Introduction to Capillary GC

GC Columns and Consumables

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Introduction to Capillary GC
CARRIER GAS

Carries the solutes down the column

Selection and velocity influences efficiency and retention time
VAN DEEMTER CURVES

\[ H \text{ (cm/sec)} \]

\[ \bar{u} \text{ (cm/sec)} \]

- **N\textsubscript{2}**
- **He**
- **H\textsubscript{2}**

Small \( \Delta \)

Large \( \Delta \)
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

- Polyimide Coating
- Fused Silica
- Stationary Phase

Expanded view of capillary tubing
DETECTORS

Purpose:

Responds to some property of the solutes
Converting 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
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

Mobile phase
Stationary phase

1
2
3
4
KC AND RETENTION

Fused Silica Tubing

Stationary Phase

Gas Flow

Stationary Phase

$K_c \Rightarrow \text{Large}$

retention

$K_c \Rightarrow \text{Small}$

retention
KC AND PEAK WIDTH
Time of Elution

Fused Silica Tubing

Gas Flow

Stationary Phase

\[ K_c \Rightarrow \text{Large} \]

\[ K_c \Rightarrow \text{Small} \]
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

Time for a solute to travel through the column

1.25

4.41
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

\[ t'_r = tr - tm \]
\[ t'_r = 4.41 - 1.25 \]
\[ t'_r = 3.16 \text{ min} = \text{time spent in stationary phase} \]
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
($\beta$)

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

$r$ = radius (µm)
$d_f$ = film thickness (µm)
DISTRIBUTION CONSTANT (Kc)

\[ K_c = k\beta \]

\[ k = \frac{t_r'}{t_m} \quad \beta = \frac{r}{2d_f} \]
RANGE OF RETENTION
PEAK SYMMETRY

Symmetry = \frac{A}{B}

Tailing : Symmetry < 1
Fronting : Symmetry > 1
PEAK WIDTH

Peak width at half height

Peak width at base

Half height
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

100°C isothermal

75-135°C at 5°/min

N = 104,000
N = 433,200
SEPARATION VS. RESOLUTION

Separation: time between peaks

Resolution: time between the peaks while considering peak widths
SEPARATION FACTOR

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

co-elution: $\alpha = 1$

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

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

\( t_r \) = retention time
\( W_h \) = peak width at half height (time)
RESOLUTION
Baseline Resolution: $Rs = 1.5$

- $W_h = 0.105$
  - $R = 0.84$
  - $\% = 50$

- $W_h = 0.059$
  - $R = 1.50$
  - $\% = 100$

- $W_h = 0.059$
  - $R = 2.40$
  - $\% = 100$
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
- \( \alpha \) = 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