An Introduction to Gas Chromatography

Presentation · October 2018

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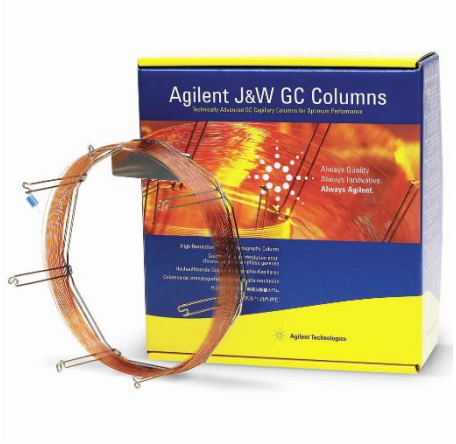
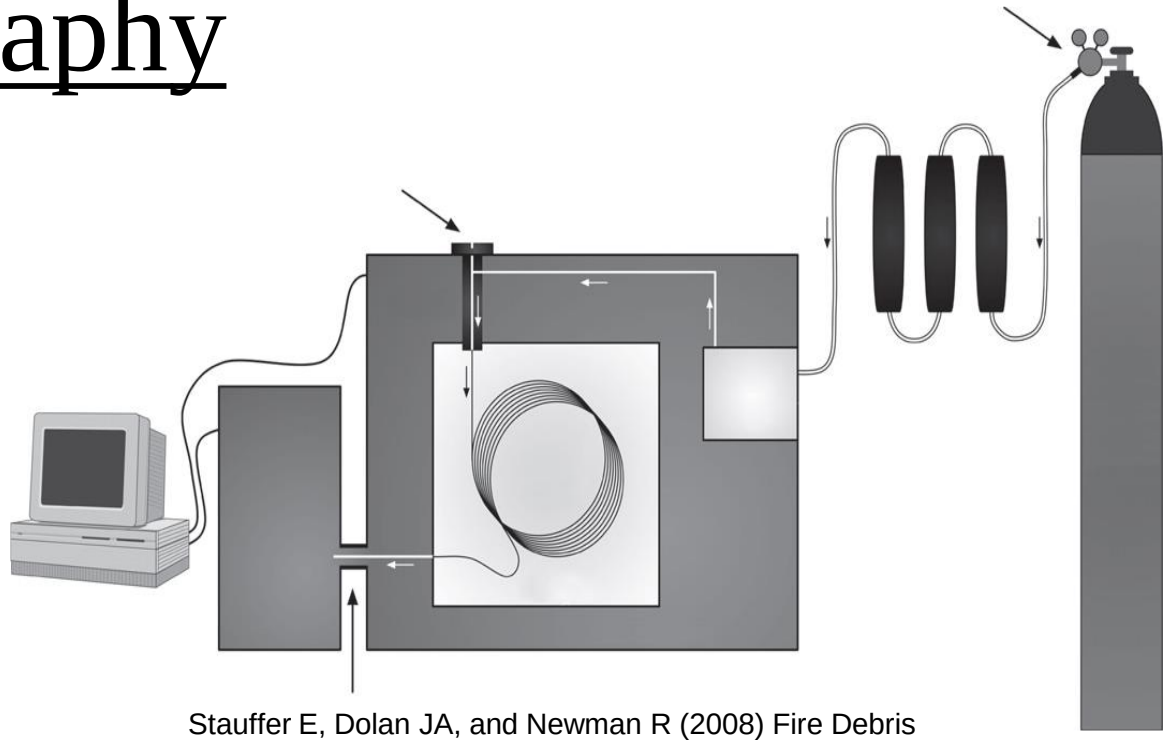
Gas chromatography



Gas chromatography

- Gas chromatography is a separation technique based on partitioning analytes between two immiscible phases: gaseous mobile phase (Carrier gas) and a stationary solid or immobilized liquid phase (packed or hollow capillary column).

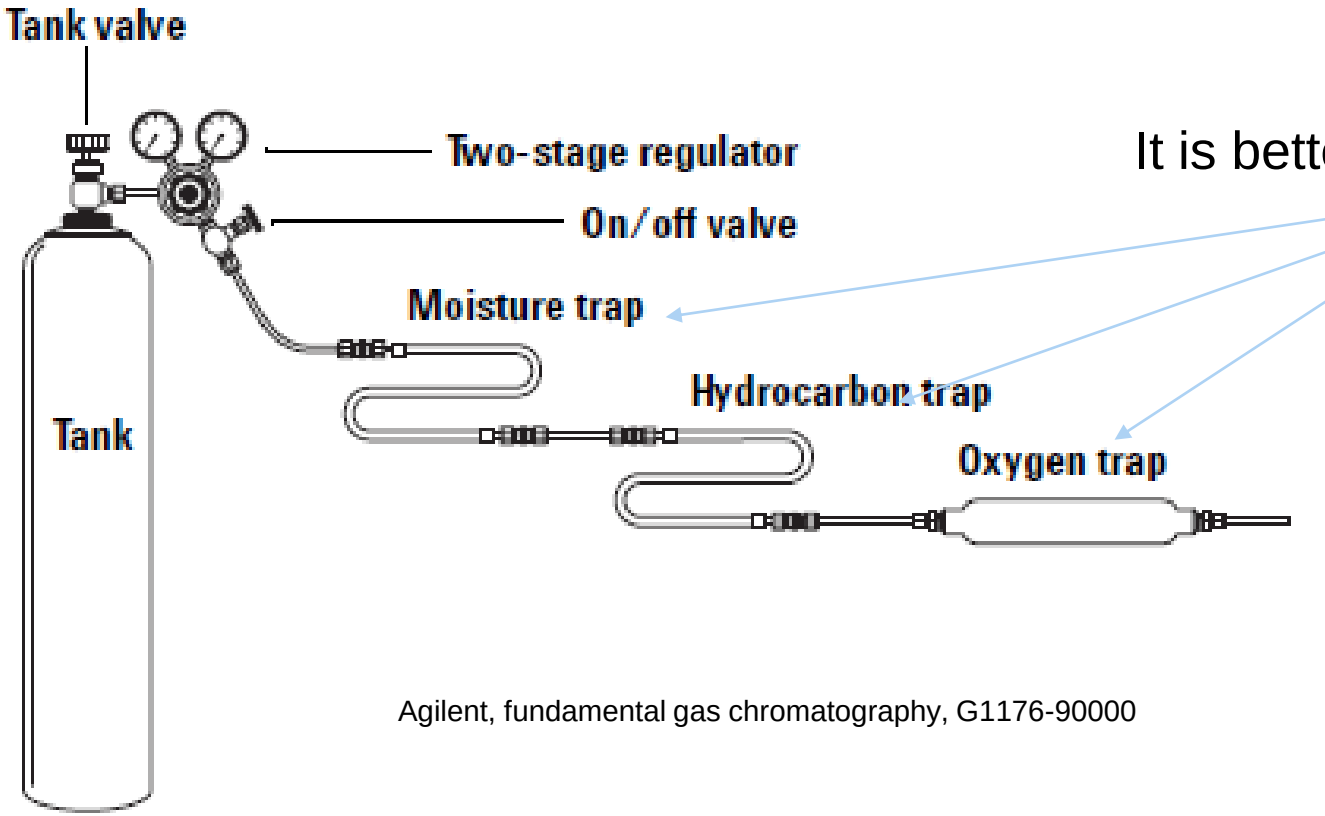


Stauffer E, Dolan JA, and Newman R (2008) Fire Debris Analysis, p. 246. Burlington, MA: Elsevier Academic Press

- The sample is first vaporized by a heated inlet system to be passed through a gaseous carrier into GC column. The passed analytes adsorbed on the stationary phase of the GC column, the adsorbed analytes eluted by applying heating program (the stronger adsorption of an analyte, the higher heat required for its elution). Therefore, GC is suitable for analysis of volatile (or semi-volatile) and a thermally stable analyte.

Carrier gas

- A carrier gas is responsible for carrying the sample molecules into the column and finally to the detector.
- Carrier gas must be: inert with the stationary phase, of high purity 99.999 %. (for residue analysis 99.9999 % is preferred).



It is better to be vertically standing

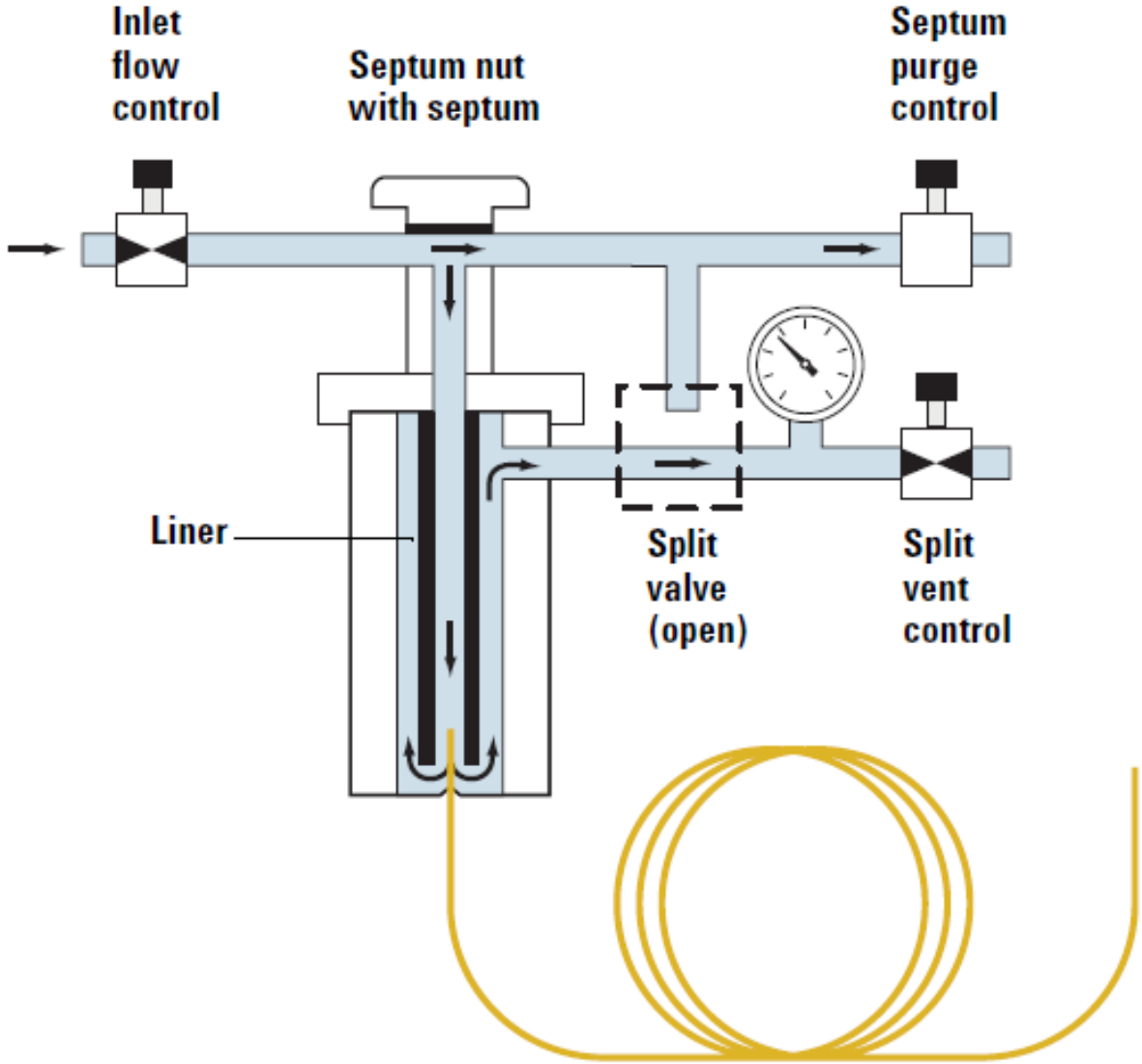
Agilent, fundamental gas chromatography, G1176-90000

GC Injection Techniques

- Split/split-less
- Programmed temperature vaporization (PTV) injection.
- On-column injection

Split injector

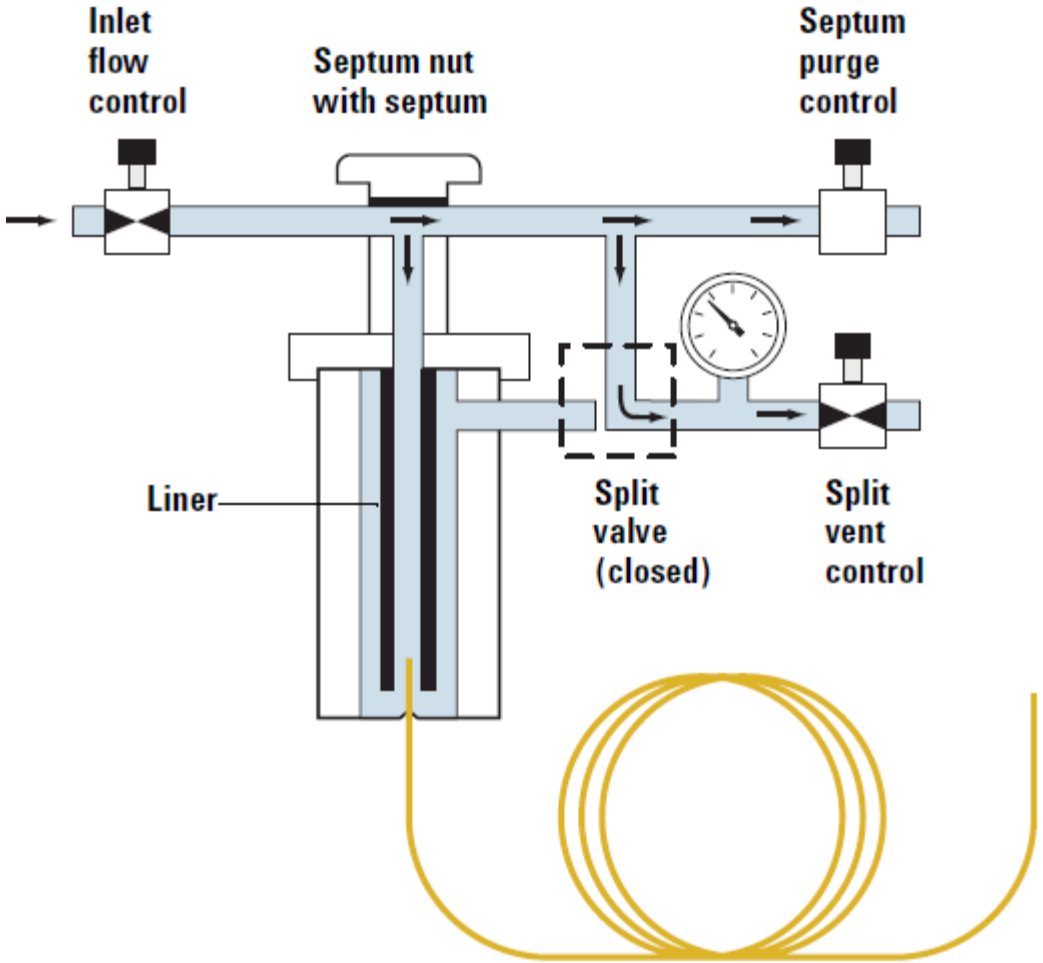
- In the split injection mode, sample enters the hot liner and volatilized rapidly.
- Vaporized sample is mixed with a carrier gas (diluted).
- Finally, a large part of the diluted vaporized sample is split away from the Colum, while a small part will enter the column.
- This mode of injection is used for analysis of samples of high analyte concentrations.



Agilent, fundamental gas chromatography, G1176-90000

Split-less injector

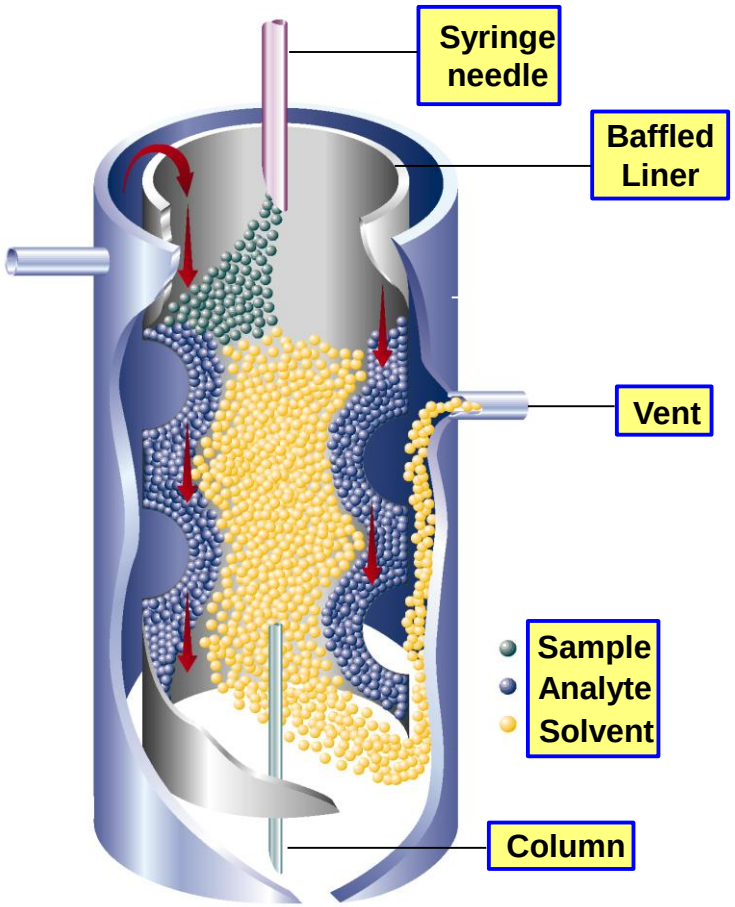
- Is the split-less injection mode, sample enter the hot liner and volatilized rapidly.
- Vaporized sample is mixed with a carrier gas (diluted).
- Finally, all the diluted vaporized sample enter the column.
- After that, the split valve is opened to remove residual vapors.
- This mode of injection is used for analysis of samples of trace analyte concentrations (residue analysis).



Agilent, fundamental gas chromatography, G1176-90000

PTV injector

- In the program temperature vaporized injector the sample is injected in a cooled liner (enable sample and analyte adsorption). While, the solvent will be split away. Therefore, injection in PTV should be carried out slowly.
- After that, split valve closed, and beginning ramping temperature increase (ramp PTV) to vaporize adsorbed sample matrix and analytes to be carried into the column



On-column injection

https://www.sigmaaldrich.com/analytical-chromatography/analytical-products.html?TablePage=110598941

- sample aliquots are directly introduced onto the analytical column (0.2-0.5) at low temperatures (6 80°C).

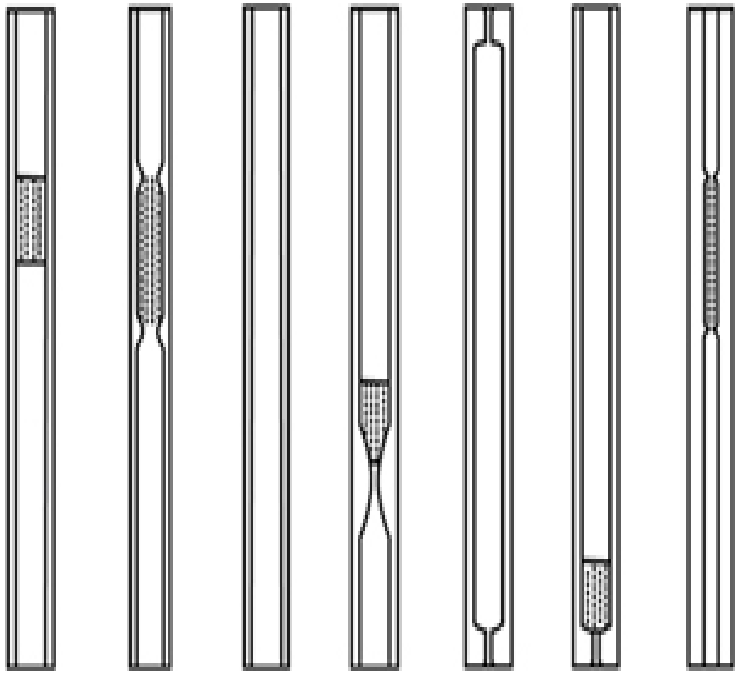
- On column injection is favored for an analyte that can be thermally degradated at the elevated heated split or split-less mode (around 200 C).

- These injection mode require careful awareness to attain a good reproducibility.

- A liner of the wider volume is favorable for this injection.

Choosing the right liner

- The used solvent (containing the analytes) shall have an expansion volume that will not exceed 75 % of the liner volume (especially for split-less mode of injection).
- Inner-surface of the used liner shall be highly inert, especially for residue analysis.
- If the sample is not so cleaned up, liners containing glass wool is more favorable.
- The temperature of the liner should be enough to evaporate the desired analytes and avoid its degradation.
- A bottom tap liner is more favorable for splitless injection.

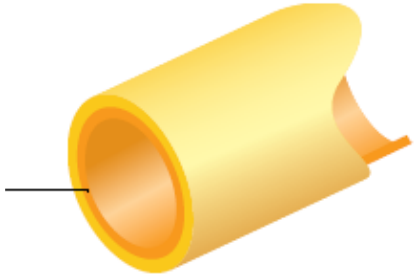


https://www.chromservis.eu/c/gc-liners

http://www.restek.com/Supplies-Accessories/GC-Accessories/Inlet-Liners-Liner-Supplies

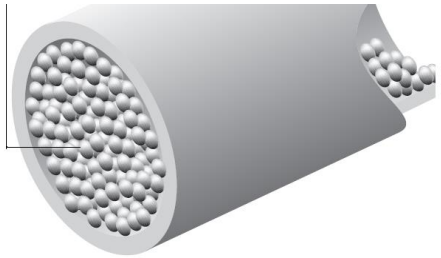
GC-COLUMN

Capillary columns
Open tubular (OT)



Agilent, fundamental gas chromatography, G1176-90000

Packed columns



Agilent, fundamental gas chromatography, G1176-90000

Category	Column diameter range (mm)	Standard commercial column diameters (mm)	Max flow-rate (mL/min) *)
Megabore	≥0.5	0.53	≥660
Wide bore	≥0.3 to <0.5	0.32, 0.45	≥85 to <660
Narrow bore	≥0.2 to <0.3	0.20, 0.25, 0.28	≥17 to <86
Microbore	≥0.1 to <0.2	0.10, 0.15, 0.18	≥1 to <17
Sub-microbore	<0.1	Various	<1

*) Flow rate calculated using helium carrier gas at 690 kPa, 200°C oven, vacuum outlet conditions and 10-m column length.

K. Maštovska, S.J. Lehotay, J. Chromatogr. A, 1000 (2003) 153

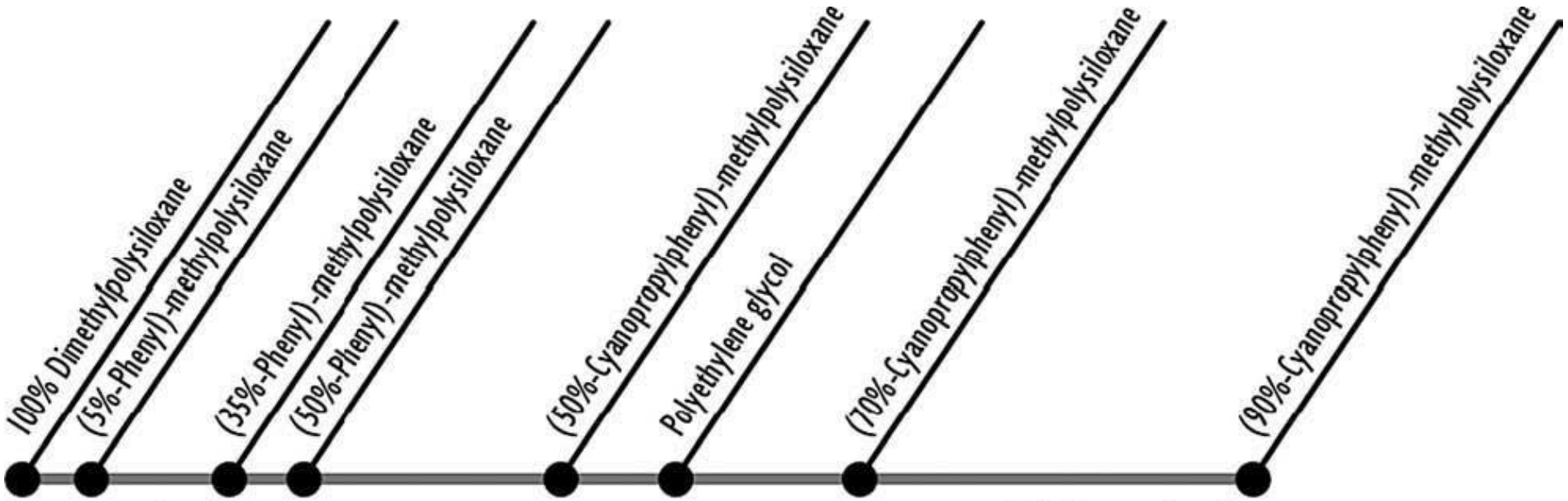
GC-COLUMN

- The commonly used stationary phase for capillary GC column is poly dimethyl siloxane [PDMS]. The separation, in this case, will depend upon dispersive interactions (van der Waals) with nonpolar part of the analytes.

- The PDMS stationary phases can be more polar by adding phenyl, cyano, or trifluoro functional groups each of these stationary phases leads to different separation results.

- In case of using PDMS with cyano, trifluoro, or (especially) hydroxyl functional groups, analyte separation can occur based on hydrogen bonding interactions (the strongest intermolecular forces in capillary gas chromatography (GC).

GC-COLUMN



Pesticides
PCBs, PBDEs
Dioxins, PAHs
Phthalates
Mycotoxins*)
Acrylamide*)
Chloropropanols*)

*) derivatised form

Dioxins

Acrylamide

Separation of isomers and congeners
(pesticides, PCBs, PAHs)

Phases suitable for 2nd dimension in GC×GC
(non-polar×polar)

Jana Hajšlová, Tomášajka, Gas chromatography–mass spectrometry (GC–MS), Food Toxicants Analysis, 2007

GC-COLUMN

- A longer column will increase the resolution (selectivity), but it will also increase analysis time, and cost.

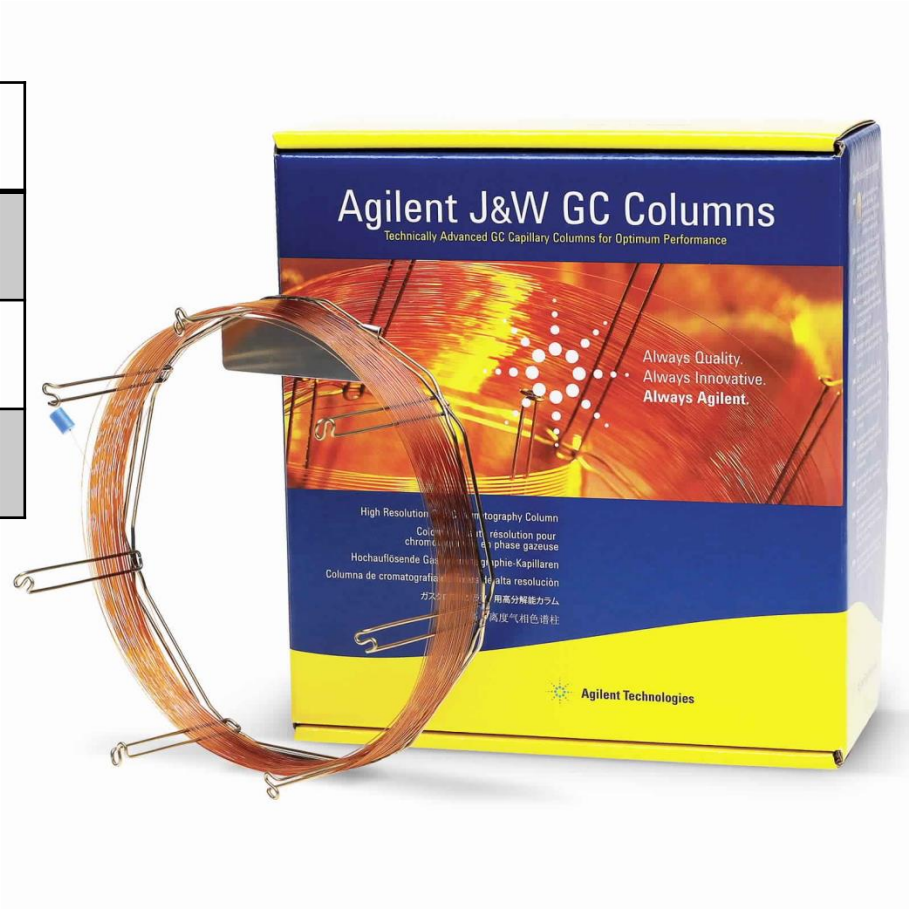
- Reduced column internal diameter double the efficiency and leads to better selectivity. This will increase retention time when using isothermal separations. such columns easily contaminated and suffer from peak broadening after routine work.

- Changes in film thickness effects retention of analyte species, interaction with the silica tubing (increased with increasing film thickness). Usually, thin films (0.10-0.25 µm)are used for trace analysis.

A common GC-COLUMN/ HP 5 MS

- Stationary phase coating is : 5 %-Phenyl-methyl poly siloxane

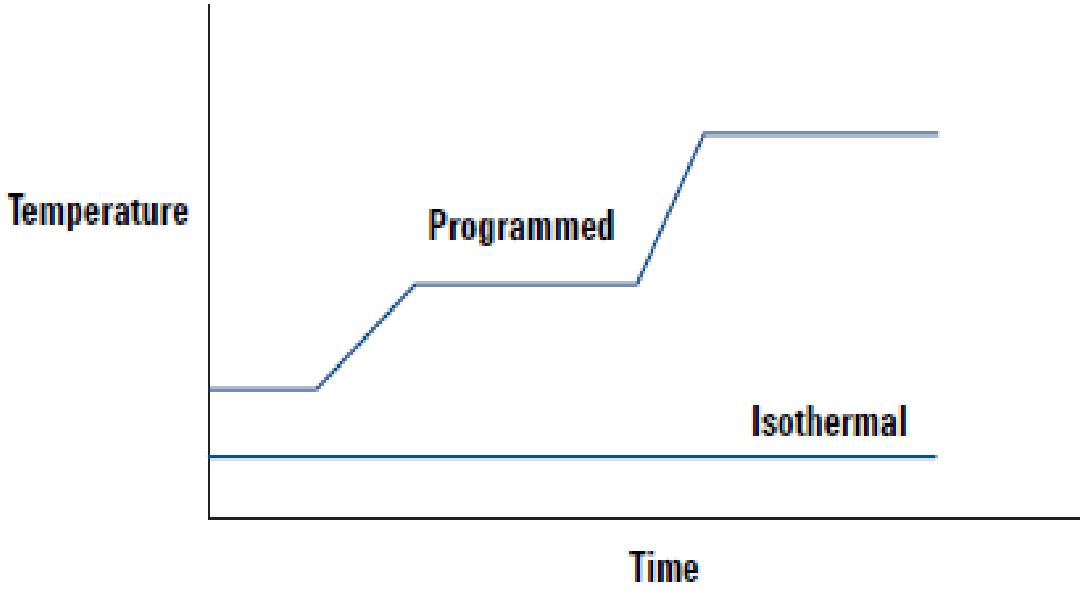
Internal diameter	**0.25 mm**
Length	30 m
Film	0.50 µm
Temperature Limits	-60 - 325/350 °C



Column Temperature

- Every capillary column has its working temperature range that maintains the stateof the used coated stationary phase.

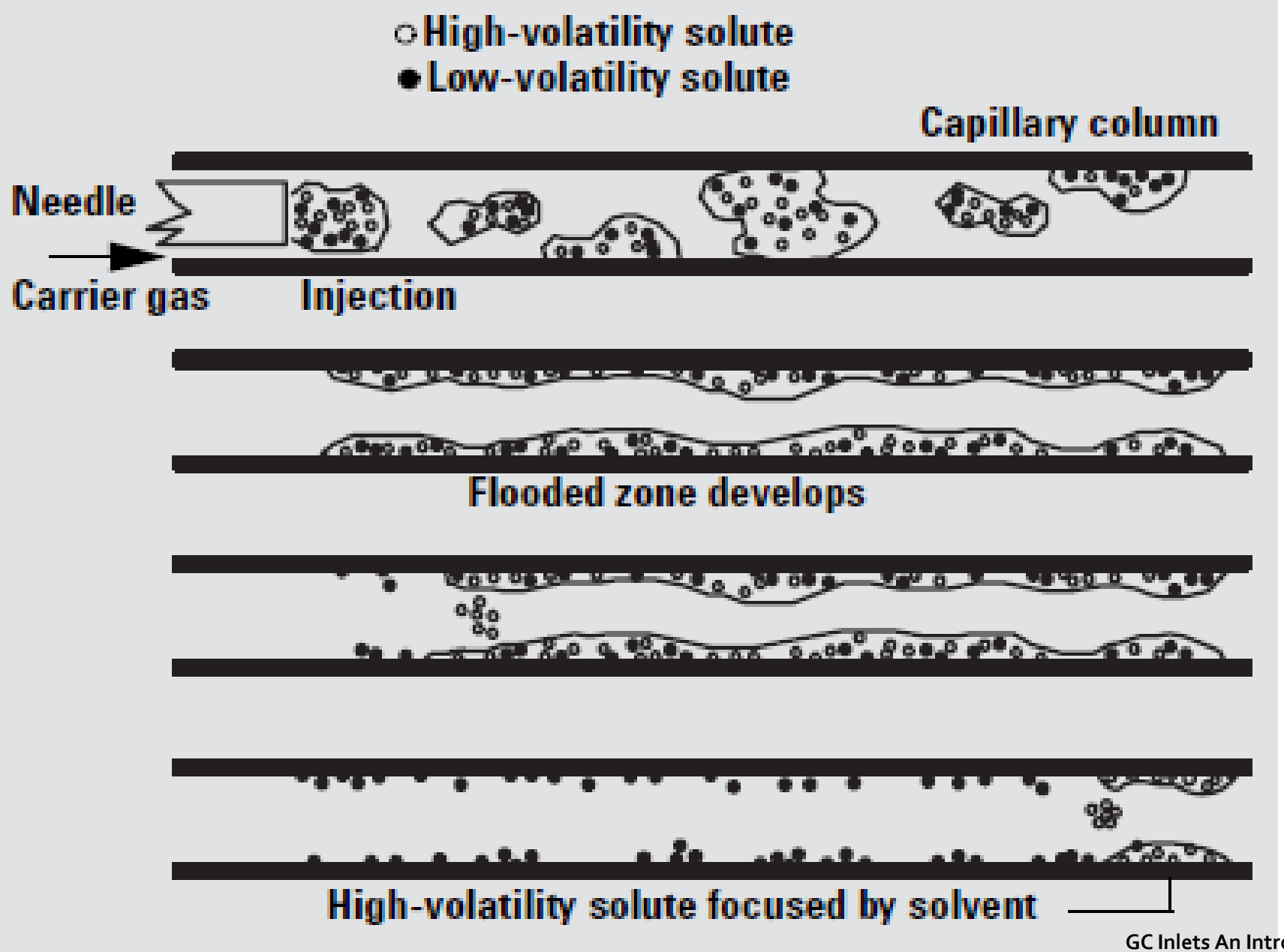
- Working on GC column should be below its maximum temperature.



Column starting temperature

- The **starting temperature** for the column (oven temperature) should be much lower than the lowest boiling points of tested analytes.
- For analytes of low boiling points, the adsorption of both solvent and these analytes is required at the front column inlet (starting column temperature of 20 C lower than the used solvent) which will subsequently enhance its peak shape.
- While, for highly boiling points analytes (late eluting peaks), adsorption of the used solvent isn't required (it will cost a longer run time) only start with a temperature below the boiling points of these analytes but that give enough adsorption at the front column inlet (no peak tailing)

Column starting temperature



GC Inlets An Introduction, Agilent. 5958-9468

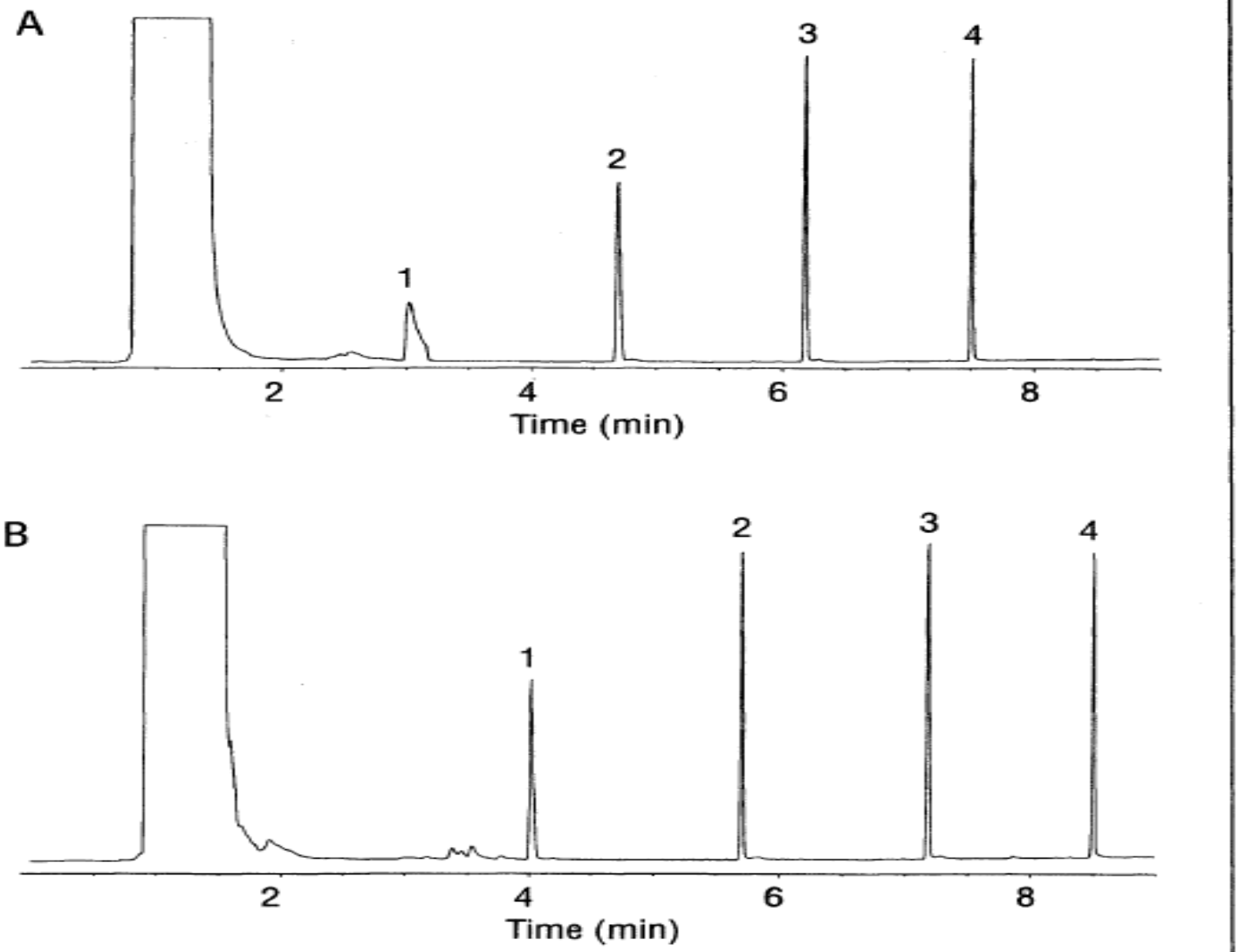
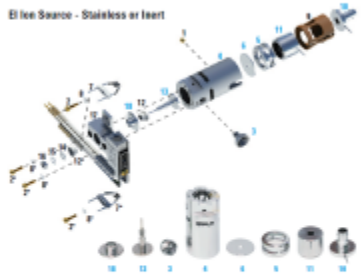


Figure 1. Conditions: column, DB-1 (15 m × 0.25-mm i.d., 0.25-µm film); injector, splitless (250°C, 0.5-min purge activation time); detector, FID (300°C); carrier gas, helium (30 cm/s); oven, (A) 70°C for 1 min, 70–210°C at 20°C/min (B) 50°C for 1 min, 50–210°C at 20°C/min. Peaks: 1 *n*-decane; 2, *n*-dodecane; 3, *n*-tetradecane; 4, *n*-hexadecane.

Journal of Chromatographic Science, Vol. 34, December 1996

GC-MS, GC-MS/MS

MASS SPECTROMETRY
for
pesticides residue analysis



Lecture
(2)

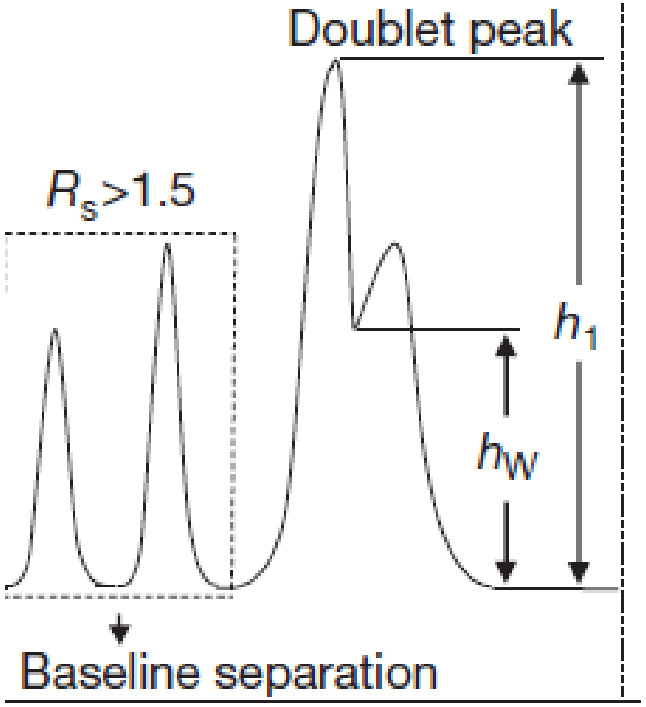
"Electron ionization and Chemical ionization"

Retention time, related terms and definitions

- Chromatogram : A plot of the detector response related to the effluent time.
- Mass spectrum: A plot of the intensities versus m/z of specific peak.
- Retention time t_R : Time taken by the carrier gas from the analyte injection to its completely detection.
- Dead time (holdup time) t_M : Is the time taken by the carrier gas from the point of injection to the detector.
- Adjusted retention time : = t_R - t_M
- Distribution constant K: The ratio of analyte concentration in the stationary phase to its concentration in the mobile phase $K = \frac{Cs}{Cm}$

Column Resolution, related terms and definitions

- Separation factor (selectivity factor): is the relative retention times of two adjacent eluted peaks, $\alpha = \frac{t_{R1}}{t_{R2}}$.
- Peak resolution based on their peak widths $Rs = \frac{2(t_{R1} - t_{R2})}{Wb1 + Wb2}$.
- Peak resolution based on their peak widths $Rh = \frac{h1 - hw}{h1}$
- The number of theoretical plates, $N = 16\ (\frac{tR}{W})^2$
- Efficiency (N) ,for thin film column, is related to column length (L) and internal diameter (dc), $N = L/dc$



https://doi.org/10.1016/B978-0-12-409547-2.14348-3

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Thank you

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