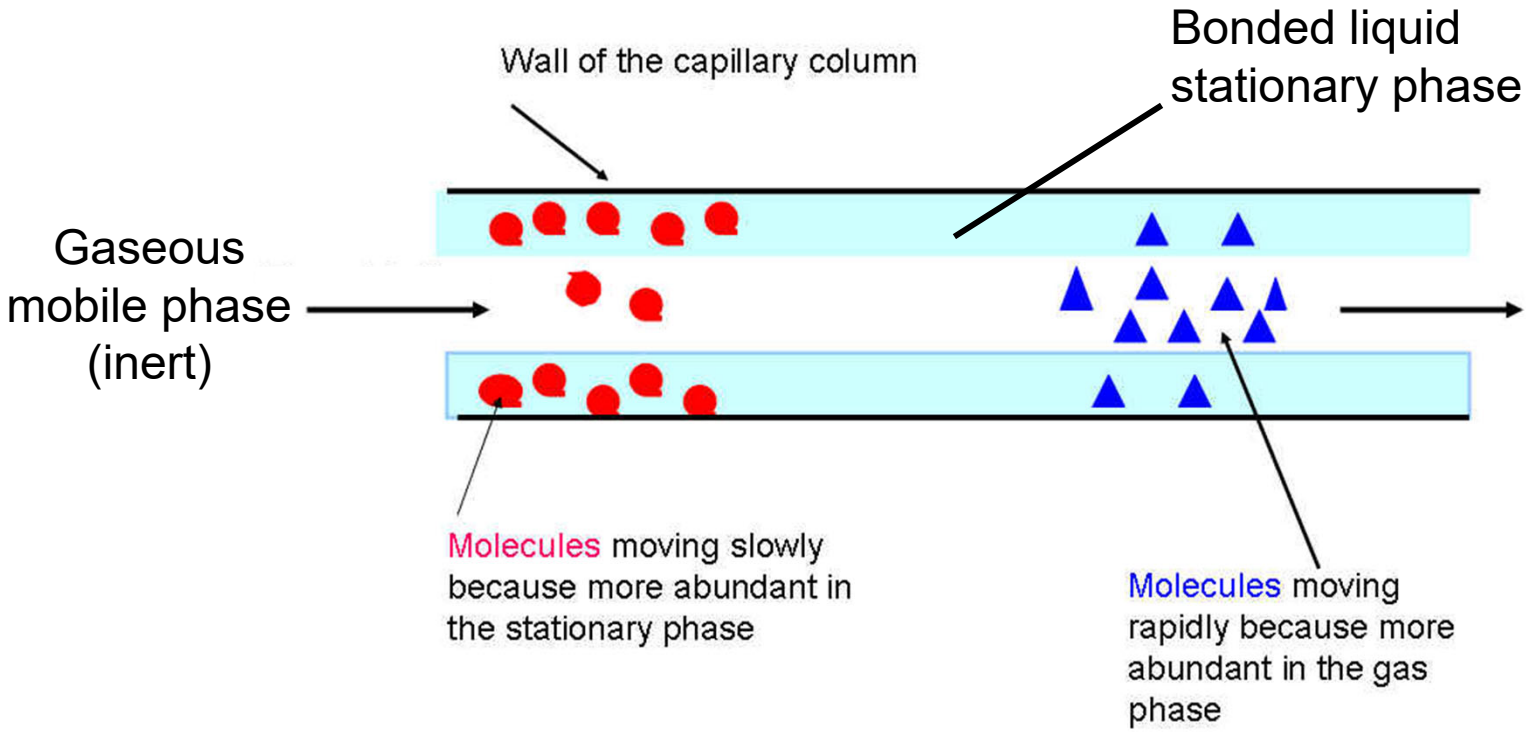
Introduction to Separation Science and Liquid Chromatography

Gas Chromatography

Gas Chromatography



- The gaseous mobile phase does *not* act as a solvent.
- Still refer to “partitioning” but temperature drives analytes into the gas phase.
- Analytes can remain in the vapour phase.
- Analytes can condense on stationary phase.
- Analytes can dissolve in the stationary phase.

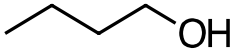
Poll

Question: Predict the order of elution of the following compounds on a GC column with a moderately polar stationary phase.

hexane (b.p. 68 °C)

octane (b.p. 125 °C)

1-butanol (b.p. 117 °C)



A. Hexane (first), 1-butanol, octane (last)

B. Hexane, octane, 1-butanol

C. Octane, hexane, 1-butanol

D. 1-butanol, octane, hexane

E. The butanol and octane will co-elute

Predict the order of elution for a series of compounds in gas chromatography.

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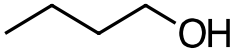

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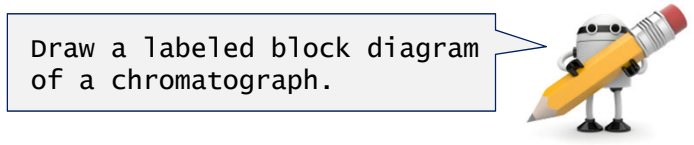
Predict the order of elution for a series of compounds in gas chromatography.

Gas Chromatographs

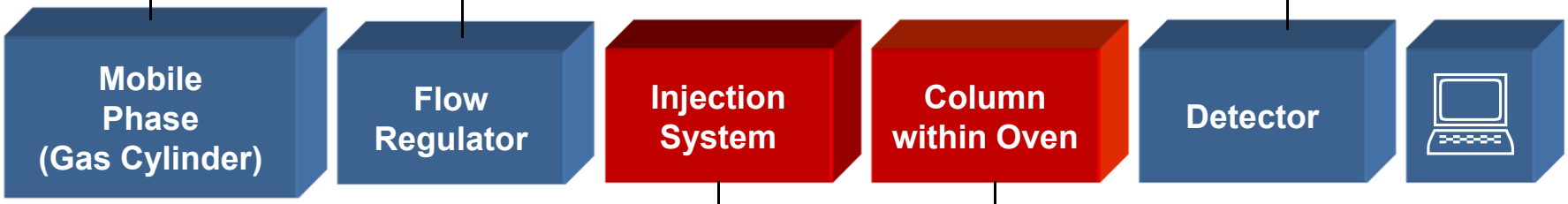
- Gaseous mobile phase
- Liquid or solid stationary phase
- Partitioning is generally independent of the mobile phase gas
- Temperature is critical



Gas must be dry and free of impurities.
Typically N_2, Ar, He or H_2.

Detect and quantify compounds eluting from the column.

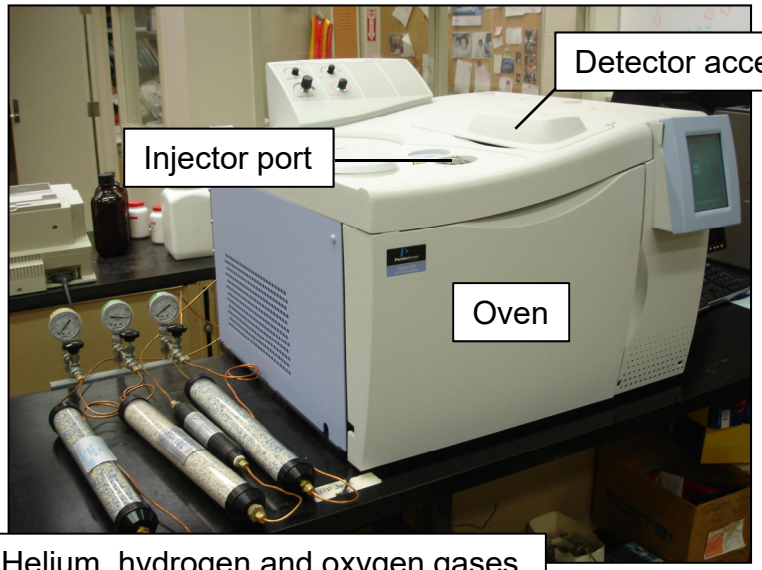
Controls the gas flow rate (volume/time).



Temperature controlled.
Often syringe based.
Introduce small amount of sample to column.

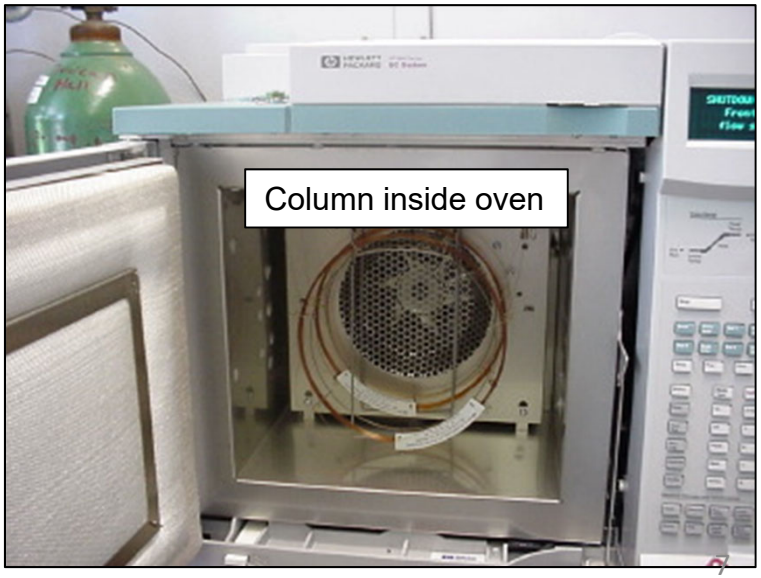
Programmable temperature control to help optimize separation.

Gas Chromatographs

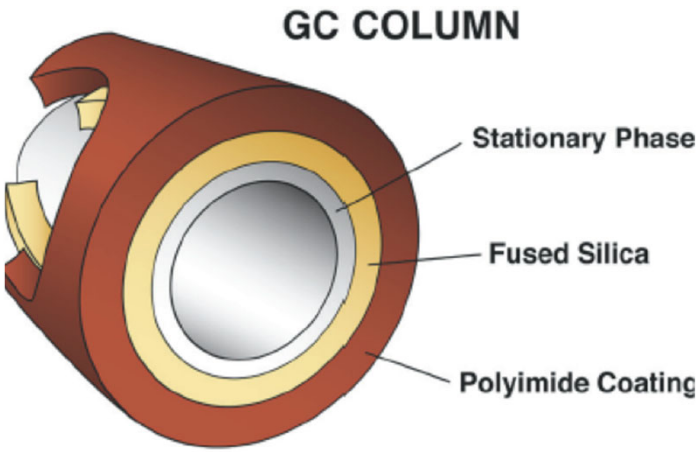


Helium, hydrogen and oxygen gases

Autosampler



Gas Chromatography Capillary Columns



Column: Long (20–50 m) and very narrow (20–300 µm)

Stationary phase: Bonded phase on inner wall of capillary column (0.1–5 µm thick)

FSCOT/WCOT: Fused silica/Wall-coated open tubular column

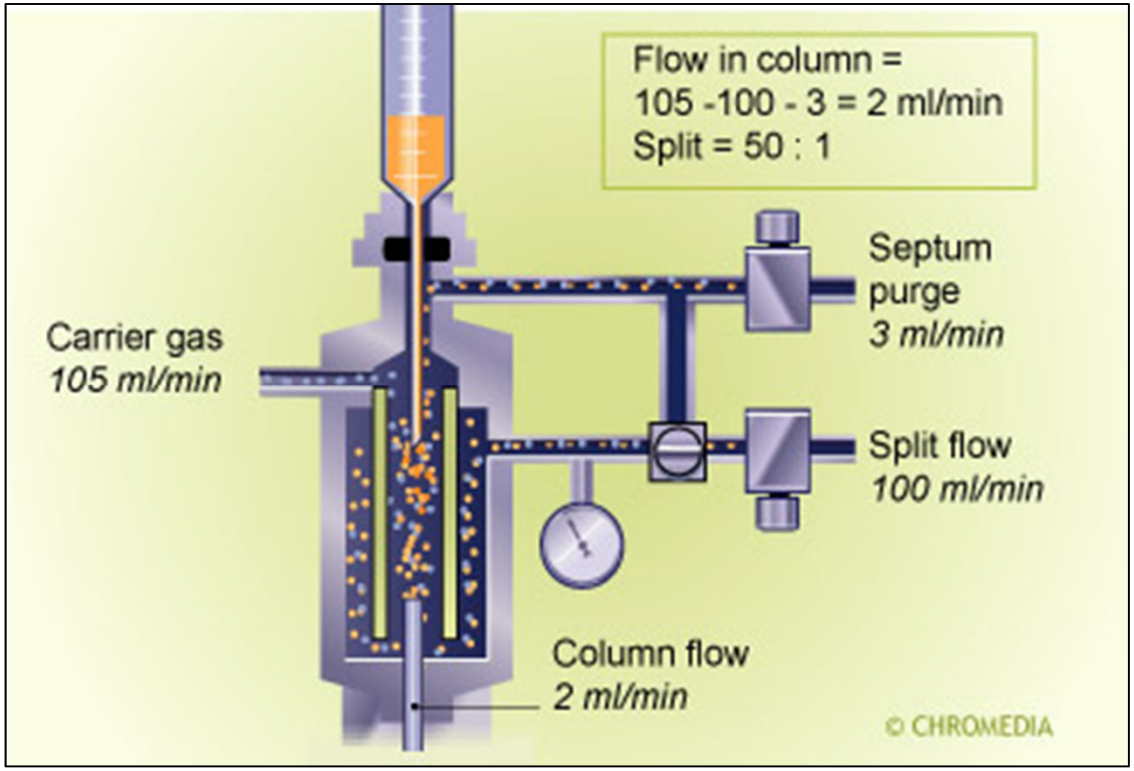
- High efficiency
- Low capacity
- Most common type

Other types of GC capillary columns change how the stationary phase is deposited in the capillary.

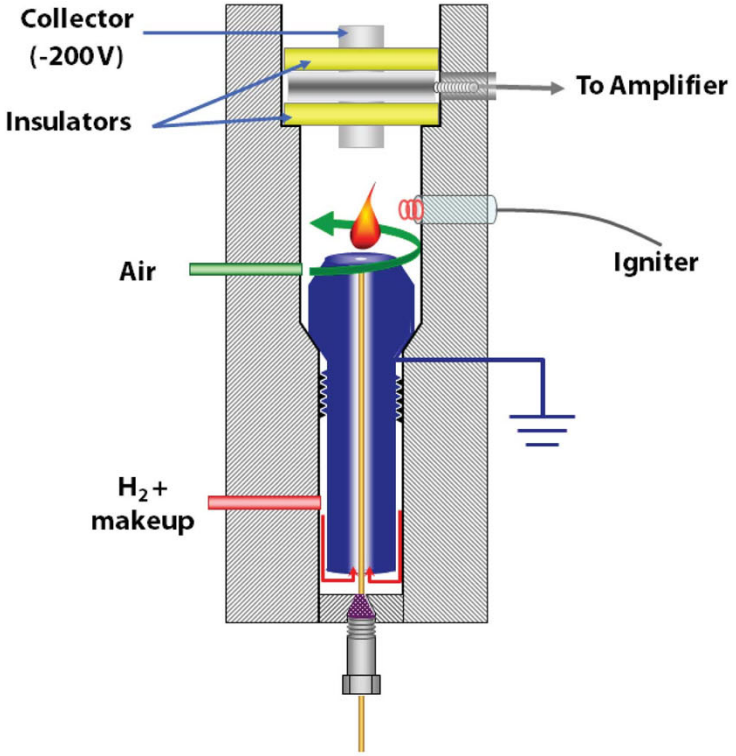
Packed GC columns are almost never used anymore.

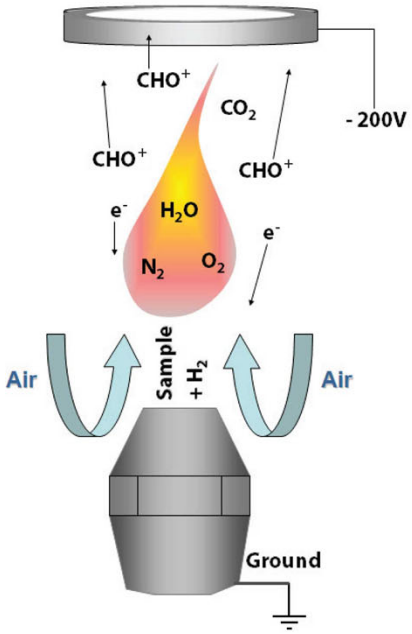
Split/Splitless Injection

- For narrow capillary columns, a splitter system is often required for sample injection.
- Inject liquid sample into a heated port using a syringe.
- Rapid vapourization of sample.
- Some of the sample is bled off to waste, some of the sample enters the column.
- Valve controls amount of sample introduced onto the column and amount split to waste.



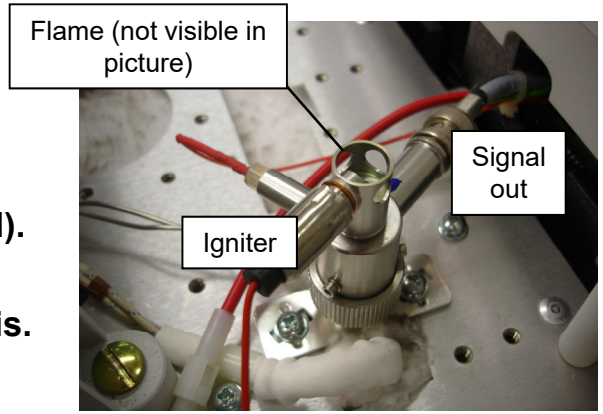
Flame Ionization Detector





- **Air/hydrogen flame.**
- **Hydrocarbons are burned to produce ions (the most prominent of which is formylium) and electrons.**
- **Ions and electrons are collected by an electrode assembly held at high potential.**
- **The current measured is proportional to the amount of material.**

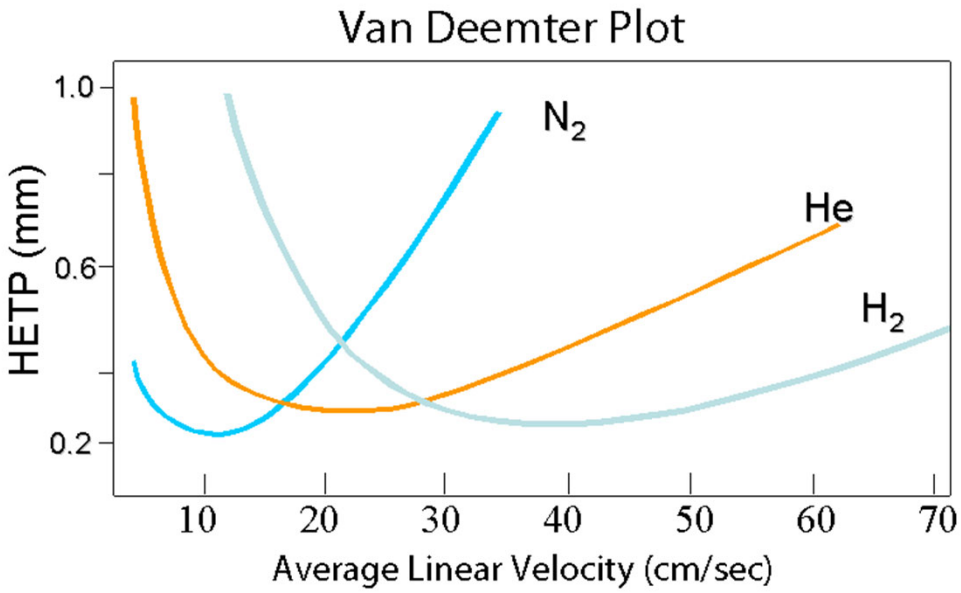
- **Insensitive to most inorganic compounds, *e.g.* O_2, N_2, SO_2, NH_3, CO_2.**
- **No response to fully oxidised carbons, *e.g.* carbonyl, carboxyl.**
- **Detection limits as low as 10^{-12} g C/s (picograms of carbon per second).**
- **Dynamic range of ~10^7.**
- **Most commonly employed "universal" detector. Good for trace analysis.**
- **Low cost, rugged, fast response.**



Optimizing GC Separations: Flow Rate

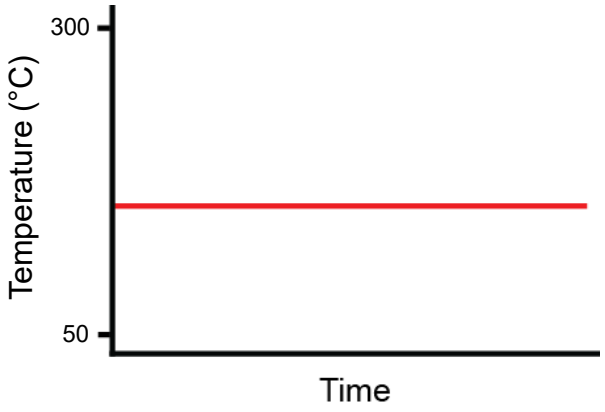
Optimization of flow rate is much more important for the efficiency of GC separations than LC separations.

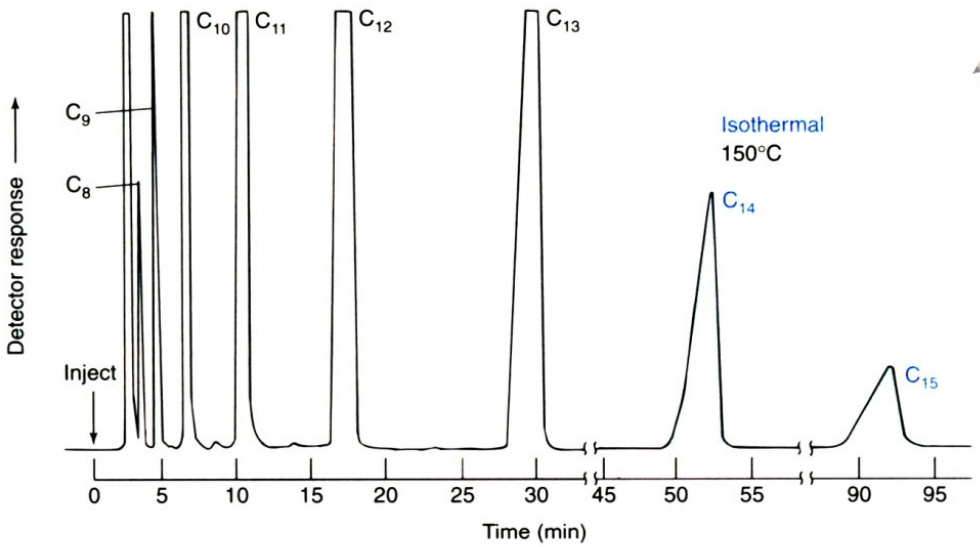
Note the more pronounced effect at higher flow rates.



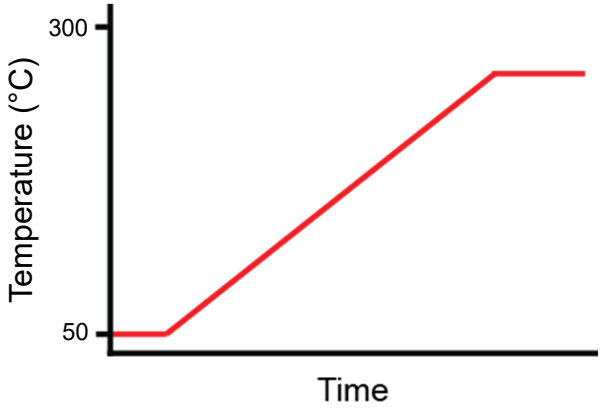
Optimizing GC Separations: Temperature

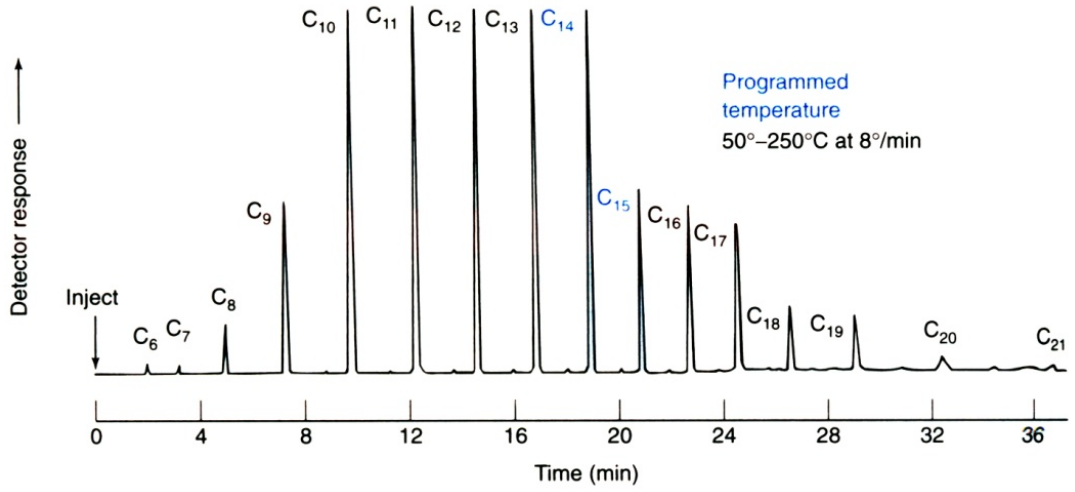
Isothermal





Temperature ramp





- Temperature programming is the GC equivalent of gradient elution in LC.
- Temperature has a much greater effect in GC than LC.

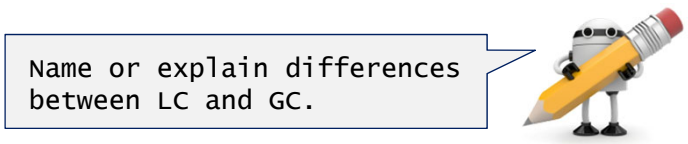
Comparison Between LC and GC

Liquid Chromatography

- Applicable to any soluble compound (*e.g.*, ions, small molecules, polymers, biomolecules)
- More versatile optimization than GC (can vary mobile phase and stationary phase)
- Purified compounds can be collected afterwards
- Often less sample preparation than GC

Gas Chromatography

- Requires volatile and thermally stable compounds
- Faster than HPLC (minutes *versus* tens of minutes)
- More sensitive than HPLC
- Better resolution than HPLC
- More “universal” detectors
- Less expensive



Summary Learning Objectives

- Explain the role of the partition coefficient in separation and extraction techniques and describe the physical and chemical principles underlying separations.
- Describe and compare the different types and modes of chromatography with reference to the stationary and mobile phases and elution methods:
    - Adsorption *vs.* partition (*vs.* size exclusion *vs.* ion exchange)
    - Reverse *vs.* normal phase
    - LC *vs.* GC
    - Isocratic/isothermal *vs.* gradient/temperature programming
    - Analytical *vs.* preparatory
- Predict the order of elution of compounds with either normal or reverse phase chromatography.
- Interpret a simple chromatogram to identify the dead time, retention time, and calculate the partition coefficient, retention factor, and/or resolution.
- Describe the relationship between retention time, peak area, resolution and analytical figures of merit in quantitative and qualitative analysis using chromatography.
- Draw and label a block diagram of LC and GC systems and explain the role of each component, the characteristics of columns, and the operation and capabilities of detectors.

Chemistry 211 provides an introduction to the concepts, problem solving skills, and technology important for the quantitative analysis of chemical systems.

Analytical concepts (Unit 1). Considerations for the design and analysis of analytical experiments, including common challenges, sources of error, and statistical tests.

Equilibrium and volumetric analysis (Unit 2). Classical titrimetric methods of analysis and essential concepts for understanding and manipulating equilibrium processes important to chemical systems.

Electrochemistry and Potentiometry (Unit 3). Basic theory of generating electric potentials from chemical energy and the use of potentials for the analysis of chemical systems.

UV-visible spectrophotometry (Unit 4). Basic theory, applications, and instrumentation related to the use ultraviolet and visible light for the analysis of chemical systems.

Separations and chromatography (Unit 5) . Application of equilibrium concepts to the analysis of complex chemical mixtures using modern instruments.

Final Exam

UBC CHEM 210
Final Examination
3:30 PM, Dec 8th, 2020

Duration: 2.5 hours (150 minutes)

25 multiple choice questions per unit.

25 × 5 = 125 questions (total)