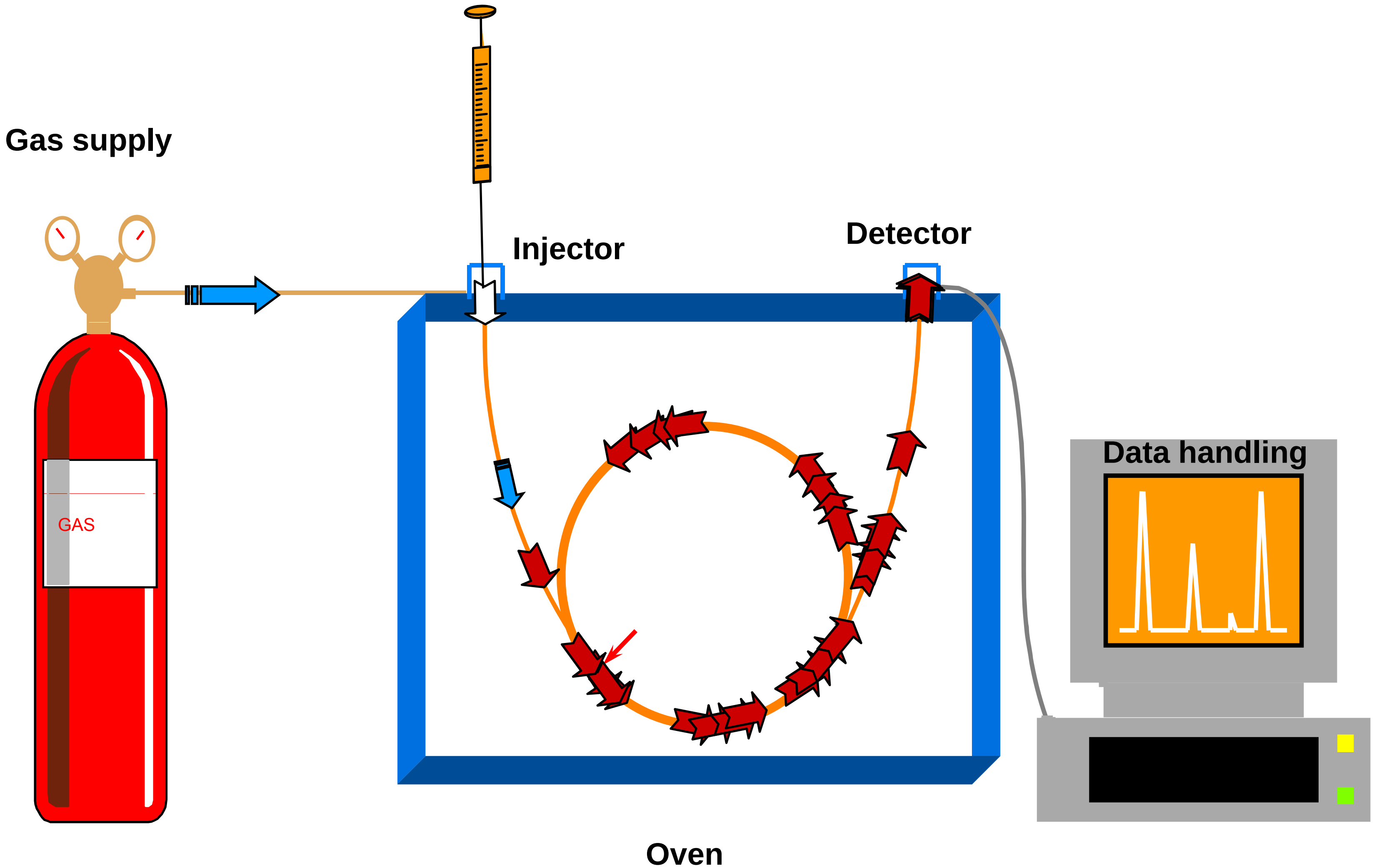
Introduction to Capillary GC

Typical GC System

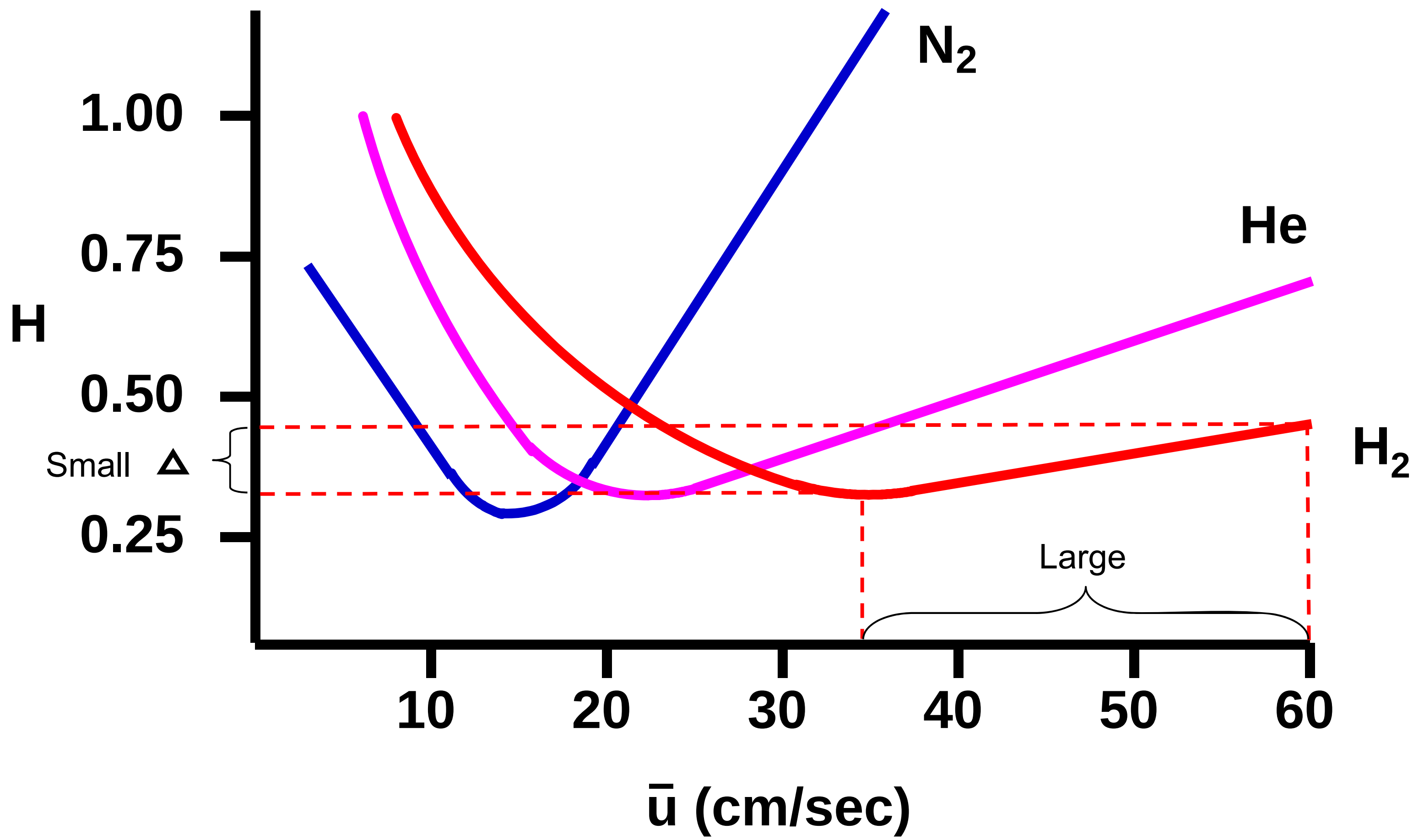


CARRIER GAS

Carries the solutes down the column

Selection and velocity influences efficiency and retention time

VAN DEEMTER CURVES



CARRIER GAS

Type	Velocity Range (u_{opt} – OPGV)
Nitrogen	8-16
Helium	20-40
Hydrogen	30-55

SAMPLE INJECTION

Goals:

Introduce sample into the column

Reproducible

No efficiency losses

Representative of sample

Sample Introduction

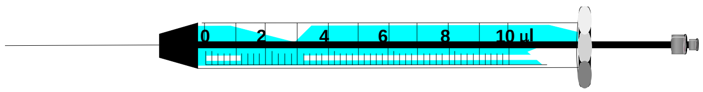
Purpose: To introduce a representative portion of sample onto the column in a reproducible manner, while minimizing sample bandwidth

Syringe Injection

Autosampler injection

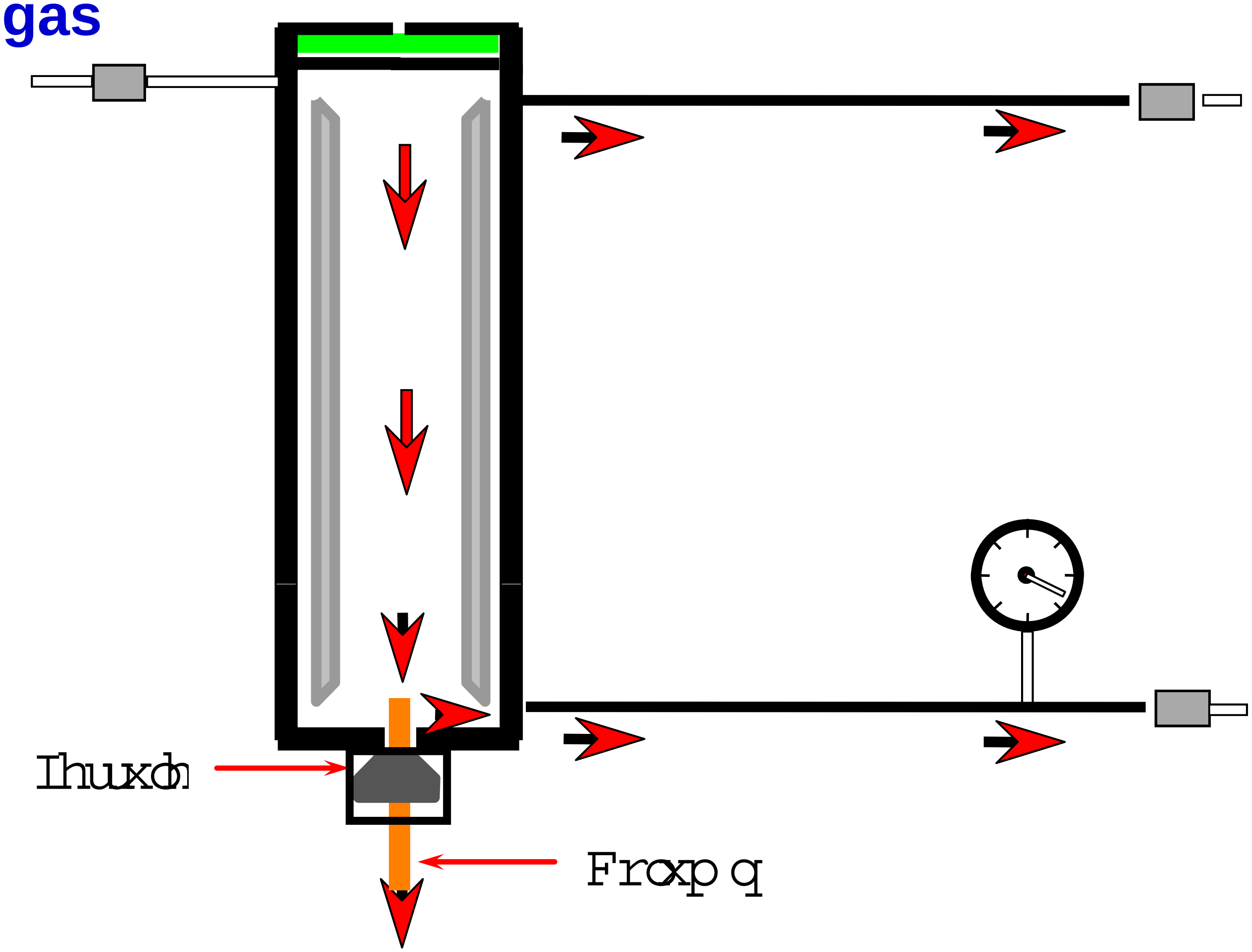
Valve Injection

- **Gas sampling valve**
- **Liquid sampling valves**



Objective: The sample must not be chemically altered , unless desired (e.g., derivatization). Success is not contamination, degradation, or discrimination.

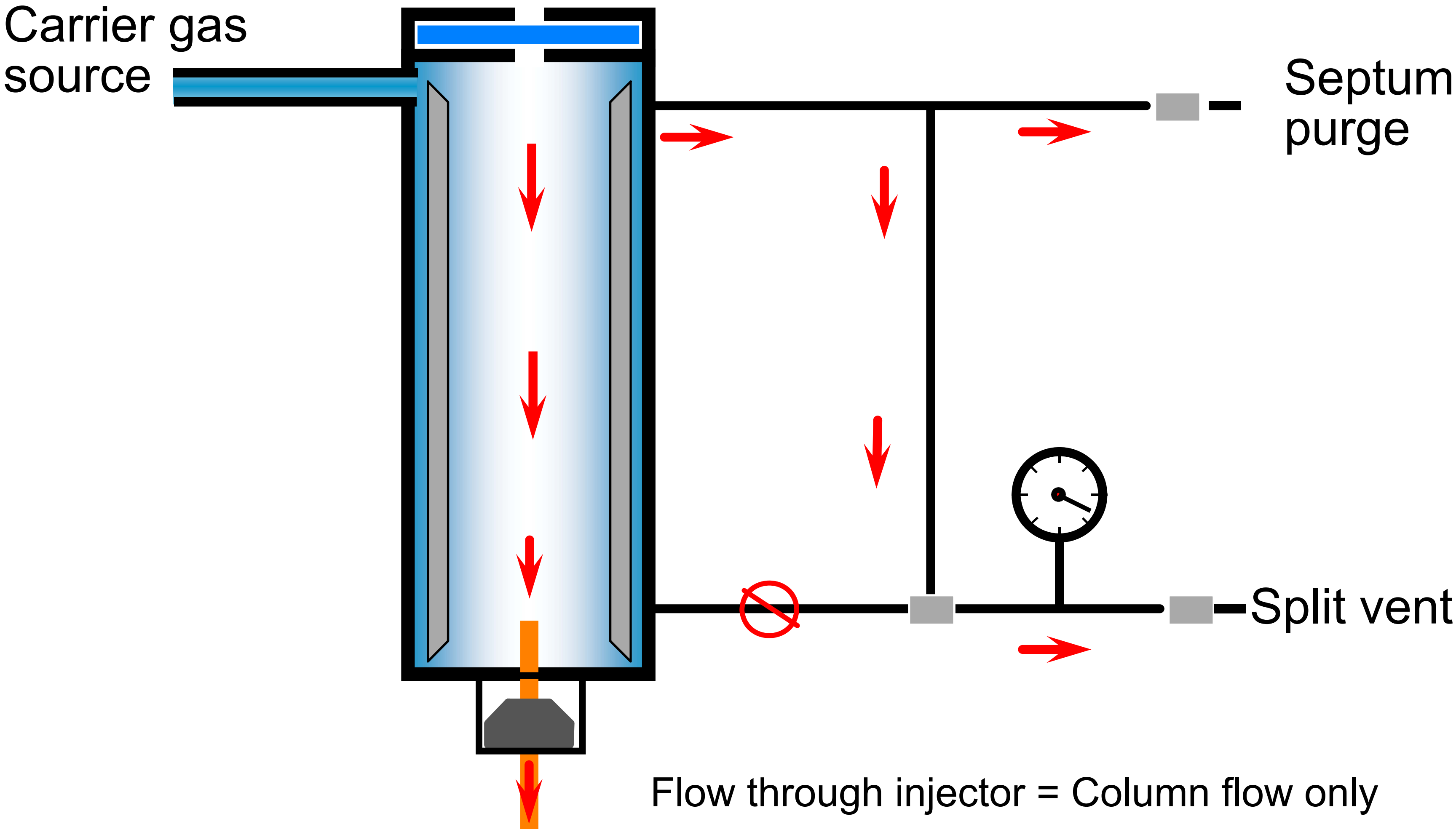
SPLIT/SPLITLESS INJECTOR



Flow through injector = Column flow + Split Vent Flow

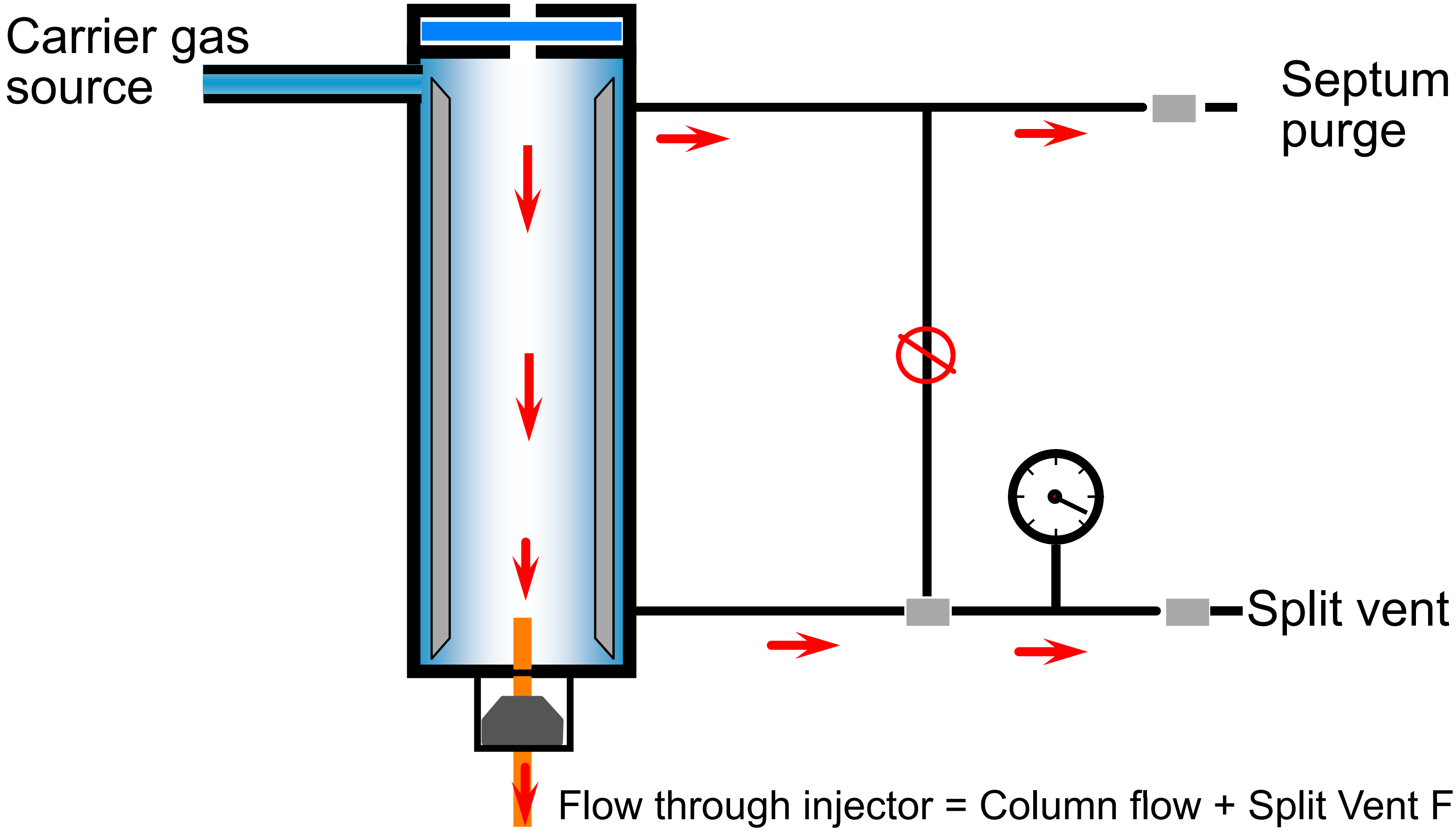
Splitless Injector

Purge Off At Injection



Flow through injector = Column flow only

Splitless Injector
Purge On After Injection



Flow through injector = Column flow + Split Vent Flow

DETECTORS

Purpose:

Responds to some property of the solutes

Converts the interaction into a signal

Immediate

Predictable

Detectors

Detector	Dynamic Range		MDL
TCD	10^5	Universal	400 pg Tridecane
FID	10^7	Responds to C-H bonds	1.8 pg Tridecane
ECD	$5x10^5$	Responds to free electrons	6 fg/mL Lindane
NPD	10^5	Specific to N or P	0.4 pgN/s 0.06 pg P /s
FPD	10^3S, 10^4P	Specific to S or P	60 fg P/s 3.6 pg S/s
SCD	10^4	Specific & Selective to S	0.5 pg S/s
NCD	10^4	Specific & Selective to N	3 pg N/s
MSD		Universal	S/N 400:1 1 pg/uL OFN

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

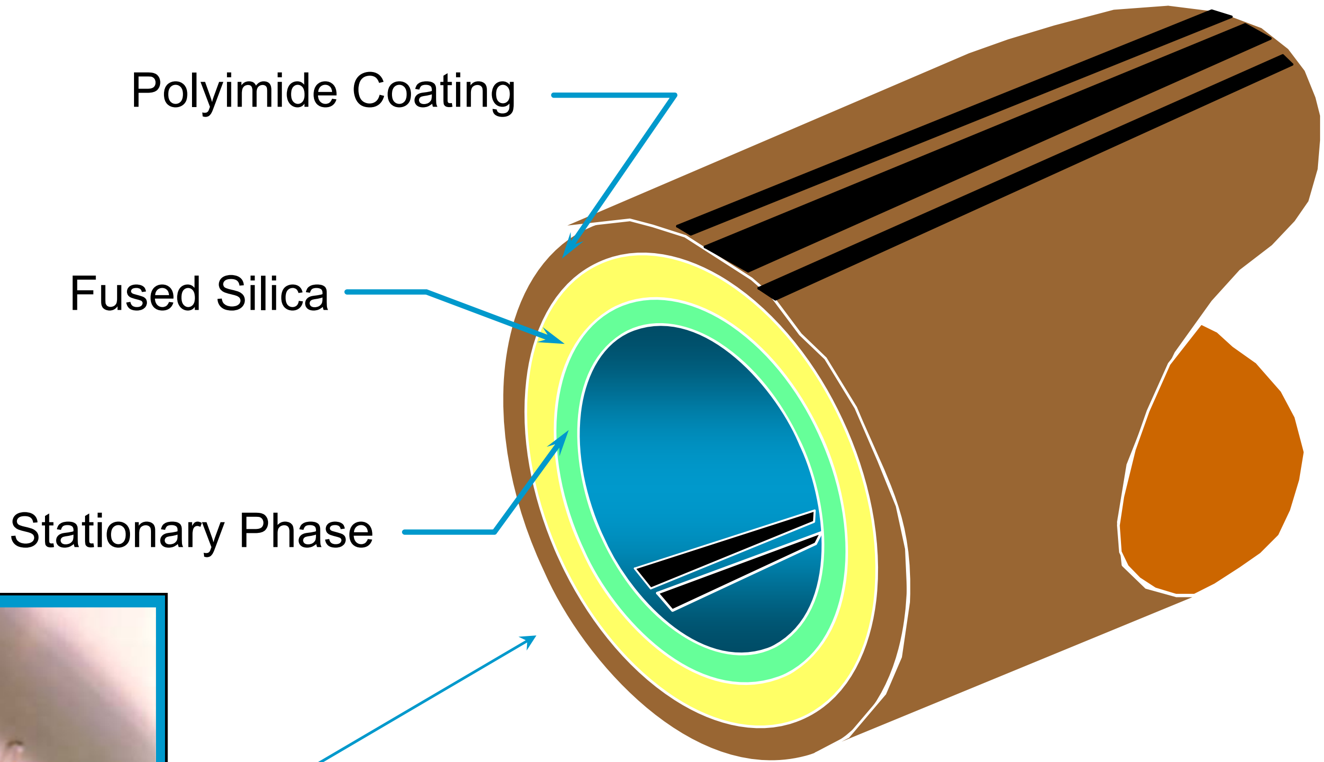
The compounds must have:

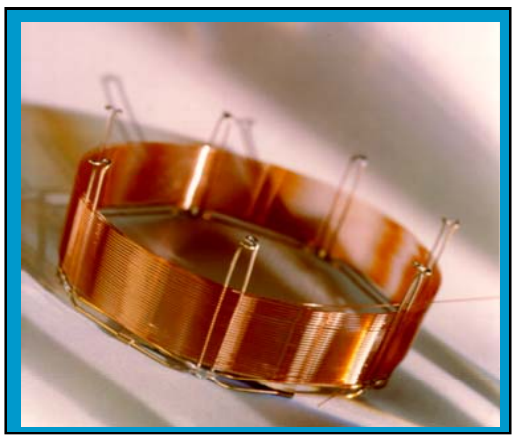
- ✓ Sufficient volatility
- ✓ Thermal stability

NO Inorganic Acids and Bases

Be mindful of salts!

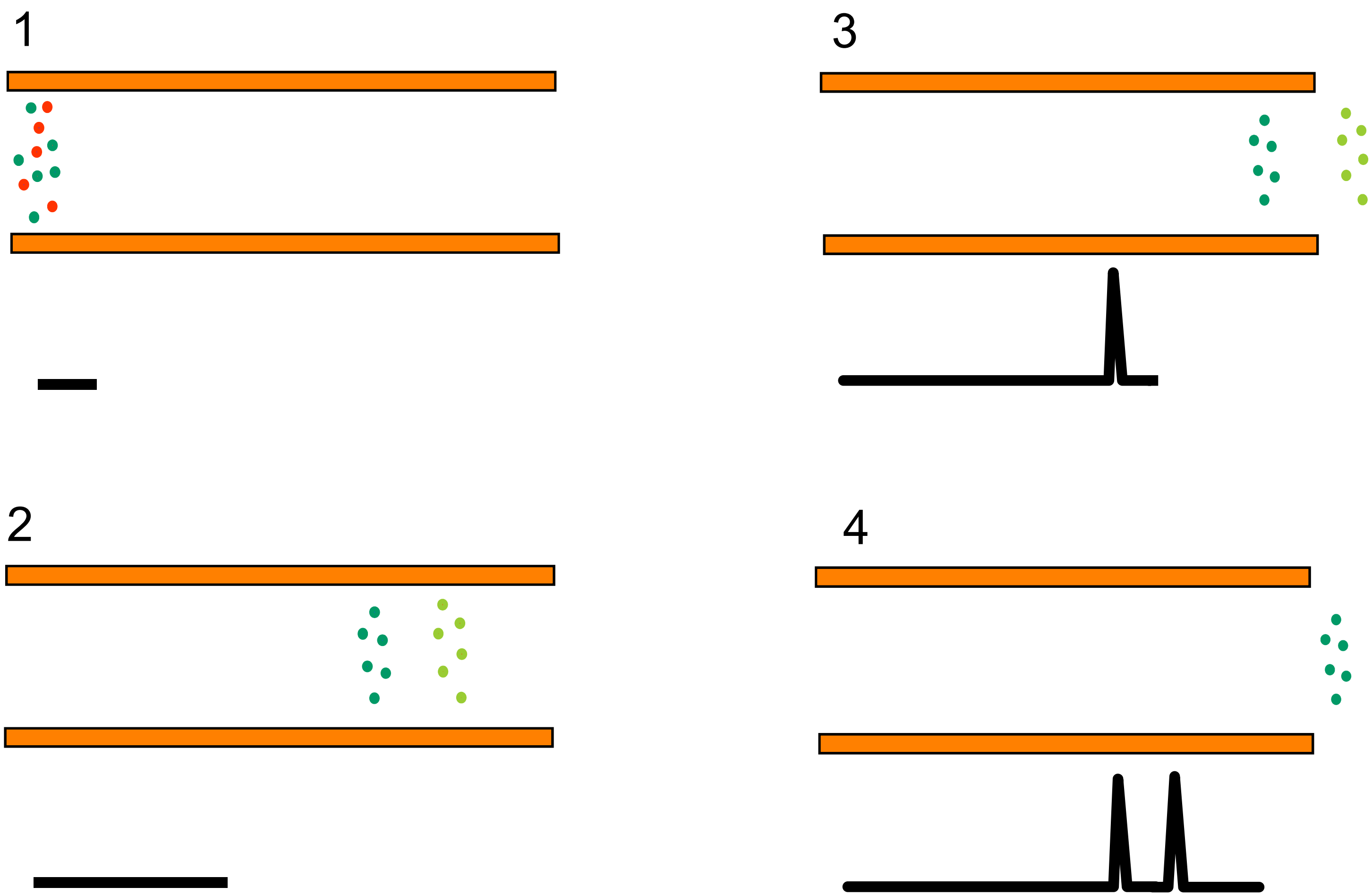
Typical Capillary Column





Expanded view of capillary tubing

SEPARATION PROCESS



TWO PHASES

Solute molecules distribute into the two phases

DISTRIBUTION CONSTANT (K_C)

$$N_C @ \frac{\text{conc. of solute in stationary phase}}{\text{conc. of solute in mobile phase}}$$

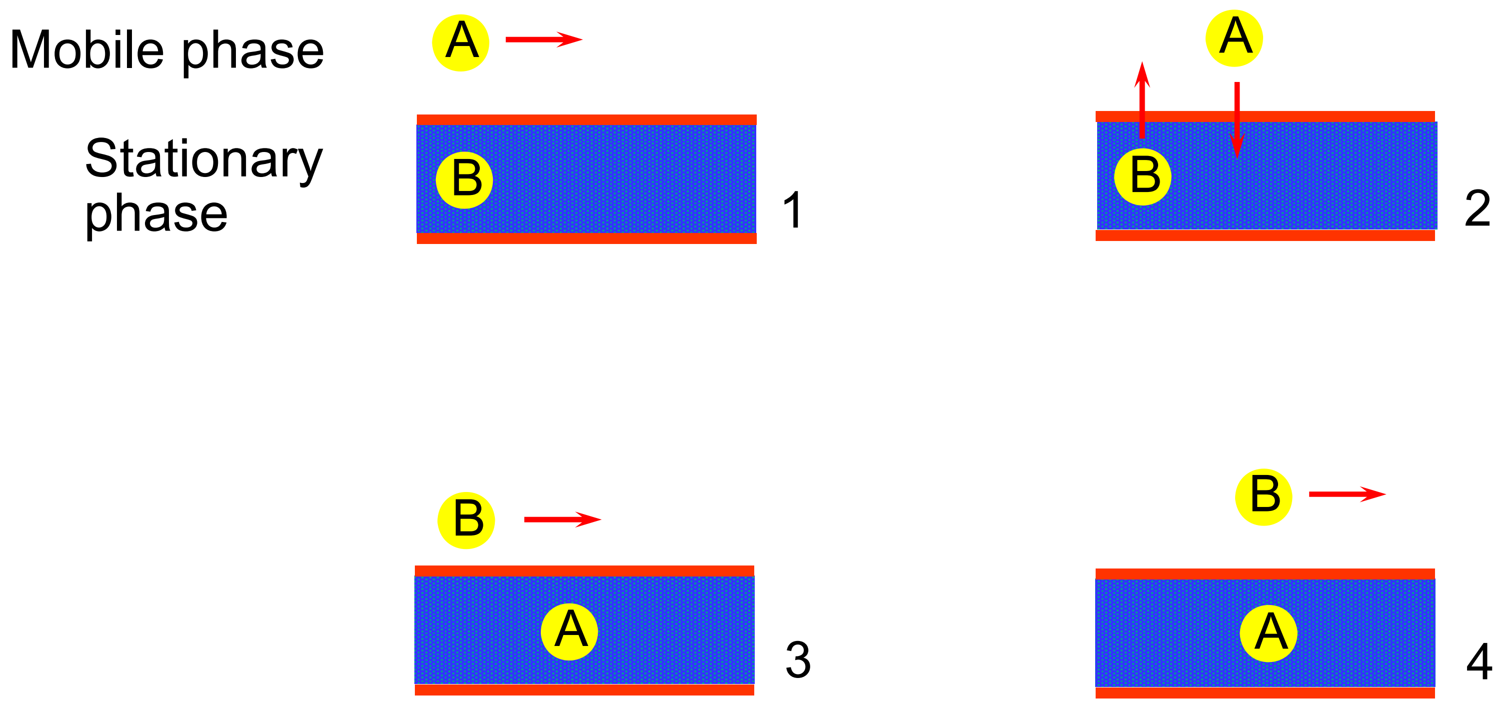
K_C formerly written as K_D

SOLUTE LOCATION

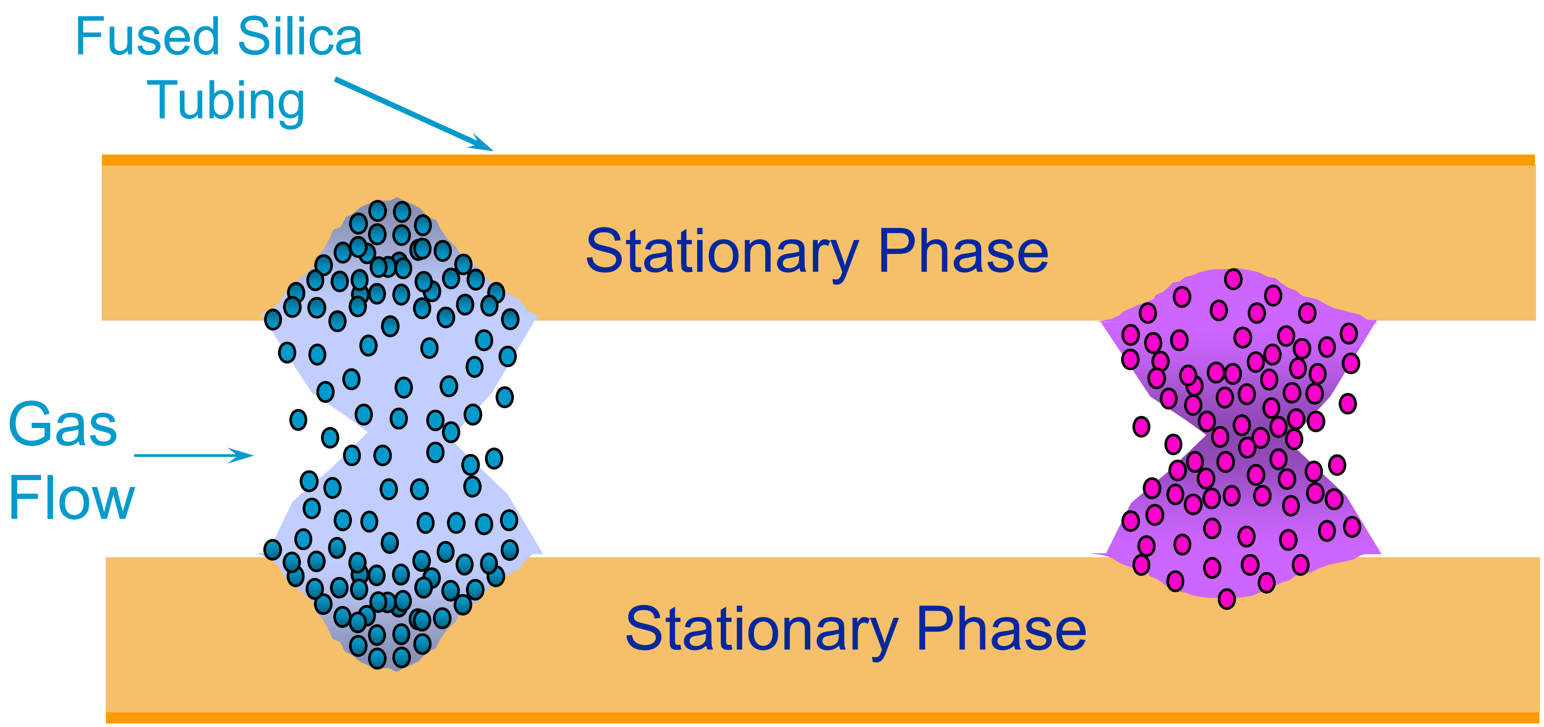
In stationary phase = Not moving down the column

In mobile phase = Moving down the column

SEPARATION PROCESS
Movement Down the Column



KC AND RETENTION



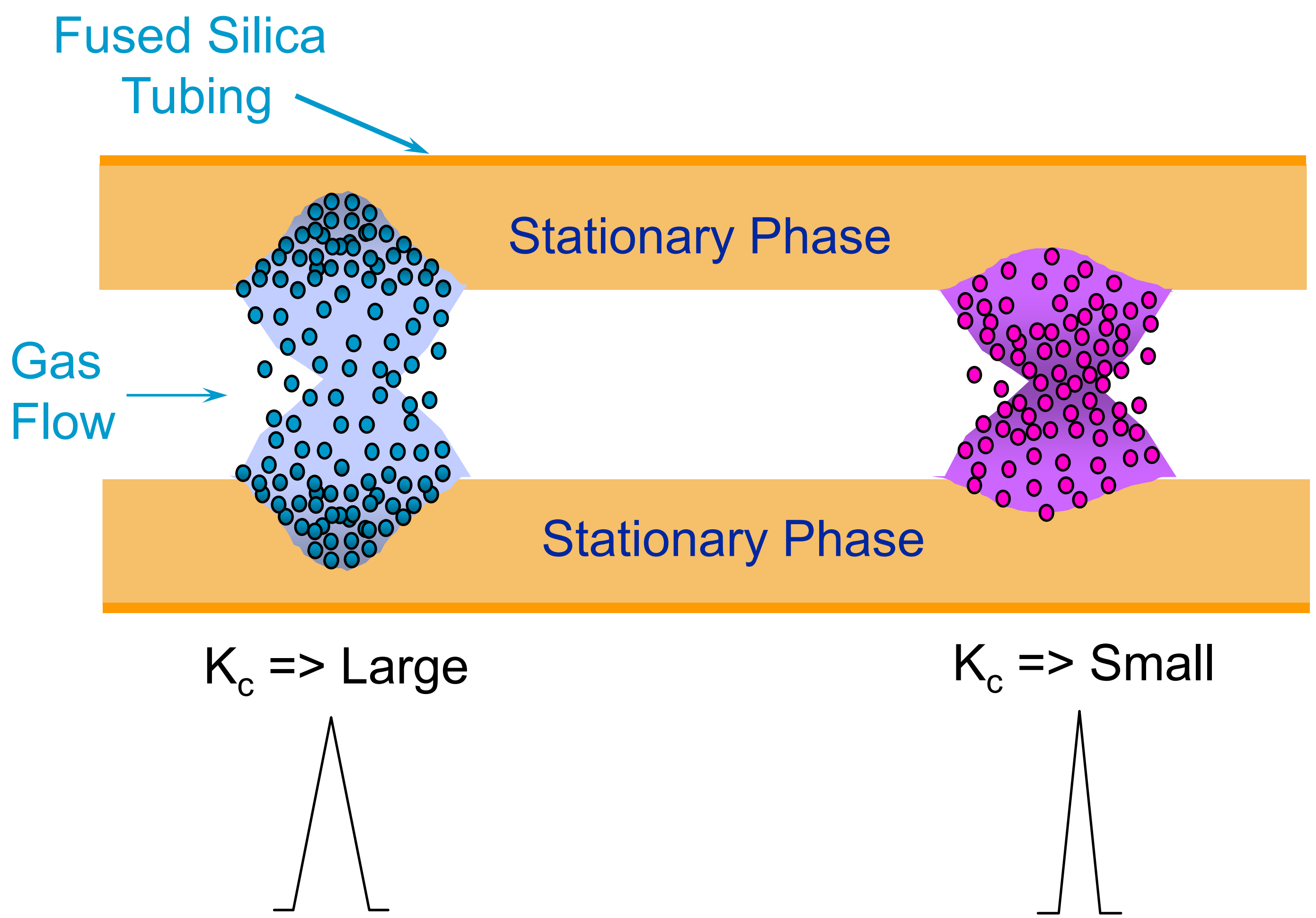
K_c => Large

retention

K_c => Small

retention

KC AND PEAK WIDTH
Time of Elution



THREE PARAMETERS THAT AFFECT K_C

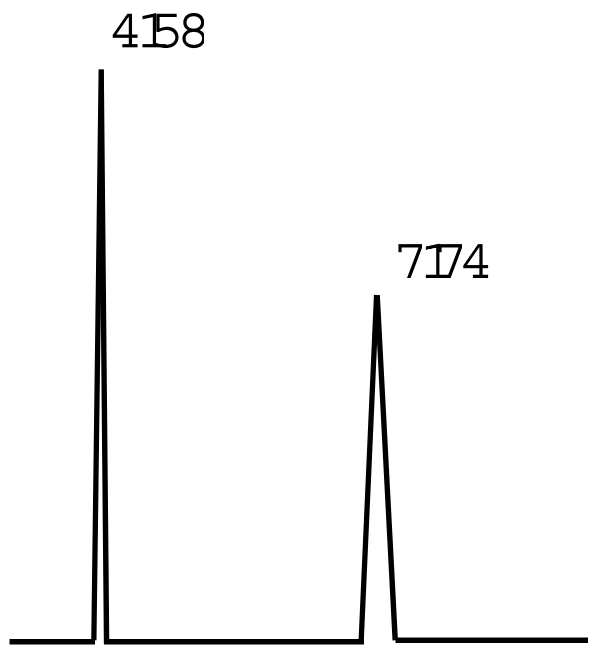
Solute:
different solubilities in a stationary phase

Stationary phase:
different solubilities of a solute

Temperature:
K_C decreases as temperature increases

RETENTION TIME

t_r



Time for a solute to travel through the column

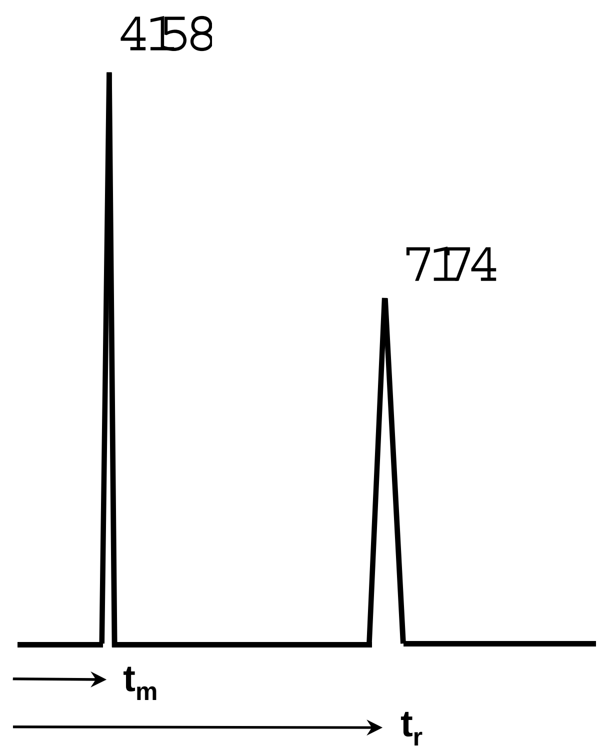
ADJUSTED RETENTION TIME t_r'

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

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)

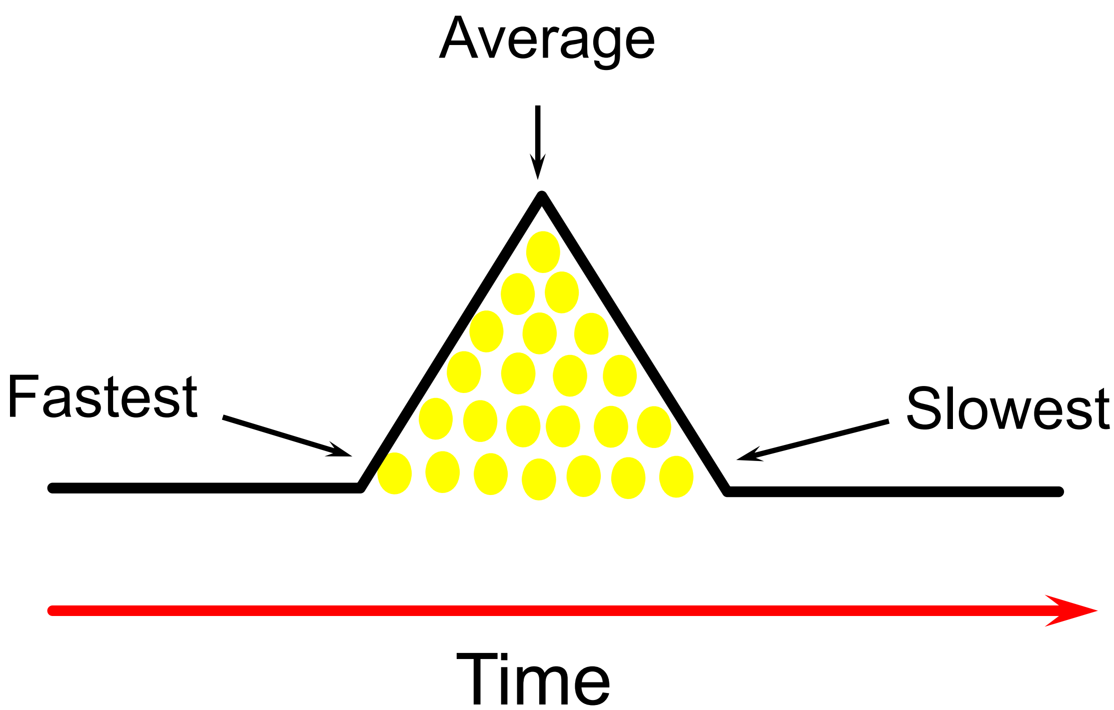
d_f = film thickness (µm)

DISTRIBUTION CONSTANT (Kc)

$$K_c = k\beta$$

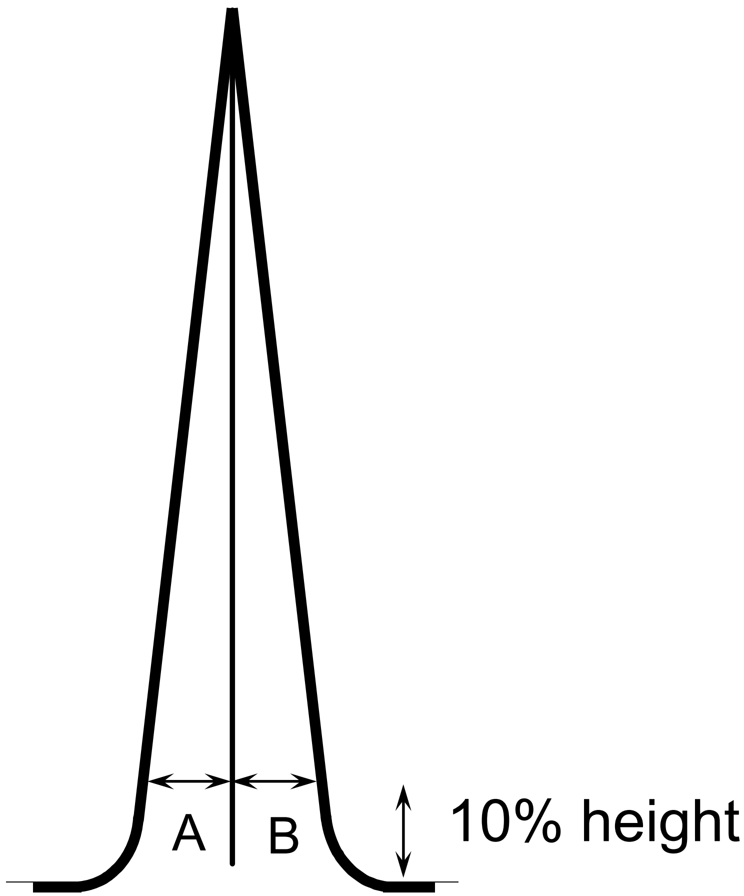
$$n \ @ \ \frac{t_r'}{t_m} \qquad \beta \ @ \ \frac{r}{2d_f}$$

RANGE OF RETENTION



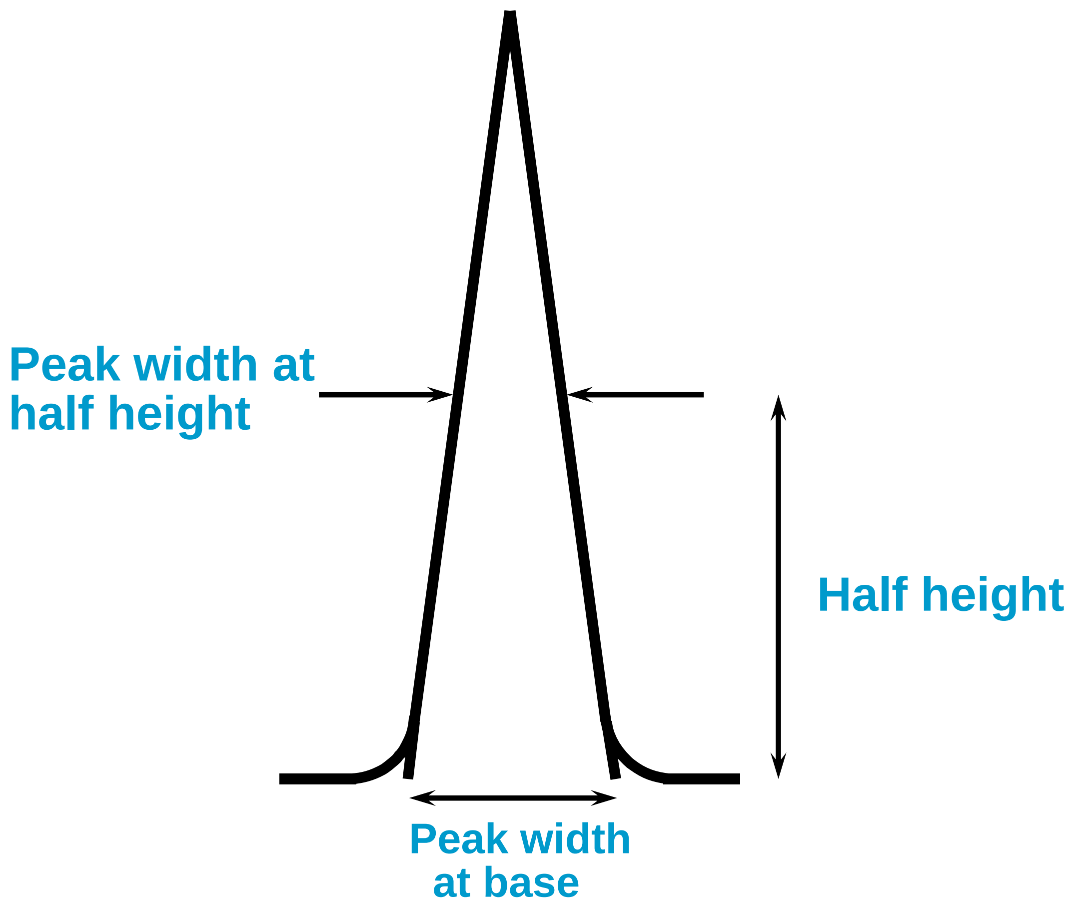
PEAK SYMMETRY

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

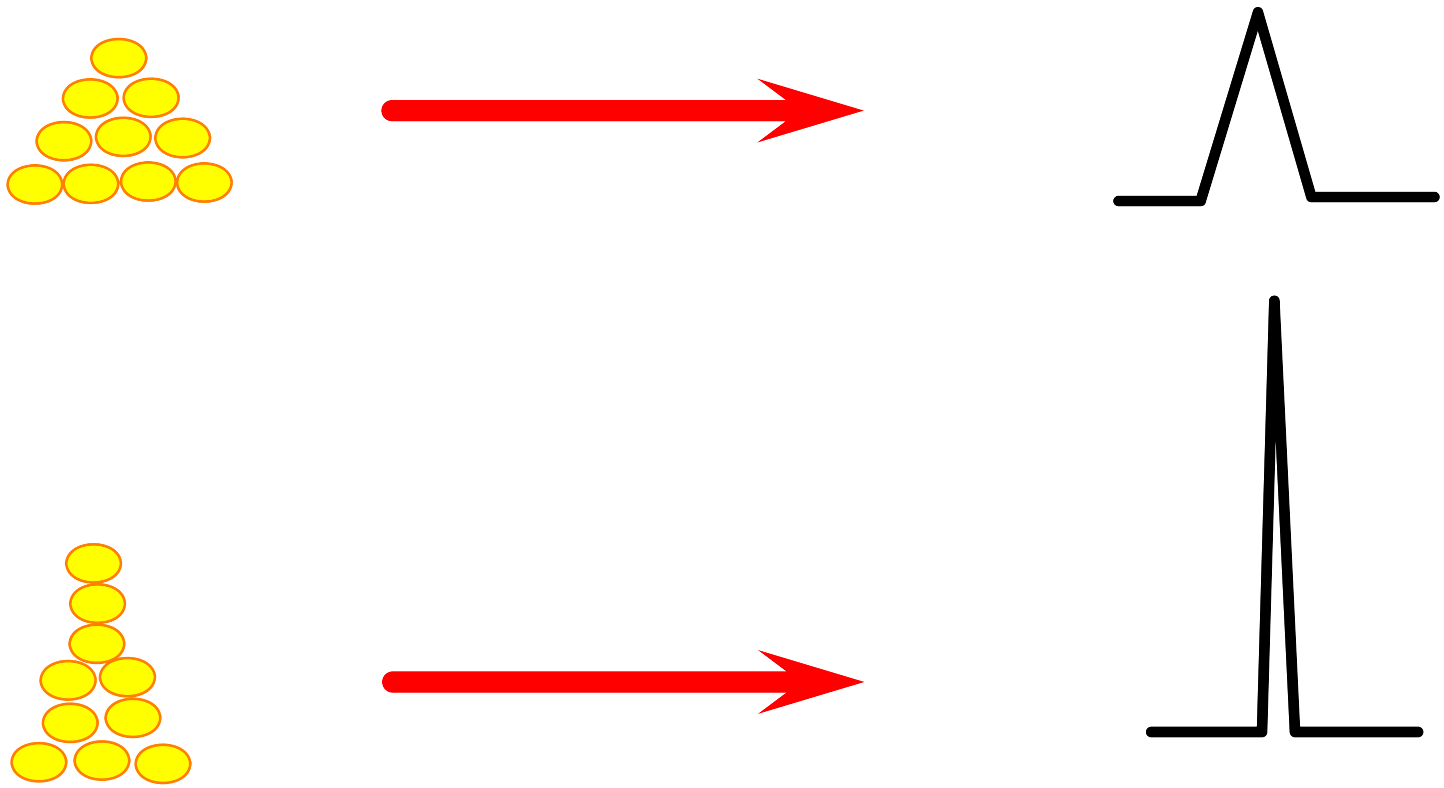


Tailing : Symmetry <1
Fronting : Symmetry >1

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)

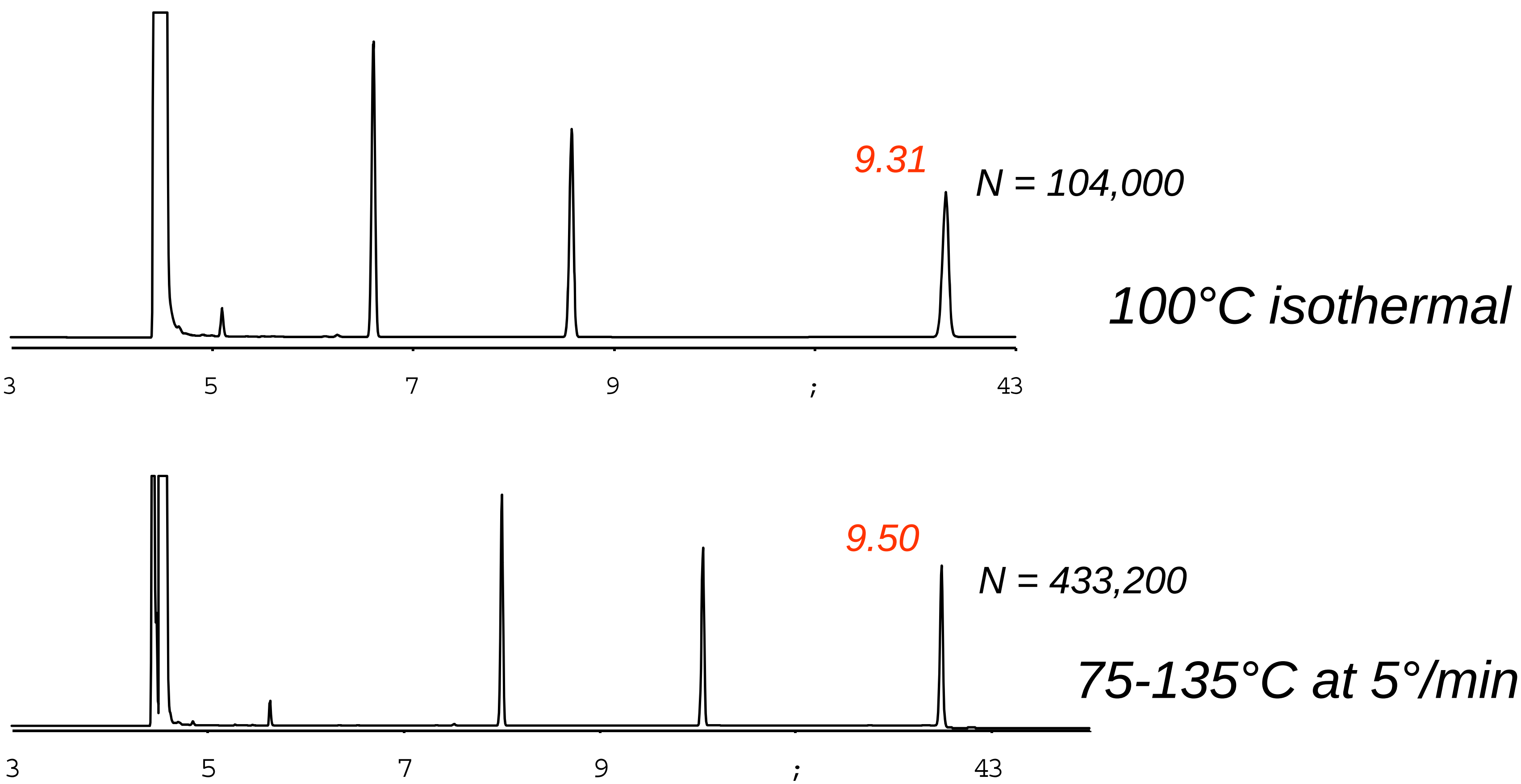
EFFICIENCY MEASUREMENT
Cautions

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: $\alpha = 1$

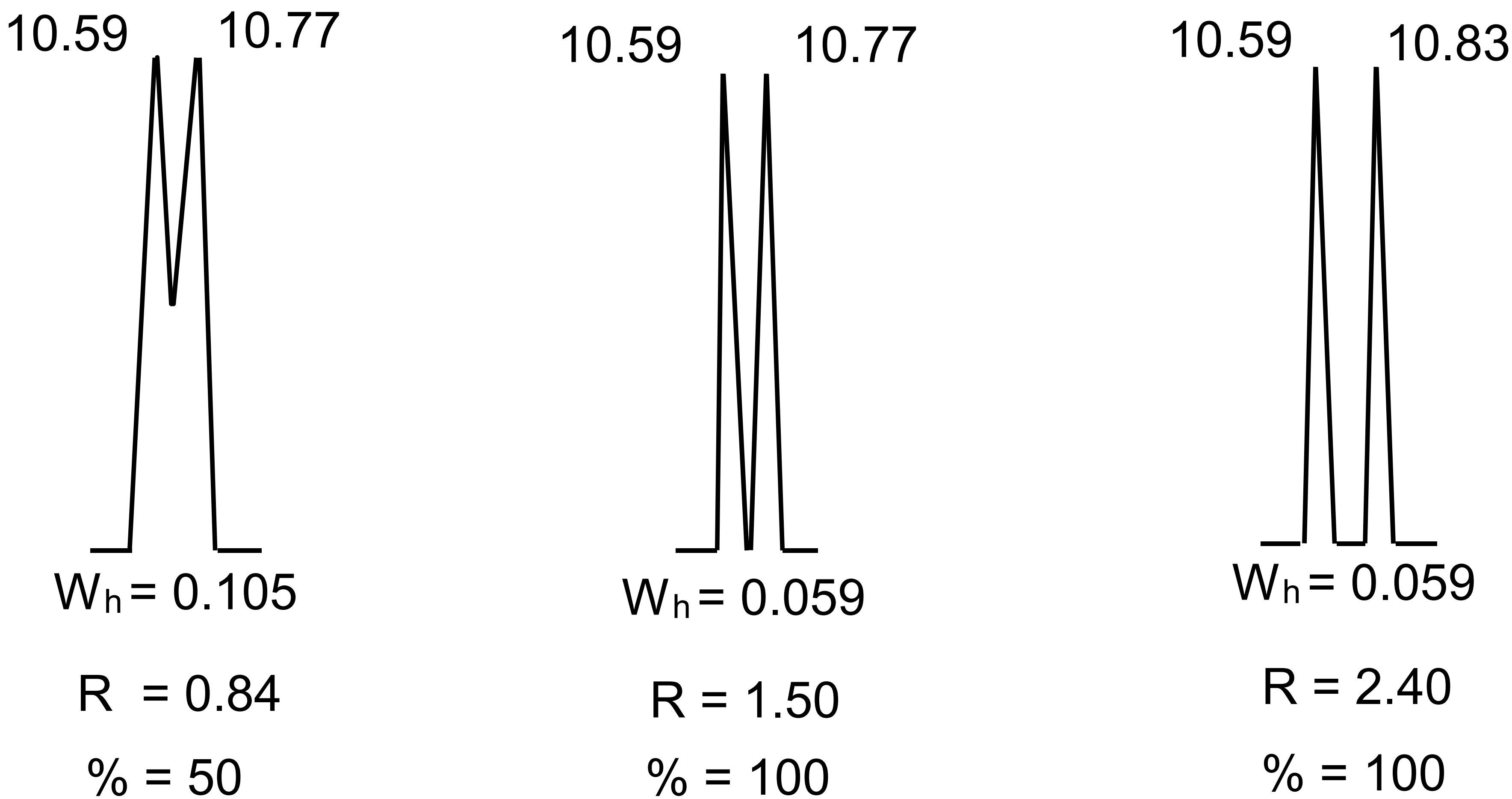
k_2 = retention factor of 2nd peak
k_1 = retention factor of 1st peak

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



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

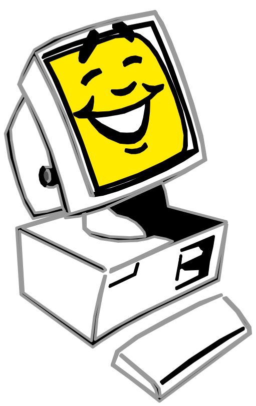
800-227-9770 (phone: US & Canada)*

**** Select option 3..3..1***

866-422-5571 (fax)

GC-Column-Support@agilent.com

www.chem.agilent.com



Wrap-up e-Seminar Questions

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