CH 2252 Instrumental Methods of Analysis

Unit – V

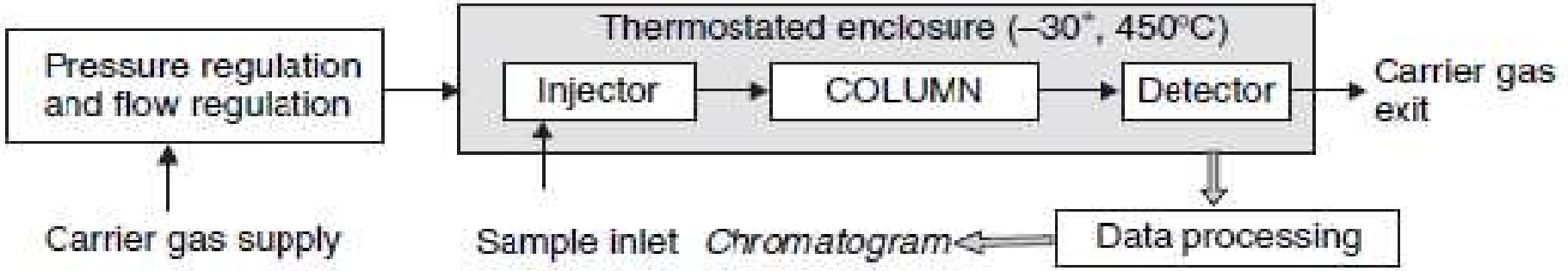
www.msubbu.in

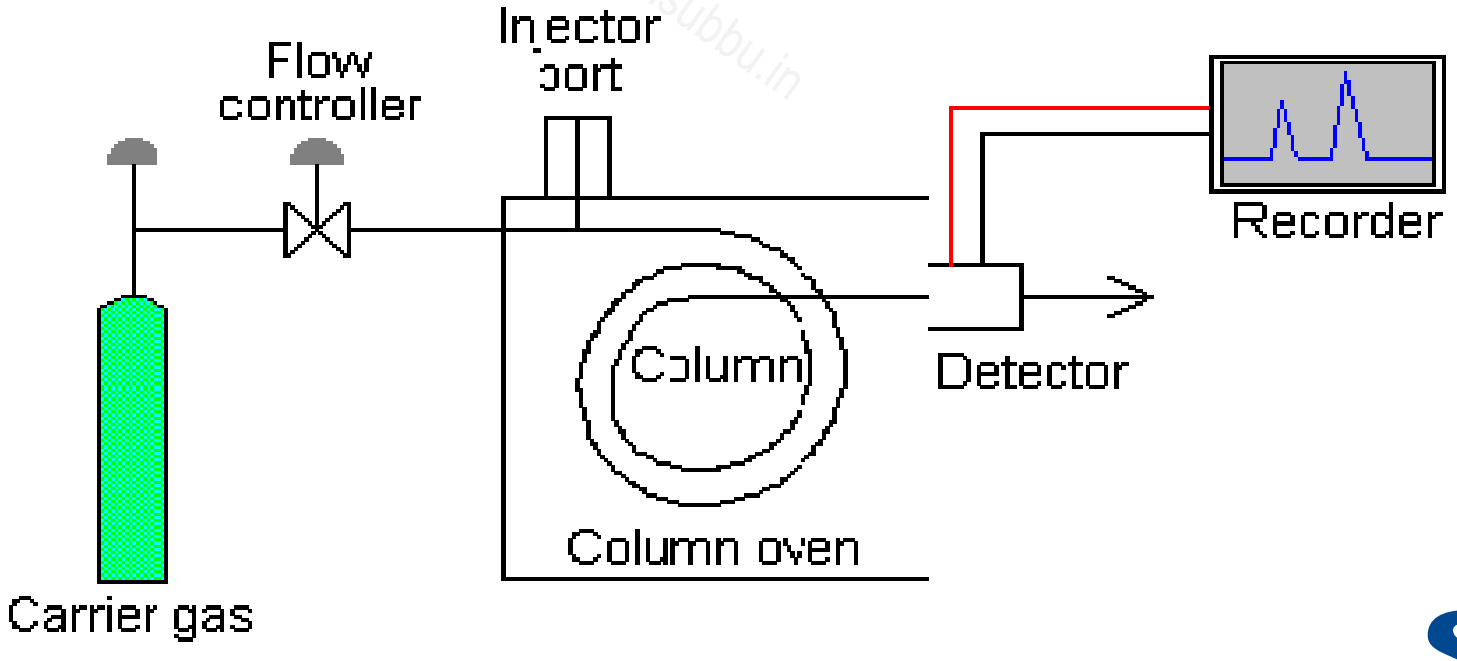
Gas Chromatography

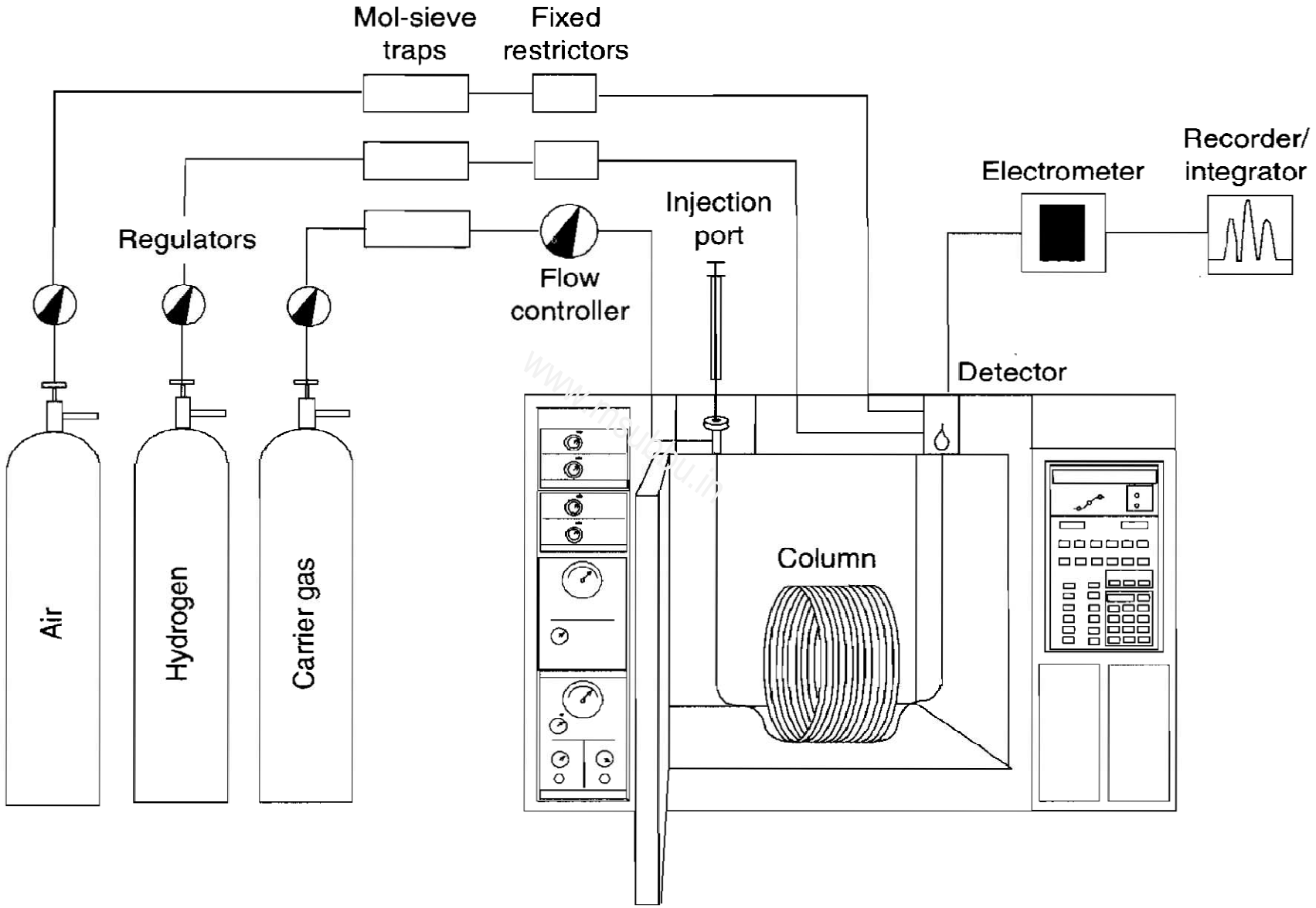
M. Subramanian

Assistant Professor
Department of Chemical Engineering
Sri Sivasubramaniya Nadar College of Engineering
Kalavakkam – 603 110, Kanchipuram (Dist)
Tamil Nadu, India
msubbu.in@gmail.com

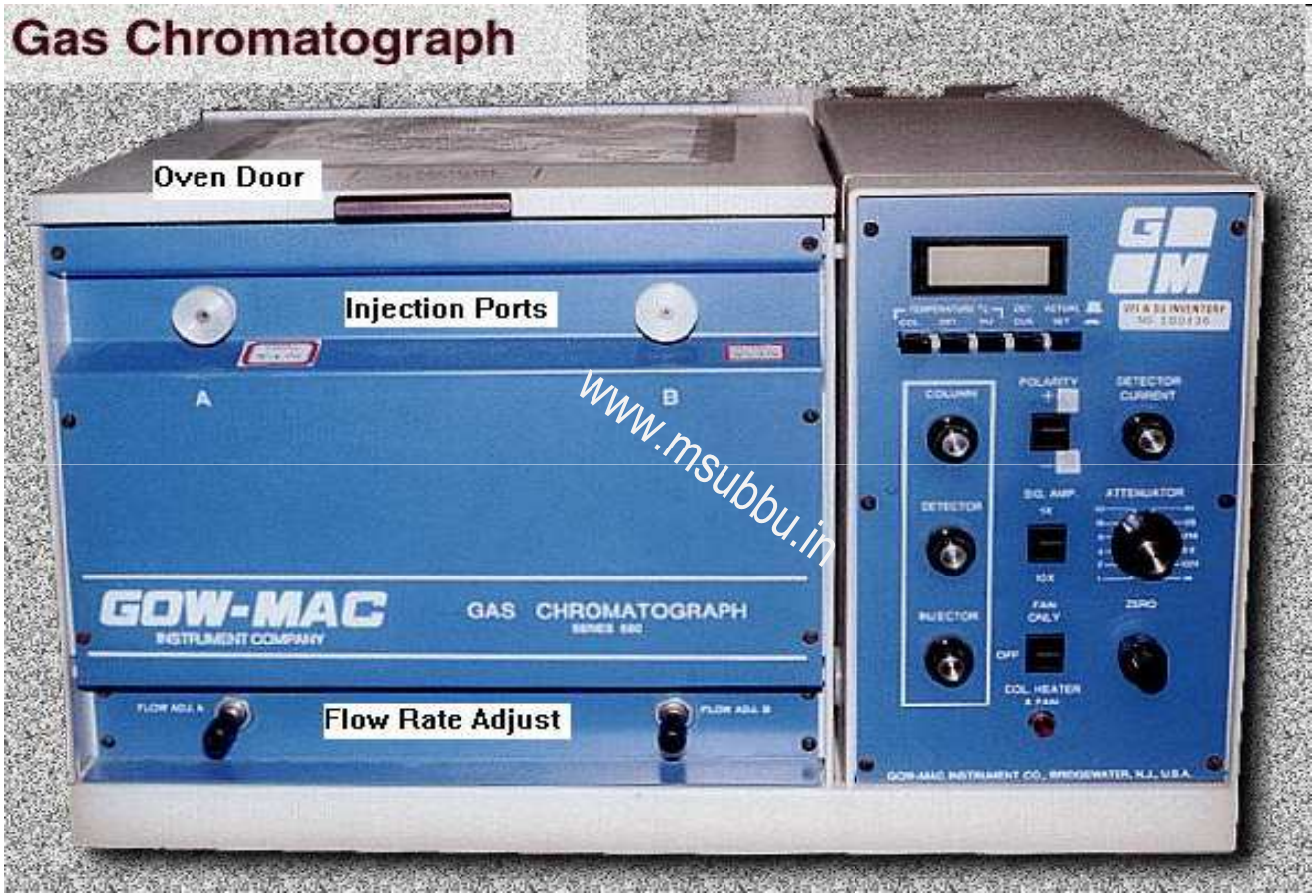
Gas Chromatography






Mol-sieve traps
Fixed restrictors
Regulators
Flow controller
Injection port
Electrometer
Recorder/ integrator
Detector
Column
Air
Hydrogen
Carrier gas

Gas Chromatograph




5765
nucon
GAS CHROMATOGRAPH
AIMIL CHROMATOGRAPHY DATA STATION
F.I.D.
BALANCE
SENSITIVITY
ATTENUATOR
MAINS
POLARITY
T.C.D.
COARSE
FINE
BALANCE
CURRENT
ATTENUATOR
MAINS
POLARITY
nucon
5765
PROGRAMMER CONTROLLER
SAMSUNG
www.msubbu.in
24 . 11 . 2007

Gas Chromatography

- The general concept of GC was put forward in a Nobel Prize winning paper by Martin and Synge in 1941, and implemented by James and Martin in 1952 under the name of vapor phase chromatography.

Gas Chromatography

- Stationary phase: solid or liquid; Moving phase: gas
- Always performed in columns
- Packed columns – accounts only for 10%; capillary columns – mostly used
- In contrast to packed columns, capillary columns may be of any length, typically 10–100 m, as there are no packing particles. The stationary phase is coated to the inside of the capillary wall in thicknesses ranging from 0.1 to 5 mm. Finally, the inside diameter of a capillary column ranges from 0.1 to 0.53mm
- Systems configured for packed columns have a similar appearance, except that the inlet, detector and column oven are designed to accommodate the larger diameter packed columns

Columns

- In GC, the column is the most important component as it is the interaction of the analyte with the column's stationary phase (and not the carrier gas) that permits separation of the analytes in the sample.

- There are two types of gas chromatography based on column characteristics:

- ***Gas-Solid Chromatography (GSC)*** in which a solid stationary phase physically adsorbs analytes leading to retention of the analyte on the column. This GC has limited use due to long retention of active or polar molecules and severe tailing of elution peaks. Its only real application is for specific low molecular weight species and it will not be discussed further in this section.

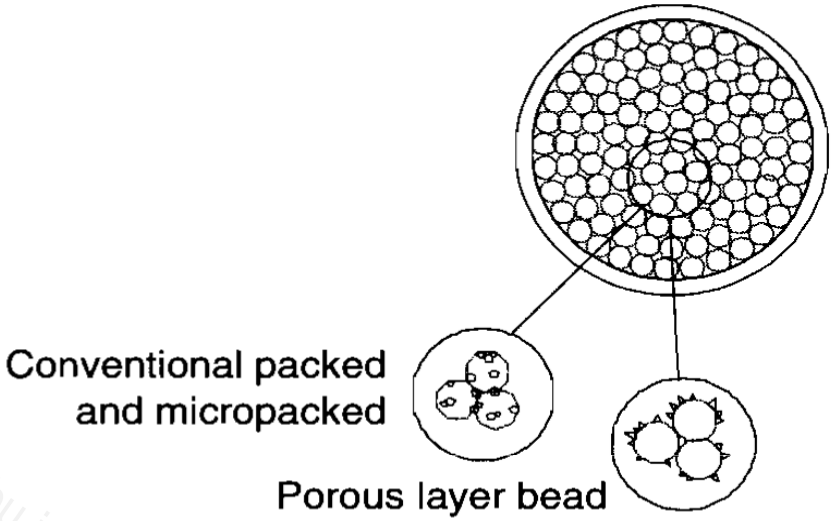
- ***Gas-Liquid Chromatography (GLC)*** in which a thin layer of a liquid stationary phase is immobilised inside the column. The sample analytes partition between the gaseous mobile phase and the liquid phase. GLC has been adopted as the most useful method and is generally now called ***Gas Chromatography (GC)*** although GLC is still mentioned in older textbooks.

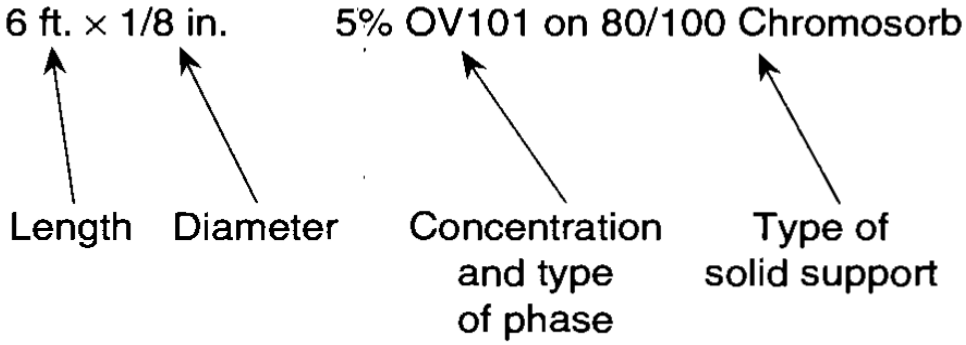
- Columns are normally coiled so that they can fit inside the oven of a GC.

Packed Columns

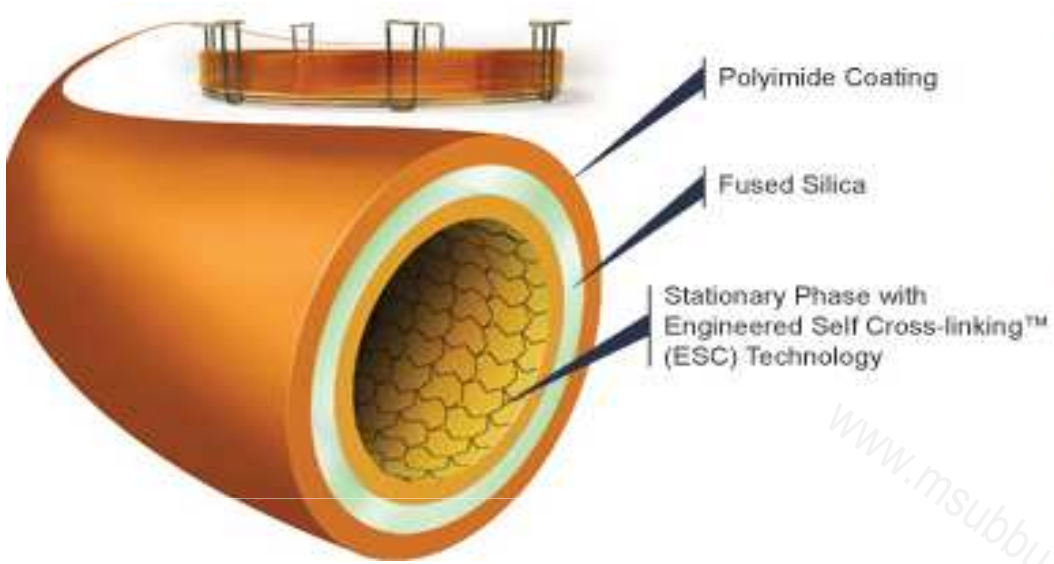
- Metal tubing 1/16 to 1/4 in. in diameter, generally made of copper or stainless steel

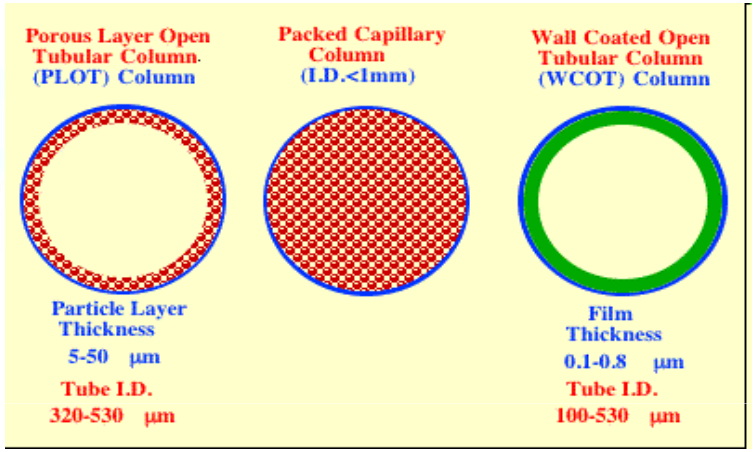
- Stationary phase: high boiling liquids dispersed on a particulate solid support





Capillary Columns





• The material of choice for the construction of capillary columns is fused silica, a highly purified and inert material. An outside protective coating, called polyimide, affords strength and flexibility.

•The most widely used columns in gas chromatography are referred to as **WCOT** (wall coated open tubular). These are capillary columns in which a liquid stationary phase is coated directly onto the column wall forming a thin film. 0.1 – 0.8 µm thick.

Characteristic	Packed Column	Capillary Column
Length (m)	1 - 6	10 - 100
Inside Diameter (mm)	2 - 4	0.1 – 0.3
Coil Diameter (cm)	15	10 - 30

Carrier Gas

- For packed column GC – nitrogen is the most commonly used carrier gas
- For capillary column GC – helium is the most common gas, followed by hydrogen and nitrogen

Samples to GC

- Liquid samples: micro-syringe, 0.1 – 10 μl; 1 μl being the most common
- Gas samples: gas-tight syringe, few μl to 1 ml or more
- Solid samples: dissolved into an appropriate organic solvent prior to injection as a solution

Detectors

- Thermal conductivity detector (TCD); also known as *katharometer*
  - it is a classical detector for both packed and capillary columns.
  - uses Wheatstone bridge circuit
  - it is most universal, but least sensitive, with concentration detection limits of about 10 ppm

- Flame ionization detector (FID)
  - For organic compound analysis, which often requires better sensitivity than TCD can provide, flame ionization is the detector of choice.
  - Flame ionization detector (FID) is also one of the classical gas chromatographic detectors and is used with both packed and capillary columns.
  - FID is significantly more complex than TCD, requiring fuel and oxidant gases, along with the carrier gas
  - In a FID, a hydrogen-air flame, with a temperature approximately 20001C is used to decompose organic analytes, producing carbon dioxide

Other Detectors

- Electron capture detector (ECD) – for detecting halogenated compounds (Cl, Br, F) such as pesticides uses this detector

GC operating procedure

- Preheat Detectors/Injector Ports
- Setting flow parameters
- Turn Gases On
- Ignite the Detectors
- Zeroing the Baseline
- Programming Column (Oven) Temperature
- Injecting a Sample
- Analyzing Chromatography
- Record Settings
- Shut Down

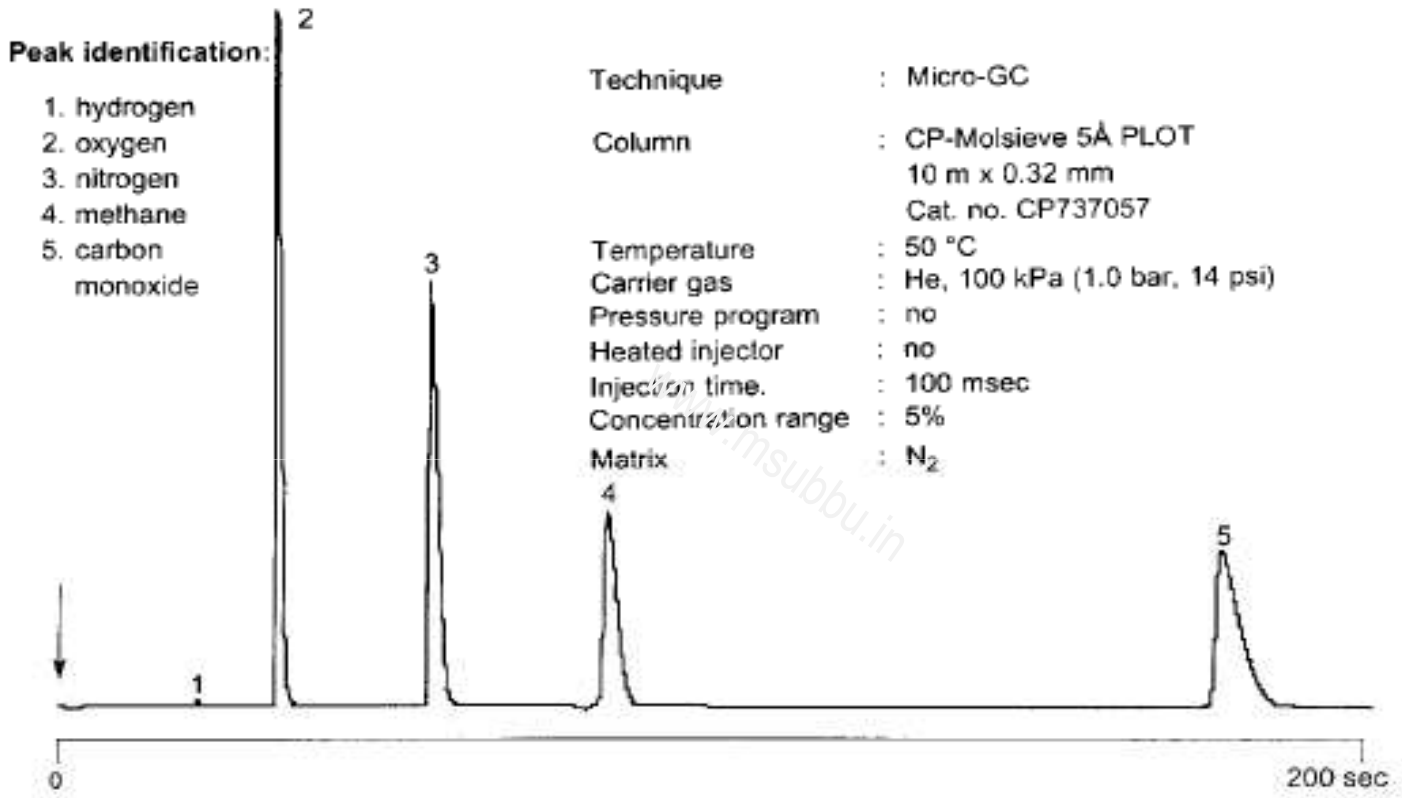
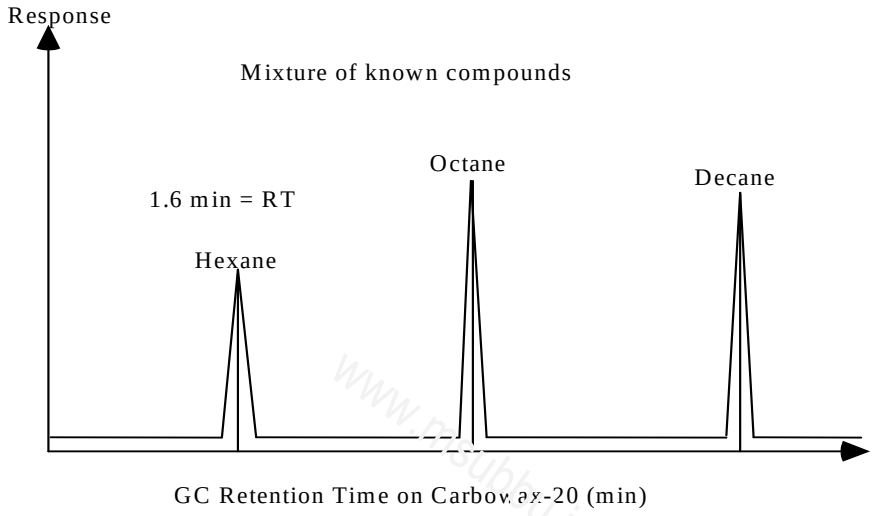
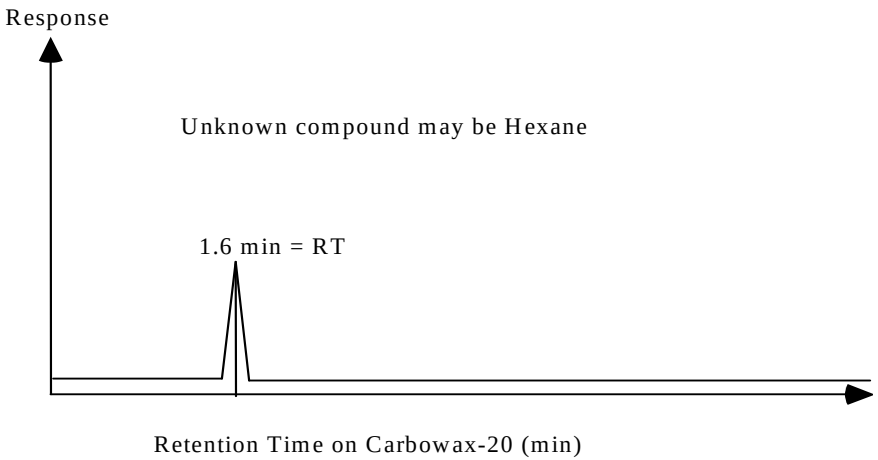


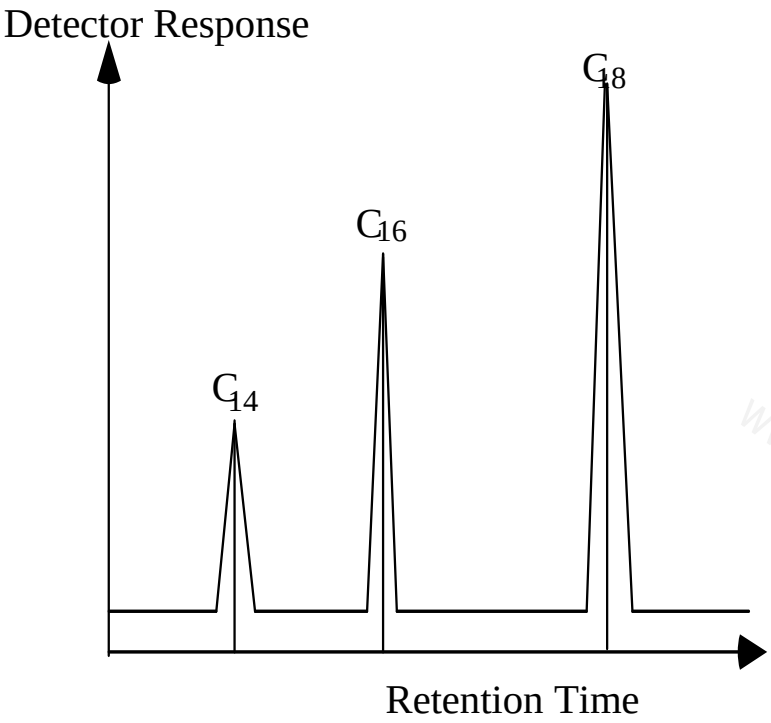
Figure 12.4 Molecular sieve PLOT column separation of fixed gases with TCD detection. (Used with permission from Varian Chrompack Inc.)

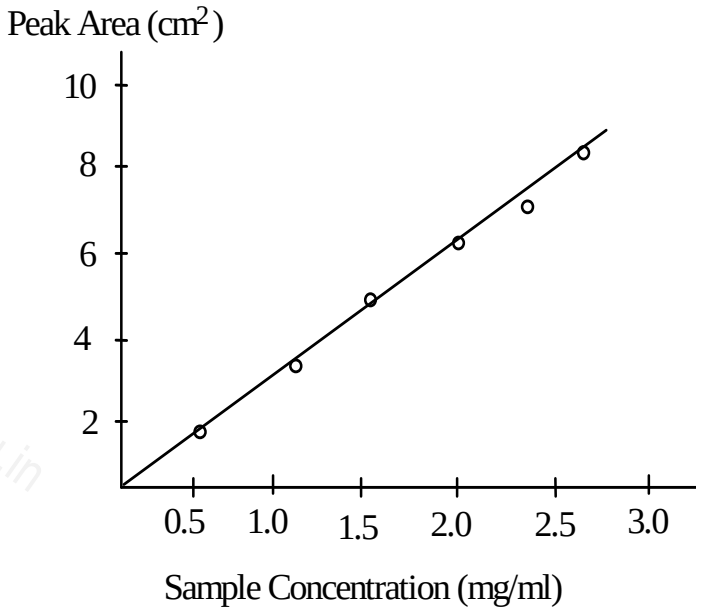
Qualitative Determination





Quantification





$$\text{The content \% of } C_{14} \text{ fatty acids} = \frac{C_{14}}{C_{14} + C_{16} + C_{18}} * 100$$

= the content % of C_{14} fatty acids

Advantages of GC over LC

- Only low pressures (5–250 psi) of gas were needed to achieve flow rates suitable for optimal separations. Such pressures could be contained by simple metal or glass tubing and standard nut and ferrule fittings

- Large dimensions of packing materials is allowed (of the order of 1 mm, in comparison to LC which requires much smaller particles of 1 to 10 μm)

- Temperature can be easily varied to control the partitioning of phases