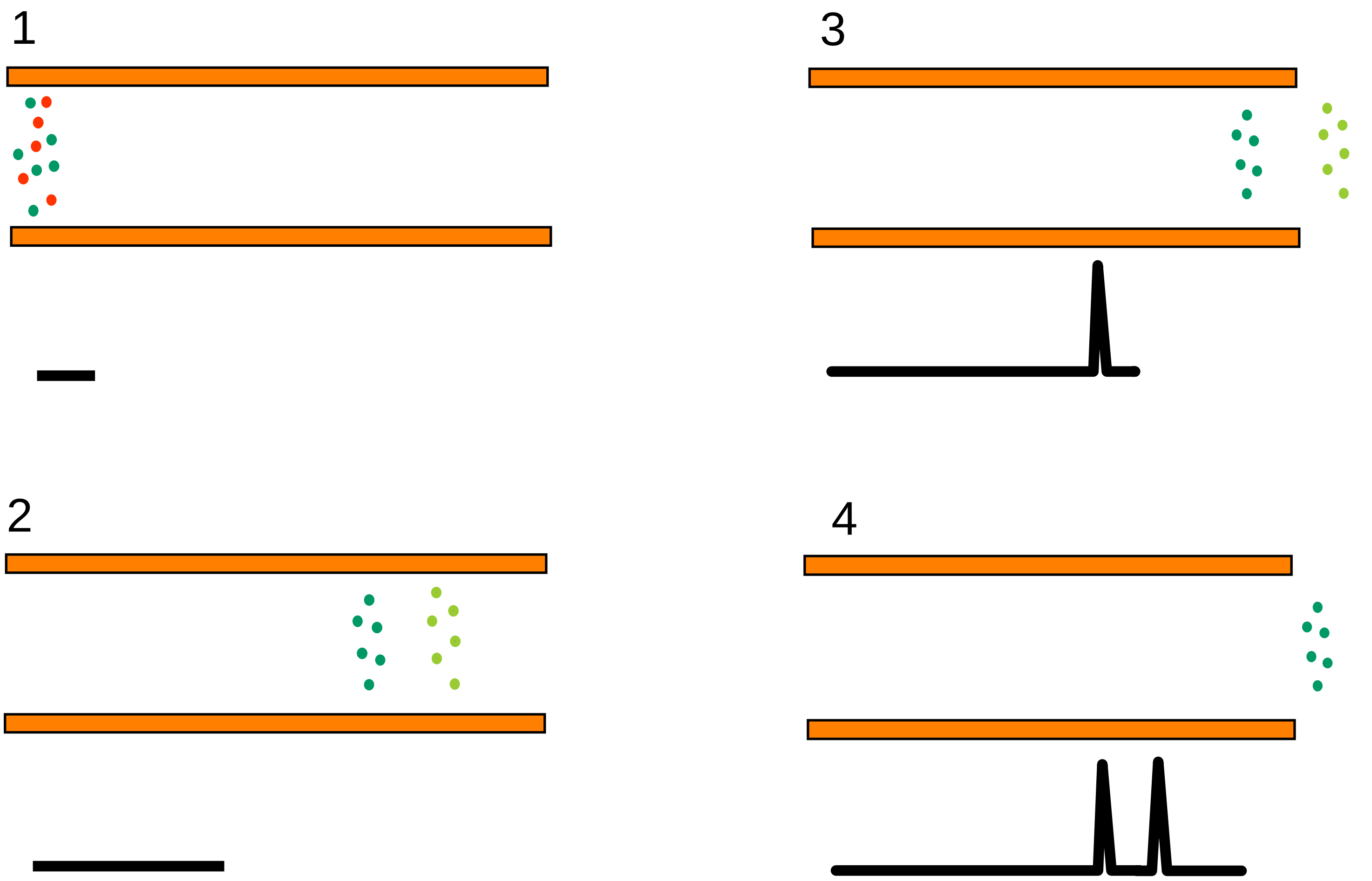
COMPOUND REQUIREMENTS FOR GC

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

The compounds must have:

- ✓ Sufficient volatility
- ✓ Thermal stability

SEPARATION PROCESS



TWO PHASES

Solute molecules distribute into the two phases

DISTRIBUTION CONSTANT (K_C)

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

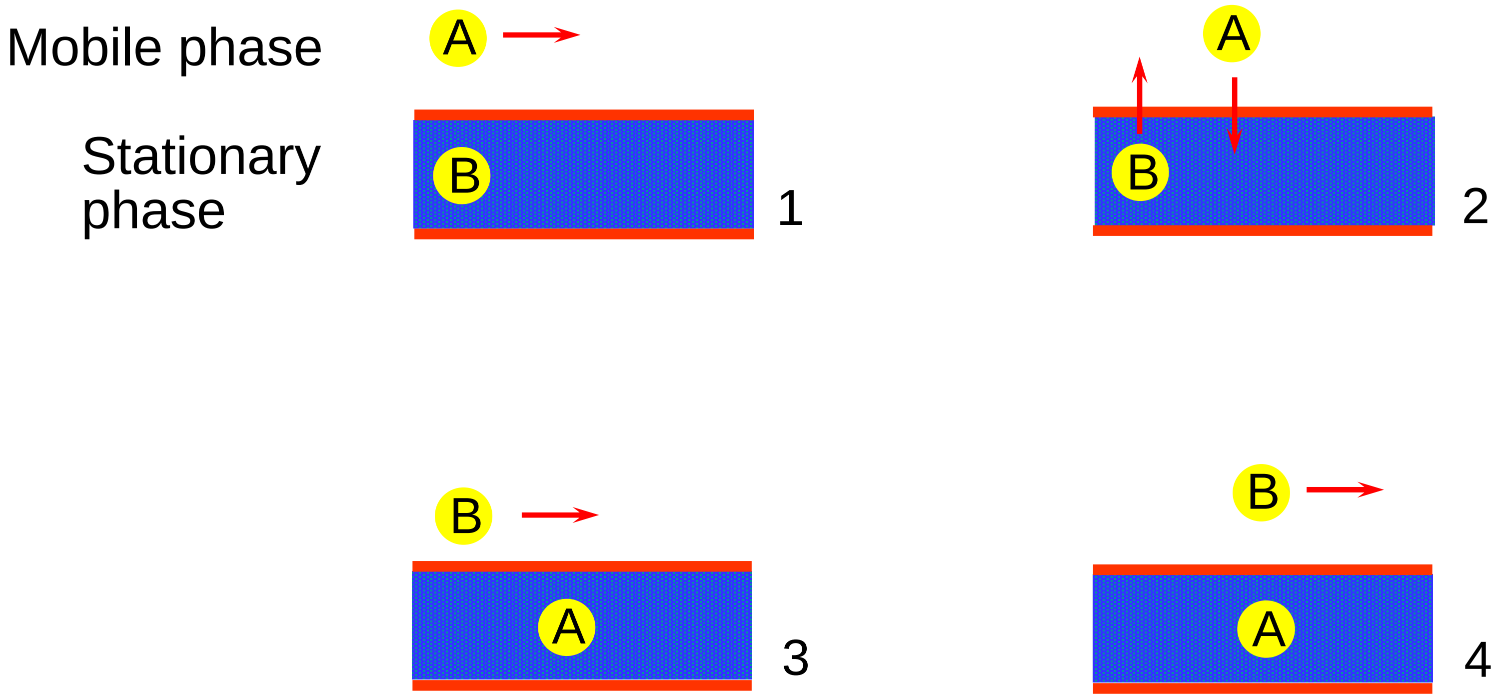
K_C formerly written as K_D

SOLUTE LOCATION

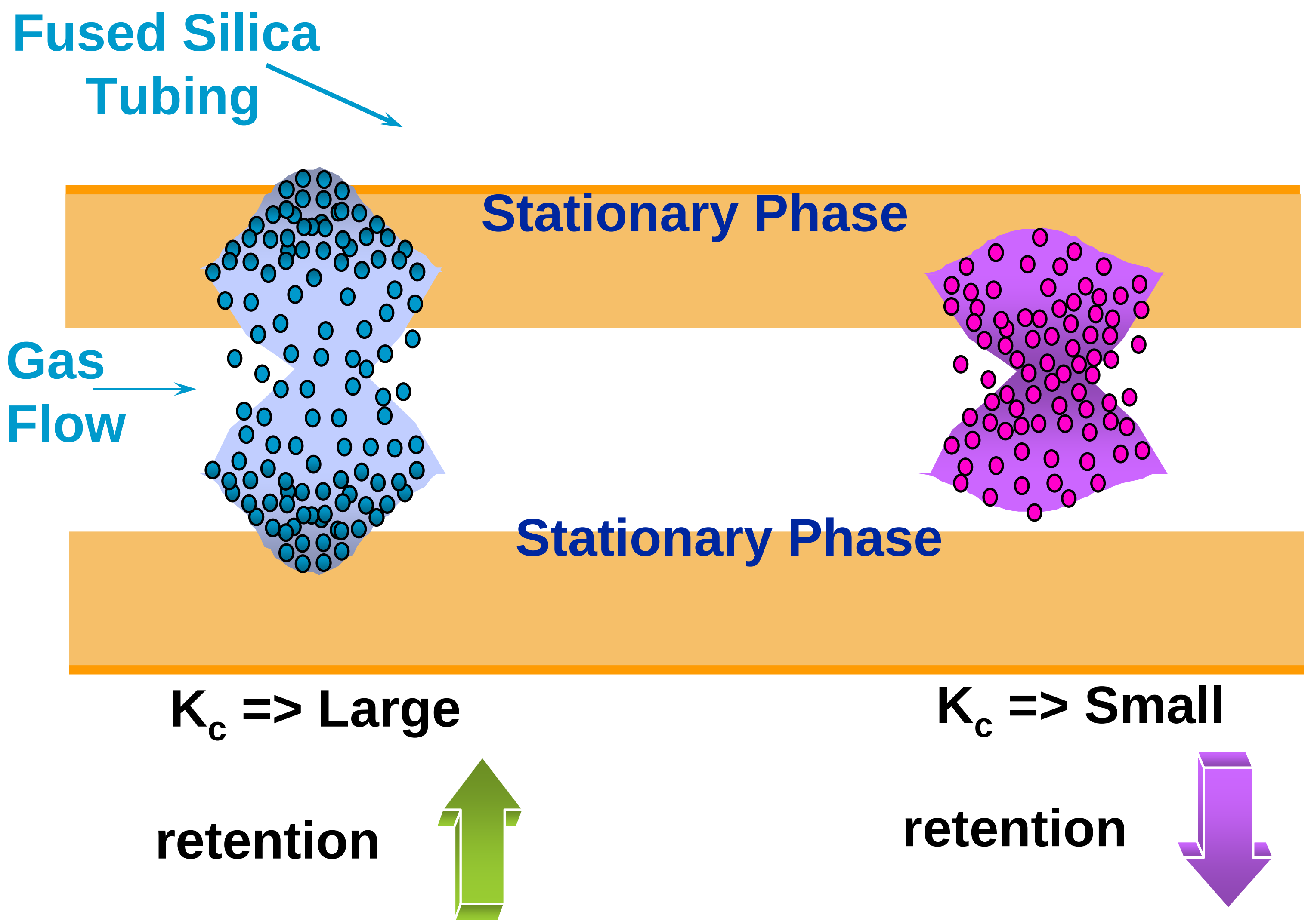
In stationary phase = Not moving down the column

In mobile phase = Moving down the column

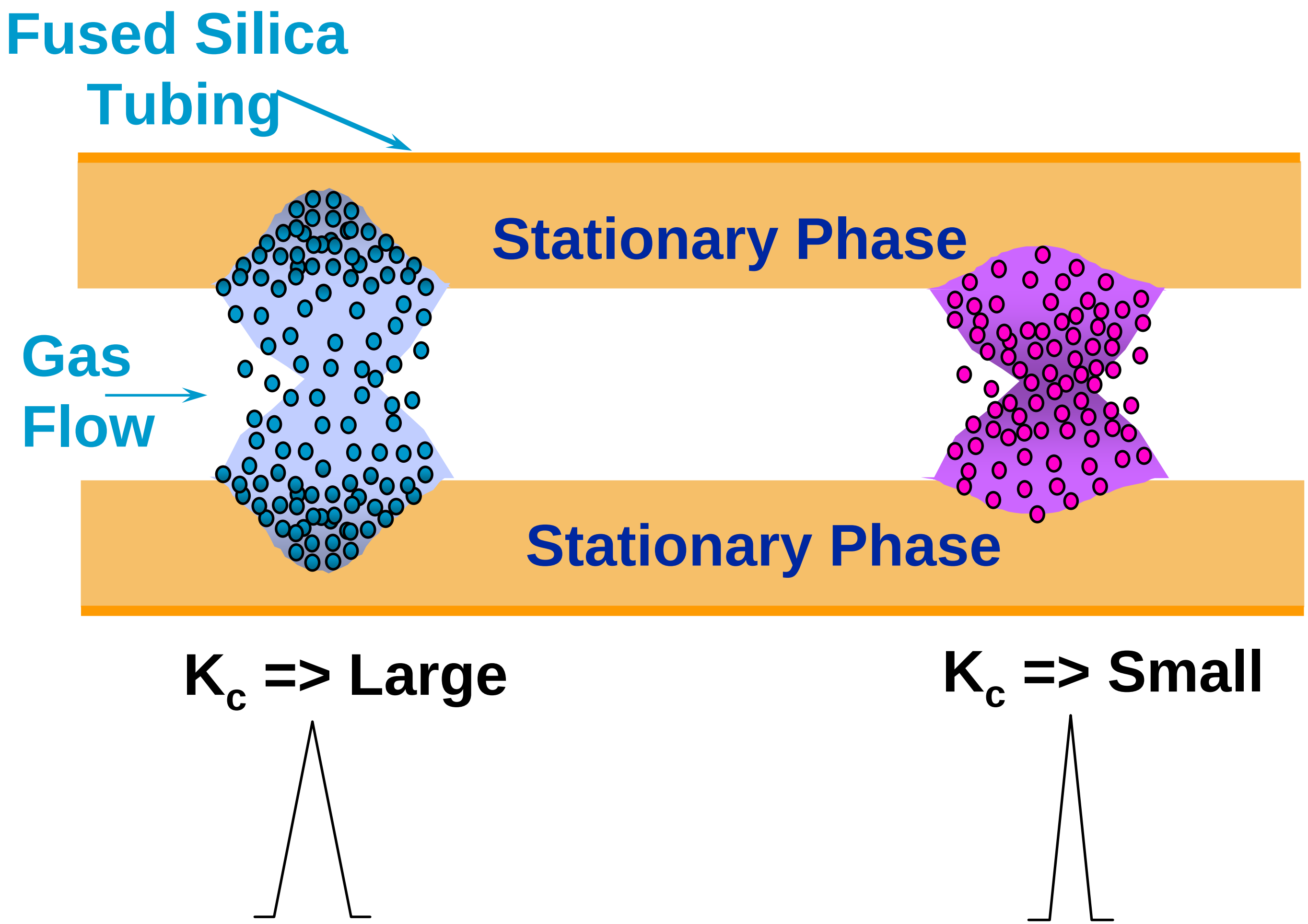
SEPARATION PROCESS
Movement Down the Column



KC AND RETENTION



KC AND PEAK WIDTH
Time of Elution



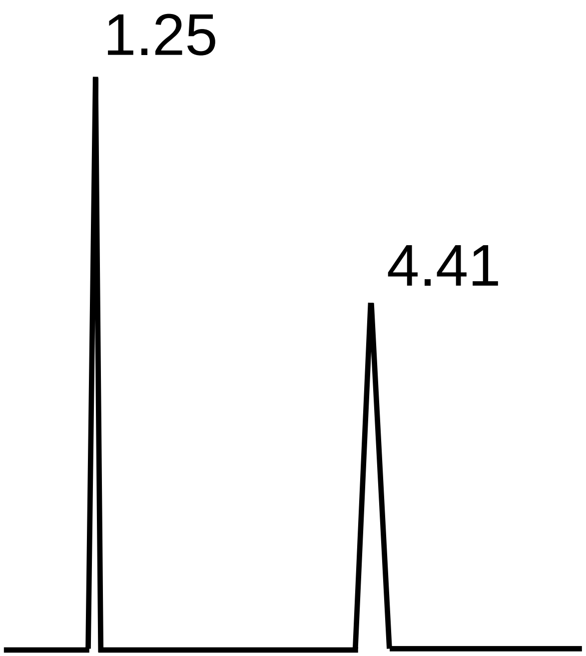
THREE PARAMETERS THAT AFFECT KC

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

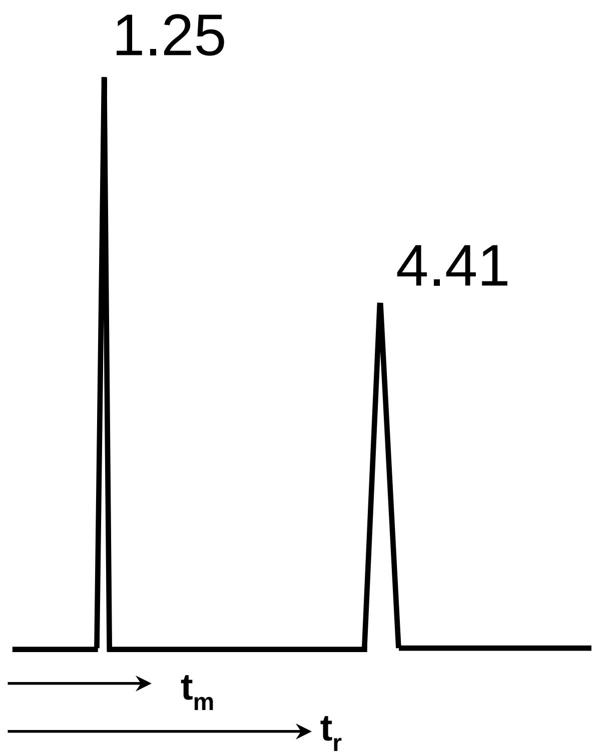
ADJUSTED RETENTION TIME
tr'

Actual time the solute spends in the stationary phase

$$tr' = tr - tm$$

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

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)

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

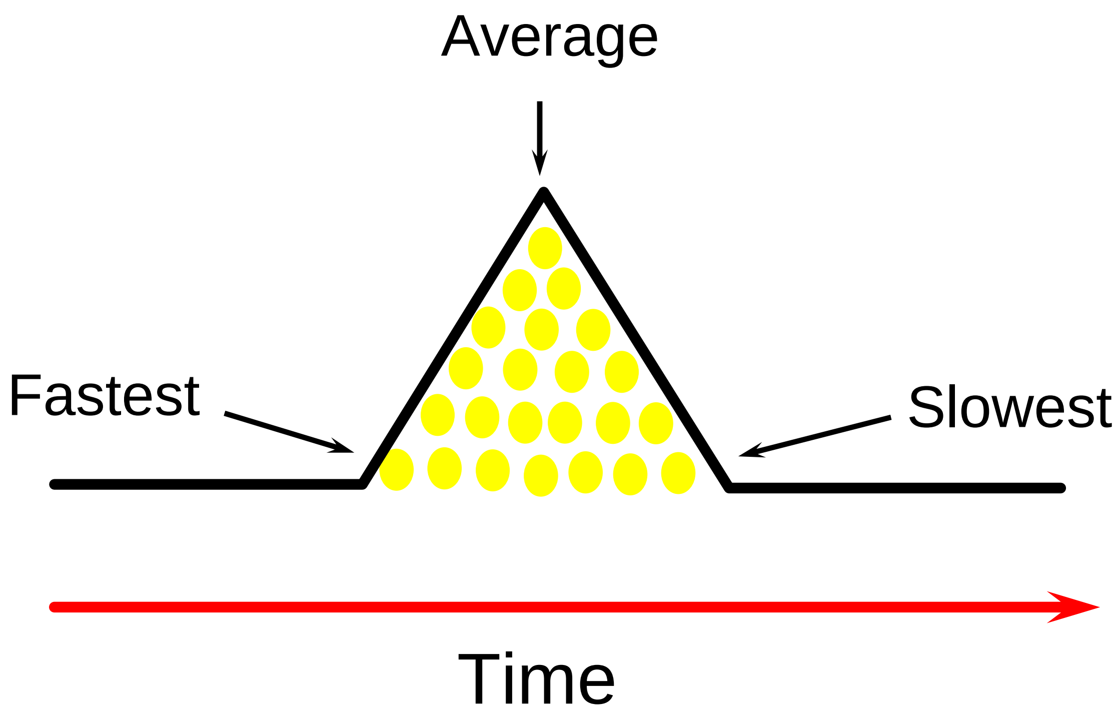
r = radius (µm)
d_f = film thickness (µm)

DISTRIBUTION CONSTANT (Kc)

$$K_c = k\beta$$

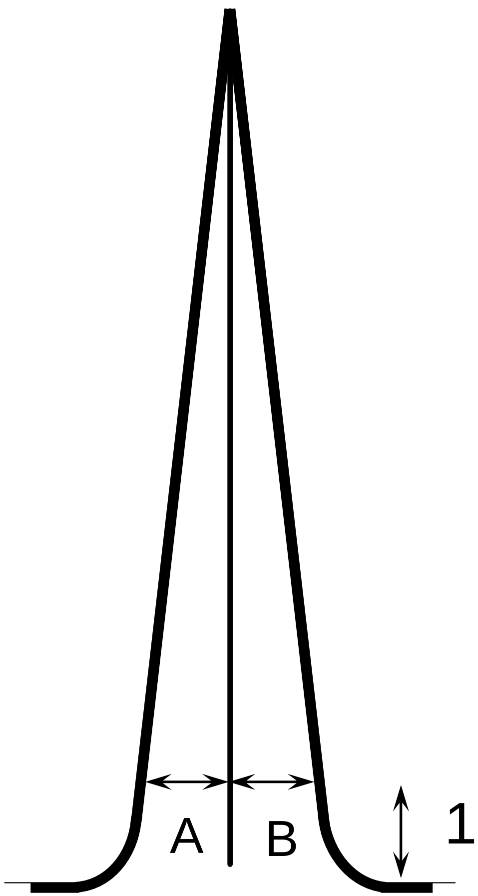
$$k = \frac{t_r'}{t_m} \qquad \beta = \frac{r}{2d_f}$$

RANGE OF RETENTION



PEAK SYMMETRY

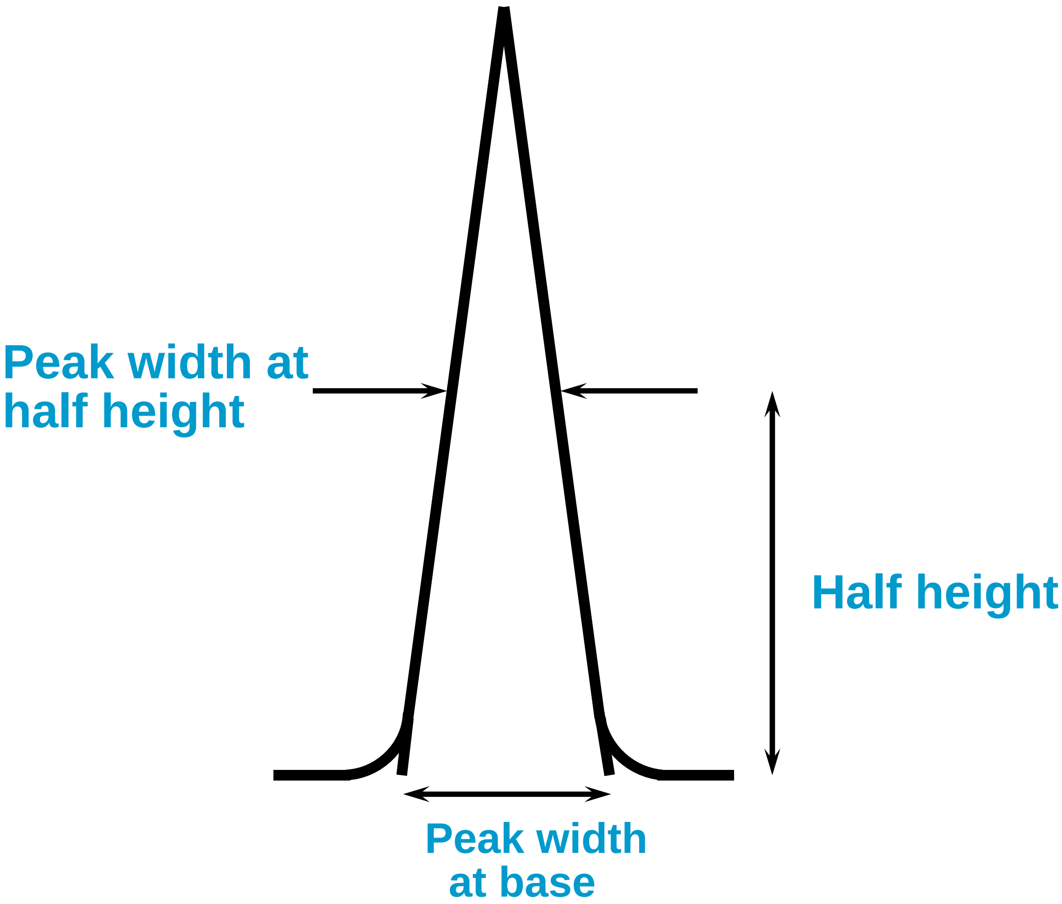
$$\text{Symmetry} = \frac{A}{B}$$



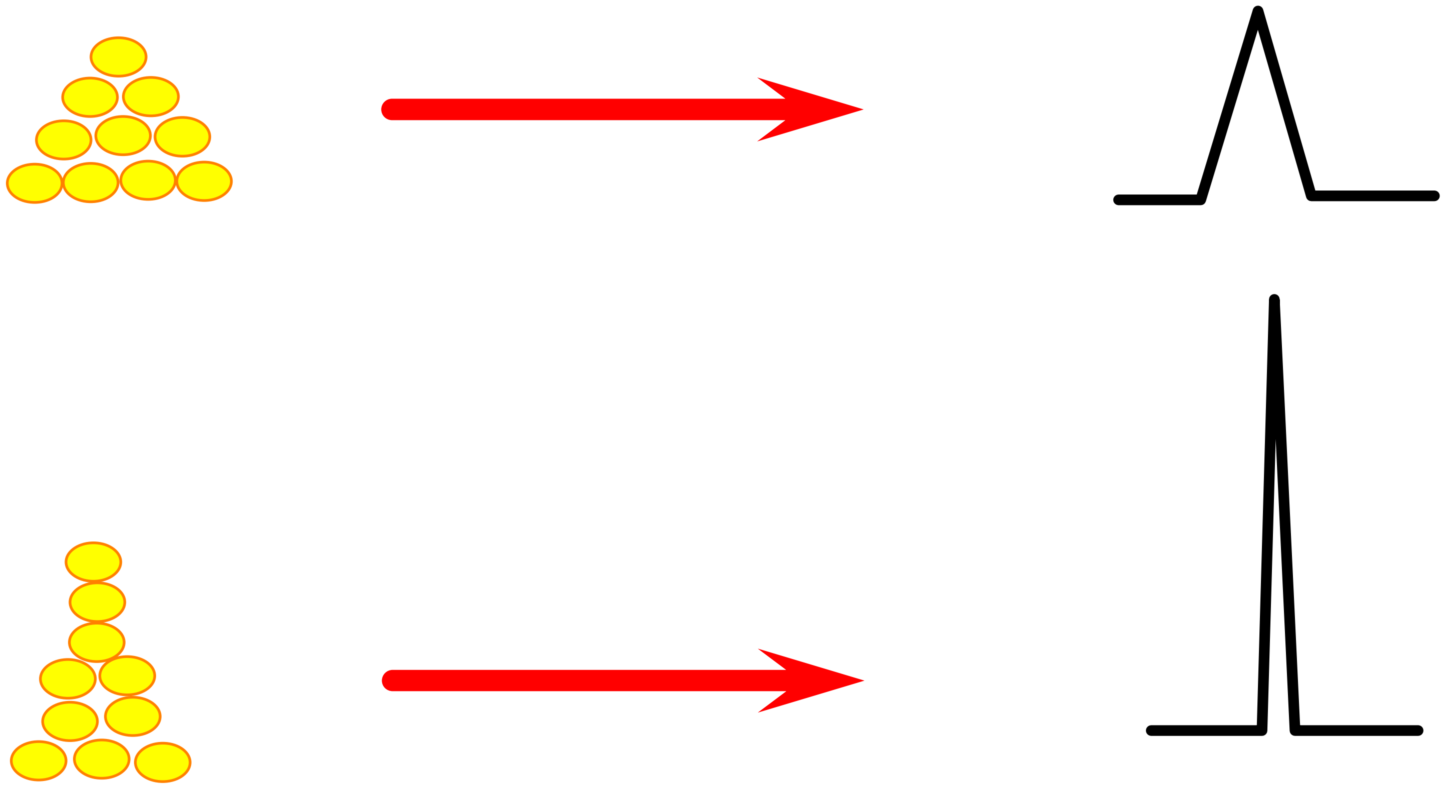
Tailing : Symmetry <1

Fronting : Symmetry >1

PEAK WIDTH



PEAK WIDTH



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)

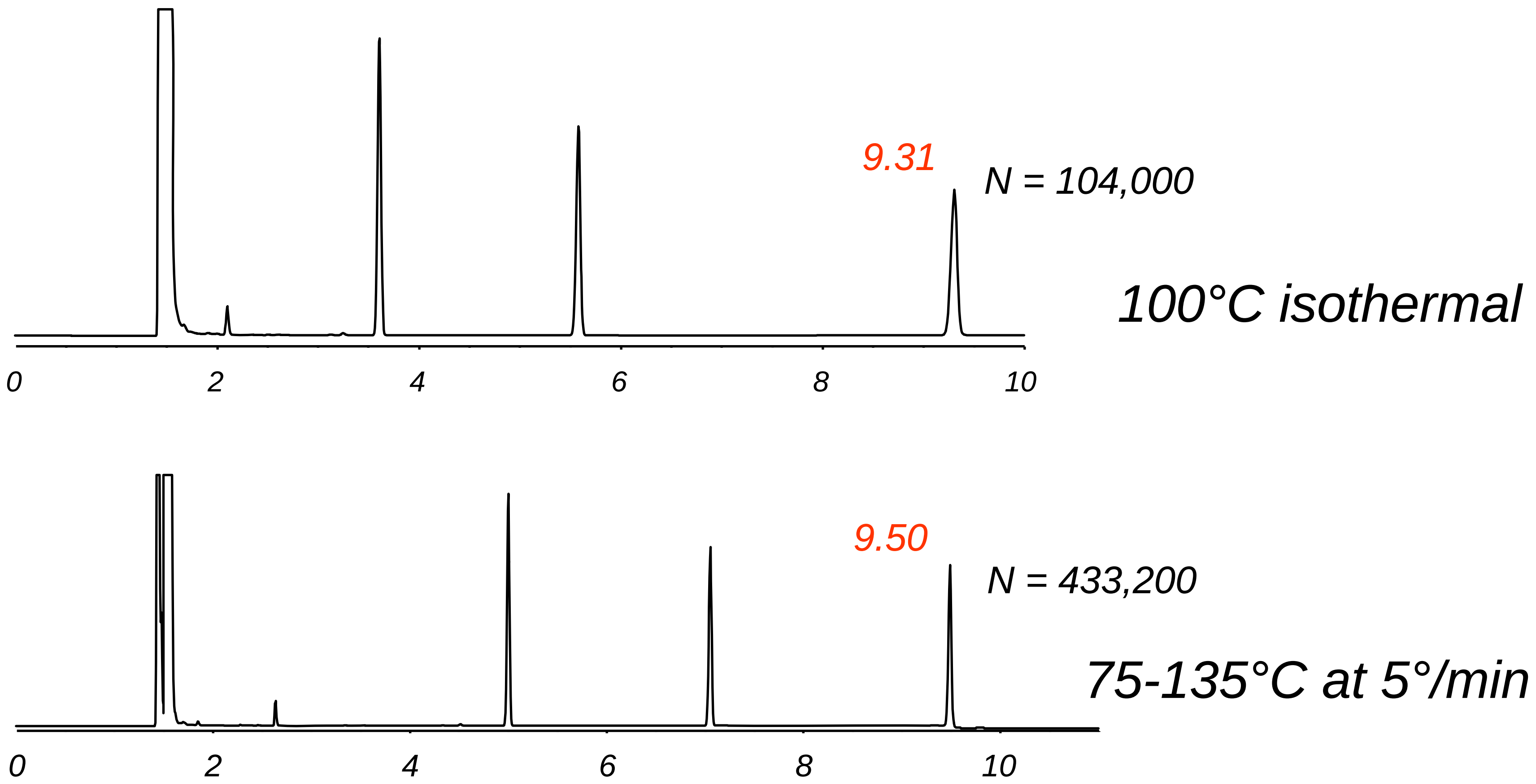
EFFICIENCY MEASUREMENT
Cautions

Actually, measurement 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

SEPARATION FACTOR (α)

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

co-elution: $\alpha = 1$

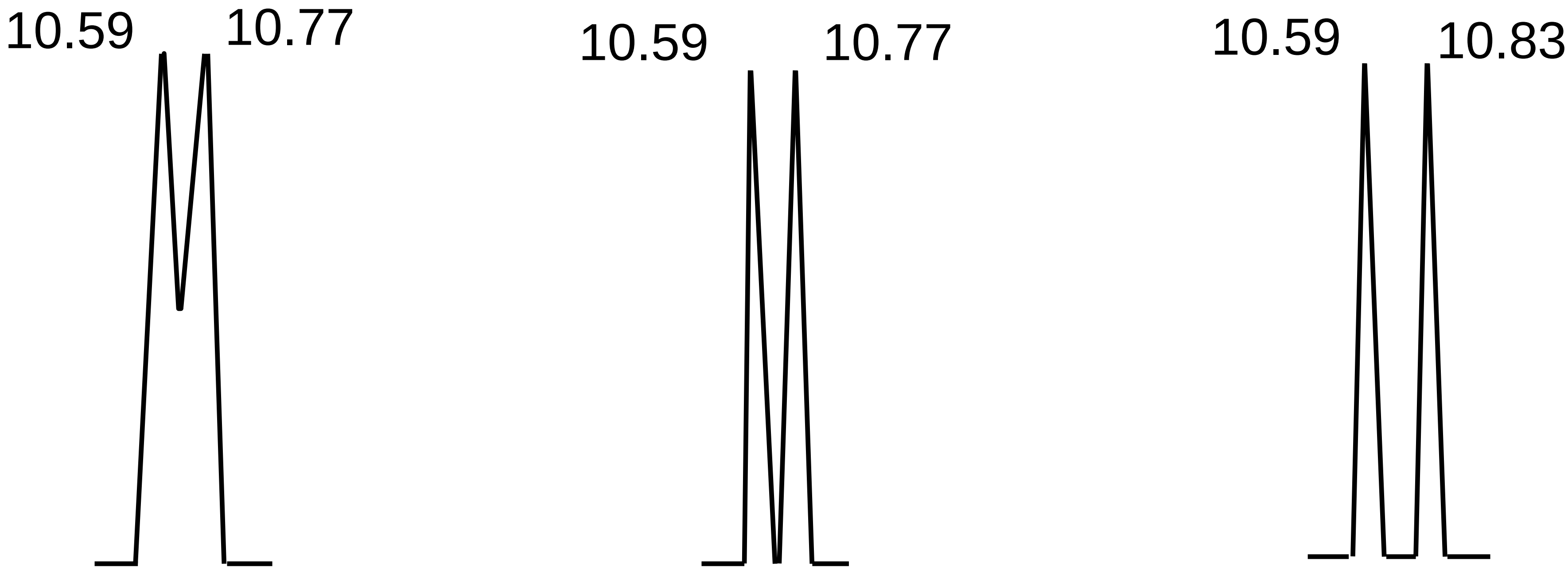
k_2 = retention factor of 2nd peak
k_1 = retention factor of 1st peak

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)

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

a: stationary phase, temperature

k: stationary phase, temperature, column dimensions

Conclusions

The GC is comprised of an inlet, column and detector that all work together to produce good chromatography

Separation (via K_c) is based on 3 things:

- Solute: different solubilities/interaction in a given stationary phase
- Stationary phase: different solubilities/interaction of a solute (correct column selection is critical!)
- Temperature: K_C decreases as temperature increases

When in doubt, contact Agilent Technical Support!

Agilent J&W Scientific Technical Support

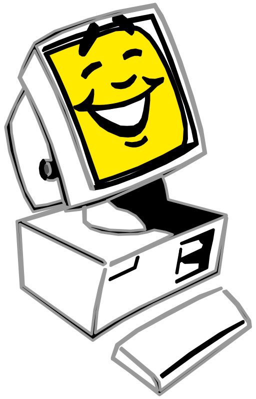
800-227-9770 (phone: US & Canada)*

**** Select option 41.***

866-422-5571 (fax)

GC-Column-Support@agilent.com

www.agilent.com/chem



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Upcoming GC and LC e-Seminars

Selection of a Capillary GC Column

March 13, 2008 – 2:00 p.m. EST

Method Development

March 18, 2008 – 2:00 p.m. EST

Installation, Care and Maintenance of Capillary GC Columns

April 22, 2008 – 1:00 p.m. EST