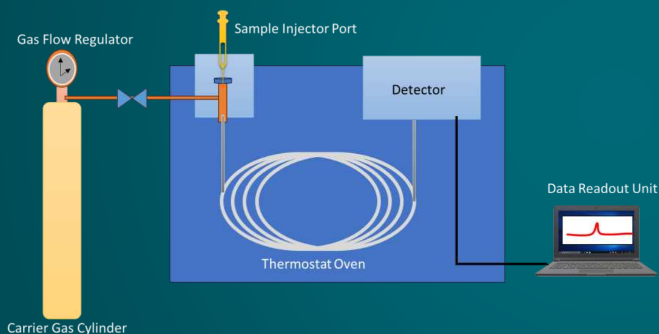
Gas Chromatography
(Part 1)

- ✓ Introduction
- ✓ Principle
- ✓ Theory
- ✓ Derivatization



Chromatography
Instrumental Analysis

Gas Chromatography

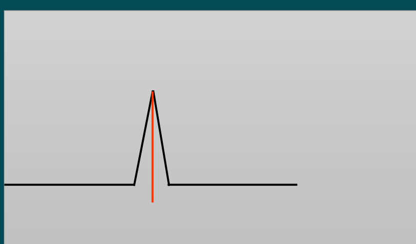
Introduction

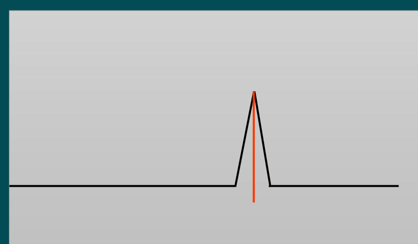
- In early 1900s, Gas chromatography (GC) was discovered by MS Tsvett as a separation technique to separate compounds.
- It is separation techniques used to analyze volatile substances in the gas phase
- Means the mobile phase is a gas and the components are separated as vapors.
- In gas chromatography, the components of a sample are dissolved in a solvent and vaporized in order to separate the analytes by distributing the sample between two phases: a stationary phase (Liquid or solid) and a mobile phase (Gas)
- Mobile phase/Carrier Gas: He, H2, N2, Ar (chemically inert not interact with component)
- Stationary phase: either solid adsorbent (GSC) or liquid on a inert support (GLC)

Gas Chromatography

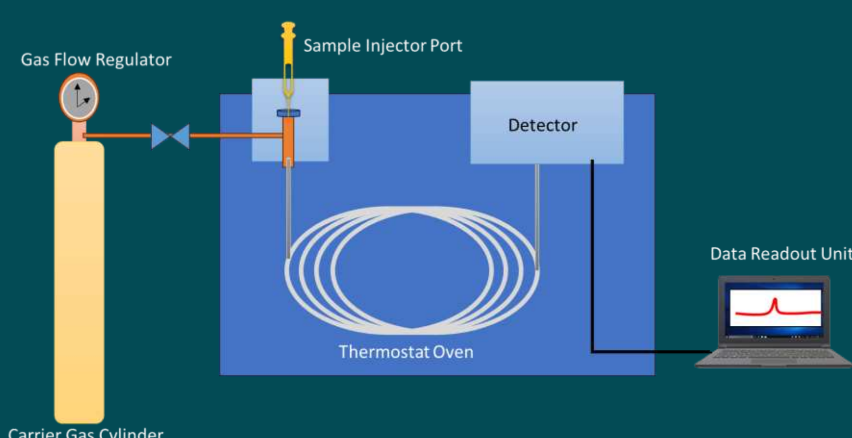
Principle

- Partition is the main principle of gas chromatography (GLC)
- Distribution of component between mobile phase (gas) and stationary phase (liquid) depends on affinity
- Sample is converted into vapour and mixed with mobile phase. The component have a greater affinity for the stationary phase spend more time in the column and thus elute later and have a longer retention time (Rt) than samples that have a higher affinity for the mobile phase.





Gas Chromatography



Basic Parts of Instrument

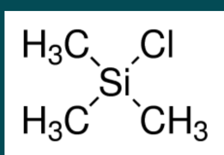
Gas Chromatography

Derivatization of Sample

- It is the technique of treatment of the sample prior to GC analysis to improve the separation and/or detection

1. Precolumn derivatization

- It is done to improve the separation by column. In this, components are chemically converted into more volatile and more thermostable derivative.
- E.g., Carboxylic acid, Sugars, phenols, alcohols can be converted into less polar compounds by using reagents like Bis trimethyl Silyl Acetamide reagent. They can also converted to acetyl or trifluoro acetyl derivatives



Gas Chromatography

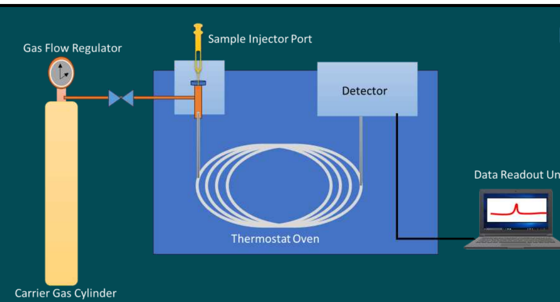
Derivatization of Sample

2. Postcolumn derivatization

- It is done to improve the detection by detector.
- In this, components may be converted in such way that their ionization or affinity towards electrons is increased

Gas Chromatography

(Part 2)

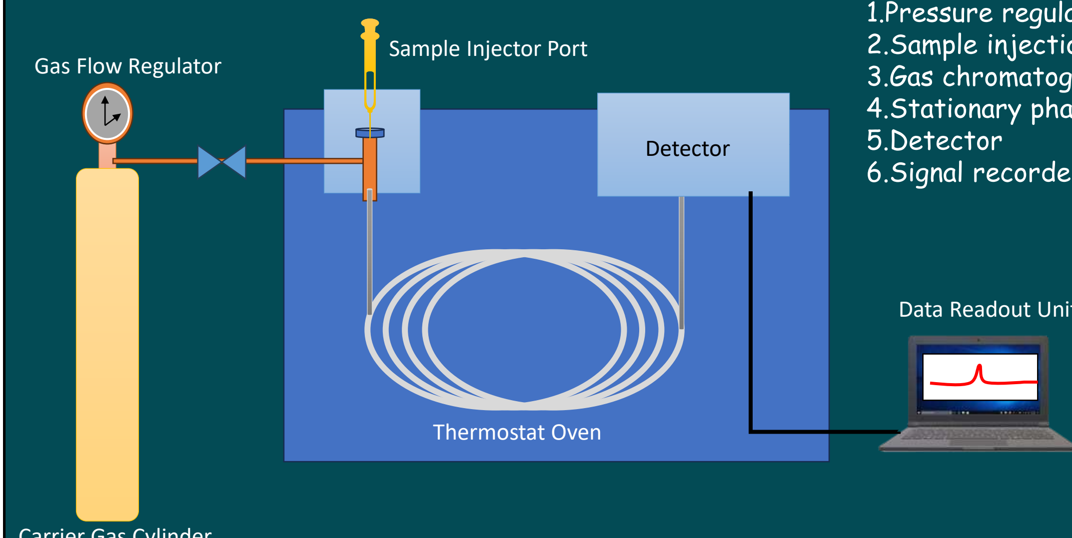


- ✓ Instrumentation
- ✓ Application

Chromatography

Instrumental Analysis

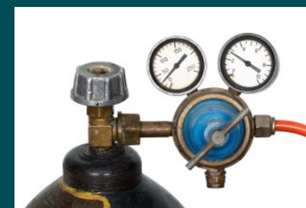
Gas Chromatography



1. Pressure regulator
2. Sample injection port
3. Gas chromatography column
4. Stationary phase
5. Detector
6. Signal recorder

Gas Chromatography

Instrumentation



1. **Pressure regulator**

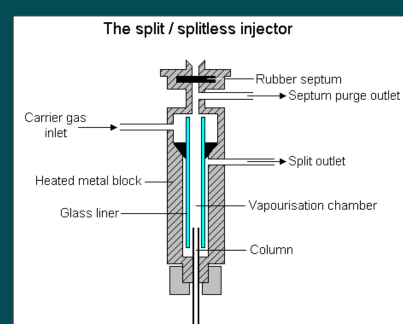
- Carrier gas from the tank passes through a toggle valve, a flow meter, (1-1000 ml/min), capillary restrictors, and a pressure gauge (1-4 atm).
- Flow rate is adjusted by means of a needle valve mounted on the base of the flow meter and controlled by capillary restrictors.
- Carrier Gas:
- The preferred carrier gas may be helium, argon, nitrogen, and hydrogen due to their high thermal conductivity.
- Helium is preferred for thermal conductivity detectors because of its high thermal conductivity relative to that of most organic vapors.
- N_2 is preferable when a large consumption of carrier gas is employed

Gas Chromatography

Instrumentation

2. **Sample Injection Port**

- Liquid samples are injected by a microsyringe with a needle inserted through a self-scaling, silicon-rubber septum into a heated metal block by a resistance heater.
- Gaseous samples are injected by a gas-tight syringe or through a by-pass loop and valves.
- Typical sample volumes range from 0.1 to 0.2 ml.

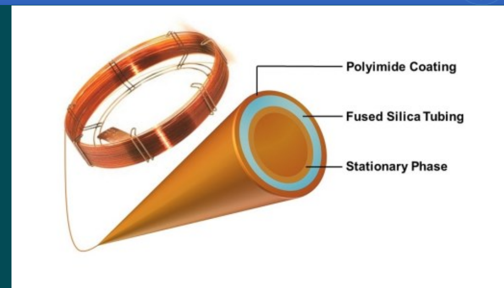


Gas Chromatography

Instrumentation

3. Columns

- The heart of the gas chromatography is the column which is made of metals bent in U shape or coiled into an open spiral or a flat pancake shape.
- Copper is useful up to 250^0
- Swege lock fittings make column insertion easy.
- Several sizes of columns are used depending upon the requirements.
- Preparative Column
- Analytical Column

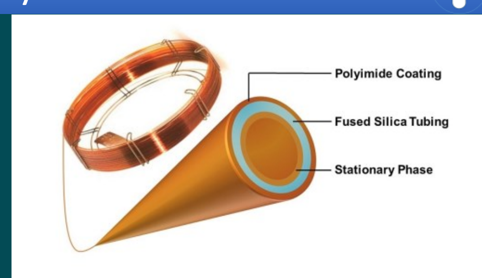


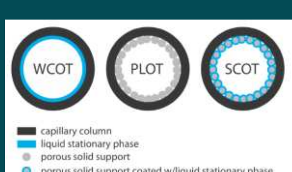
Gas Chromatography

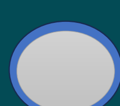
Instrumentation

3. Columns

- Analytical Column
- Length- 1-1.5 m, outer dm- 3-6 mm
- Made-up of glass or stainless steel
- Preparative Column
- Length- 3-6 m, outer dm- 6-9 mm
- Made-up of glass or stainless steel







Capillary columns are of three principal types. In a wall-coated open tubular column (WCOT) a thin layer of stationary phase, typically 0.25 nm thick, is coated on the capillary's inner wall. In a porous-layer open tubular column (PLOT), a porous solid support—alumina, silica gel, and molecular sieves are typical examples—is attached to the capillary's inner wall. A support-coated open tubular column (SCOT) is a PLOT column that includes a liquid stationary phase.

https://chem.libretexts.org/Bookshelves/Analytical_Chemistry/Instrumental_Analysis_%28LibreTexts%29/27%3A_Gas_Chromatography/27.03%3A_Gas_Chromatographic_Columns_and_Stationary_Phases

Gas Chromatography

Instrumentation

4. Stationary Phase

- Gas liquid chromatography can be available in almost an infinite variety of liquid partition materials.
- The liquid or stationary phase in gas chromatography can be divided into nonpolar, intermediate polarity, polar carbowaxes, and hydrogen bonding compounds like glycol
- Non-Polar – Parafin, squalane, silicone greases, apiezon L, silicone gum rubber. These materials separate the components in order of their boiling points.
- Intermediate Polarity – These materials contain a polar or polarizable group on a long non-polar skeleton which can dissolve both polar and non-polar solutes. For example. diethyl hexyl phthalate is used for the separation of high boiling alcohols.

Gas Chromatography

Instrumentation

4. Stationary Phase

- Polar – Carbowaxes – Liquid phases with a large proportion of polar groups. Separation of polar and non-polar substances.
- Hydrogen bonding – Polar liquid phases with high hydrogen bonding e.g. Glycol.
- Specific purpose phases – Relying on a chemical reaction with solute to achieve separations. e.g AgNO3 in glycol separates unsaturated hydrocarbons.

Stationary phase in gas chromatography

Type	Stationary phase
GLC	Squalene, silicon oil, nonpolar polyethylene, glycol, glass Teflon beads, etc.
GSC (usual)	Silica gel, alumina, charcoal, molecular sieve inorganic salts, mineral, porous polymers.
GSC (reverse-phase)	Silica alumina coated with organic or inorganic compounds or complexes.

Gas Chromatography

Instrumentation

5. Detector

A. Flame Ionization Detector (FID)

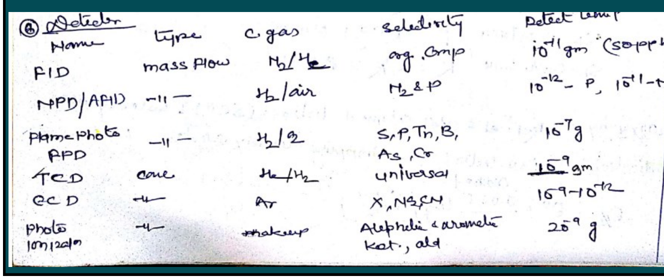
B. Nitrogen Phosphorus Detector or alkali flame-ionization detector (AFID)

C. Flame Photo Detector (FPD)

D. Thermal Conductivity Detector (TCD) of Katharometer

E. Electron Capture Detector (ECD)

F. Photo Ionization Detector (PID)



(8) Detector

Name	type	c. gas	selectivity	Detect limit
FID	mass flow	H_2/He	org. cmp	10^{-11} gm (S0ppb
NPD/AFID	—"—	H_2/air	H_2 & P	10^{-12} – P, 10^{-11}–N
Flame Photo FPD	—"—	H_2/O_2	S, P, Tn, B, As, Cr	10^{-7} g
TCD	conc	He/H_2	universal	10^{-9} gm
ECD	—"—	Ar	X, NO_2, CN	10^{-9}–10^{-12}
photo 10m/120m	—"—	makeup	Aliphatic & aromatic ket., ald	2×10^{-9} g

Gas Chromatography

Application

Table. Representative Applications of Gas Chromatography	
area	**applications**
environmental analysis	green house gases (CO_2, CH_4, NO_x) in air. pesticides in water, wastewater, and soil. vehicle emissions. trihalomethanes in drinking water.
clinical analysis	drugs blood alcohols
forensic analysis	analysis of arson accelerants detection of explosives
consumer products	volatile organics in spices and fragrances trace organics in whiskey monomers in latex paint
petrochemical and chemical industry	purity of solvents refinery gas composition of gasoline

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