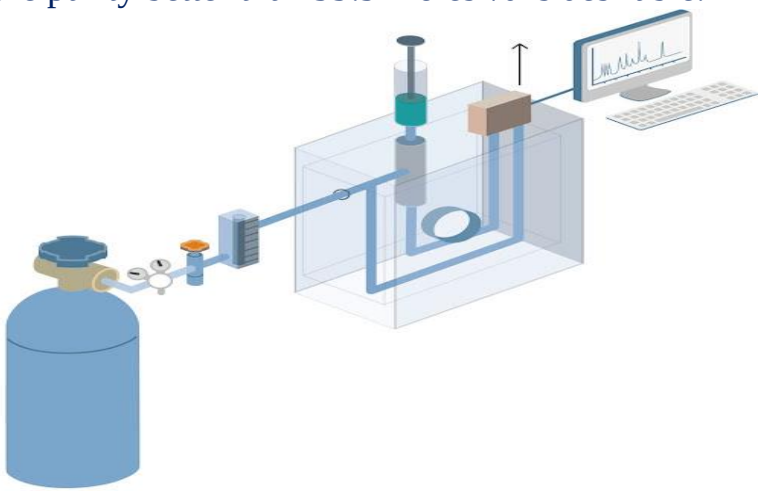
INSTRUMENTATION OF GAS CHROMATOGRAPHY

1) CARRIER GAS:

a) It should be chemically inert and should not interact with sample or stationary phase.

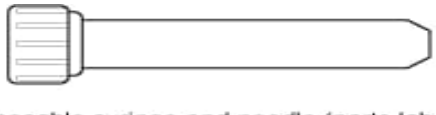
b) It should be suitable for detector to be utilised and the type of sample analysed.

c) It should give best column performance constituent with the desirable speed of analysis. Purity of carrier gas is very important in gas chromatography, usually the purity better than 99.9 moles % is desirable.

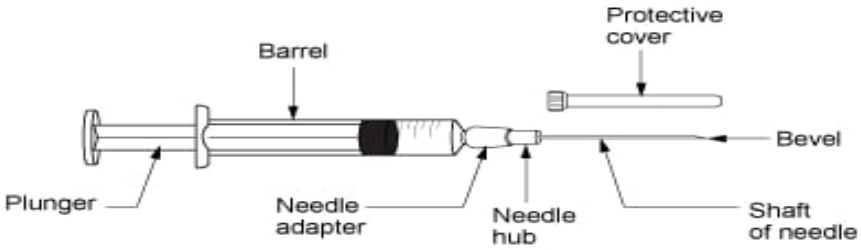


2) SAMPLE INJECTION SYSTEM:

The temperature of sample injection port is kept 2050^0 above the column temperature. Gases can also be injected by similar syringes which have gas tight plunger but are of larger capacity (1 to 10 ml).



Disposable syringe and needle (parts labelled)

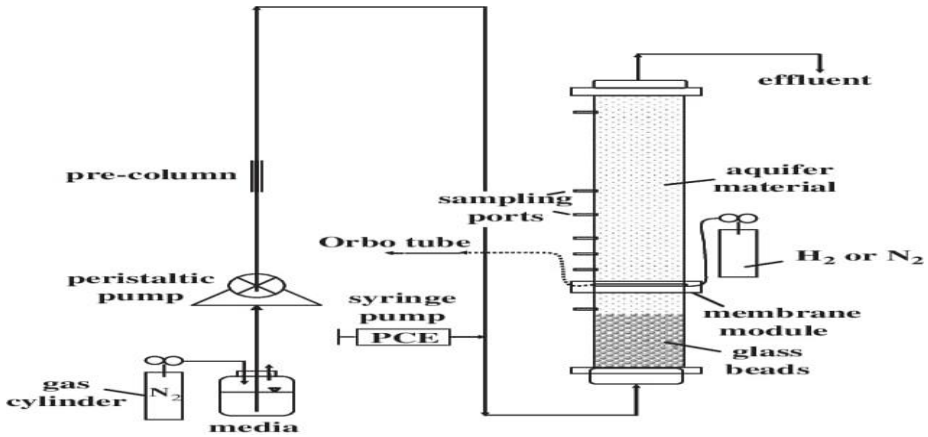


3) COLUMN TECHNOLOGY:

i. THE COLUMN: There are two types of column shapes as coiled helix and U-tube type. Tues may be 2-10 mm in diameter and from 2-4 meters in length Gas is frequently used in U-tubes.
Example: biological product.

ii. SUPPORT MEDIUM: Most common support are available from diatomaceous earth. Glass beads, porous polymer, unglazed tiles, sand, fluorinated resins etc.

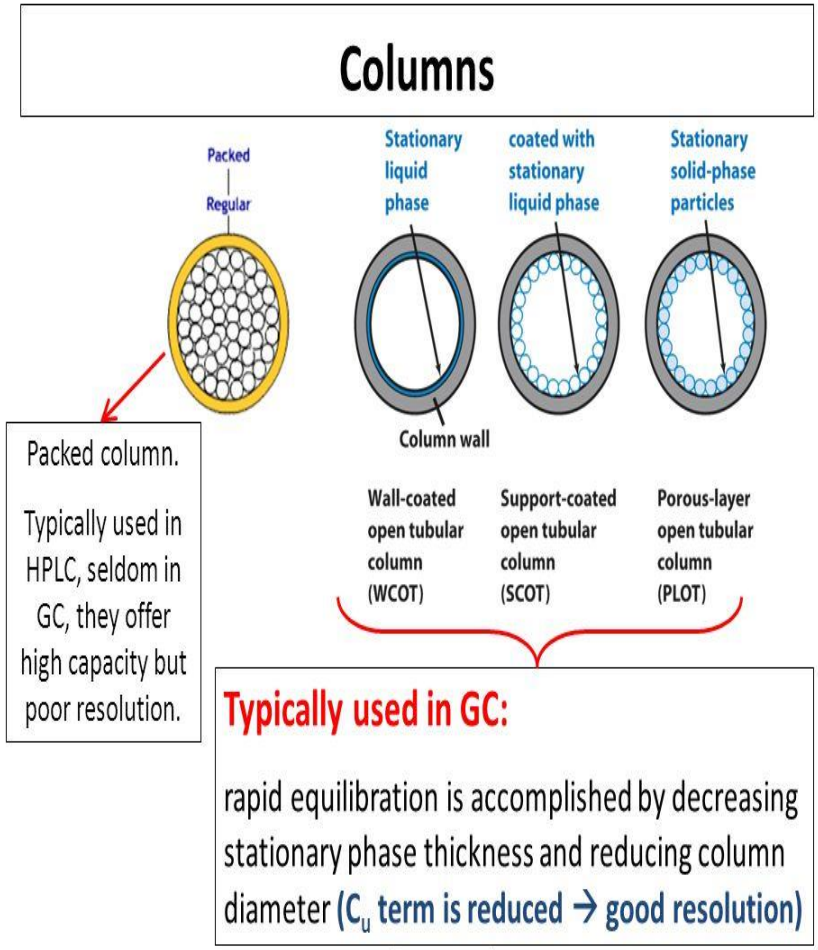
iii. LIQUID PHASE:



IV) PREPARATION OF CHROMATOGRAPHIC COLUMN:

i. Packed columns – These are prepared by packing metal or gas tubing with granular stationary phase.

ii. Open tubular columns – These columns are called as capillary or glory columns and are prepared from long (100 to 130 ft) capillary tubing.



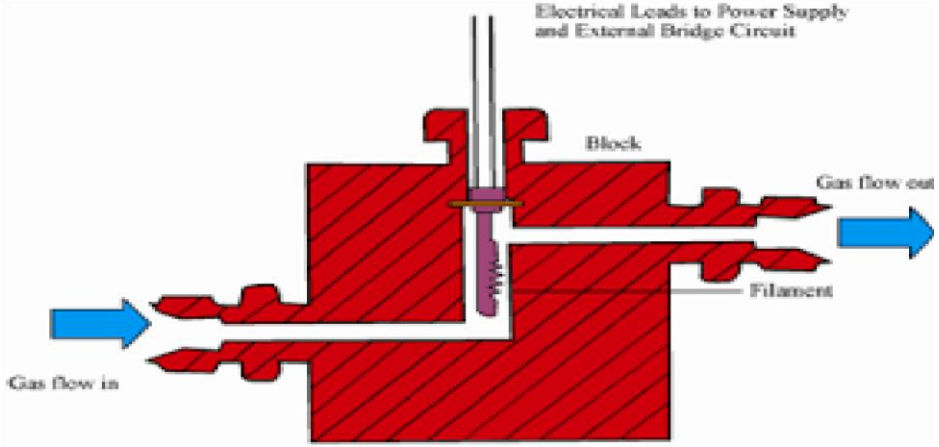
4) DETECTOR:

Thermal conductivity detector: (Katharometer).

Principal = It is based on thermal conductivity difference between carrier gas and that of component.

Generally helium with high thermal conductivity and inertness is the most preferred carrier gas in this case.

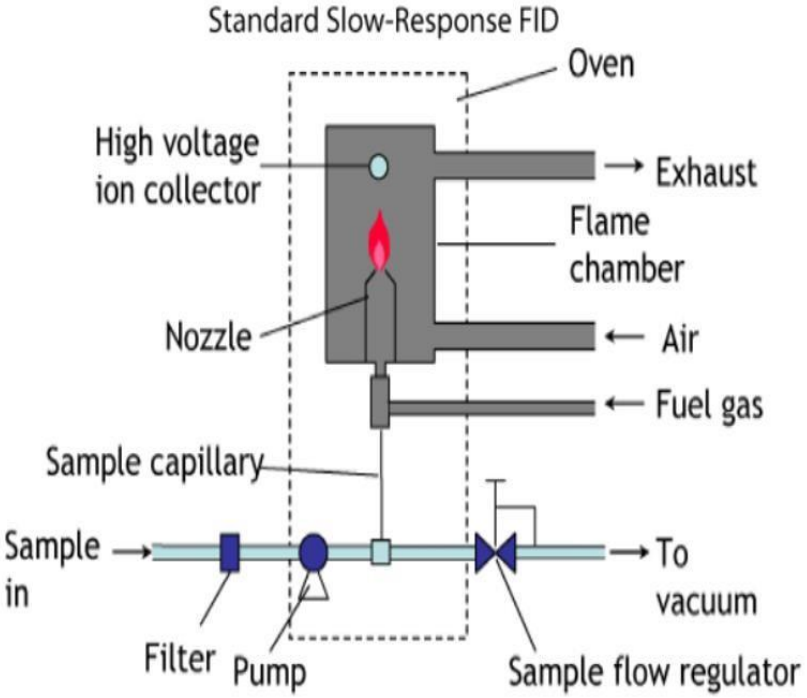
The conductivity has two channels through the cell, a sample channel and a reference channel.



FLAME IONIZATION DETECTOR: The most organic compound readily pyrolyse when introduced into a hydrogen oxygen flame.

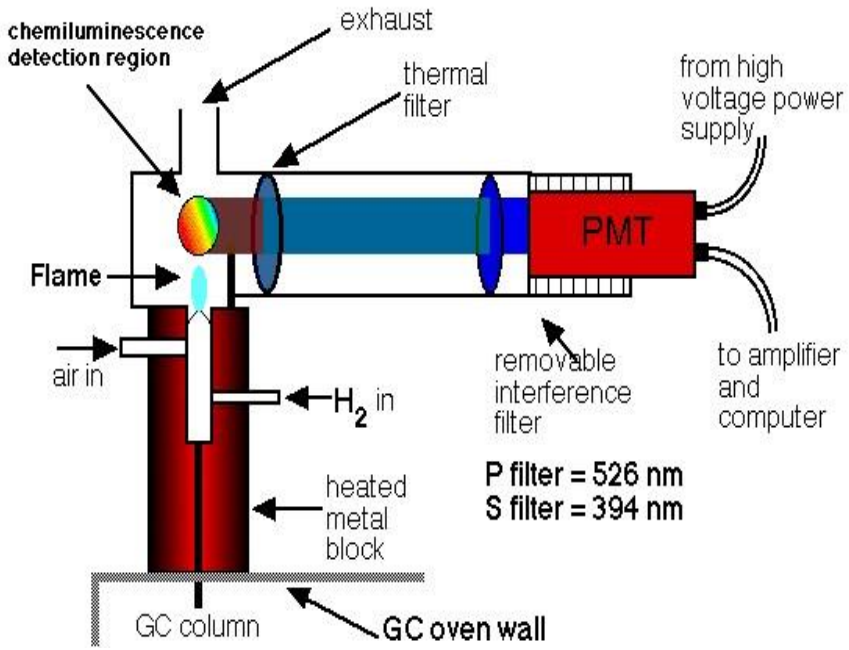
1. . The heated bead forms a plasma having temperature 600-800^{0}C.
2. This detector is remarkably insensitive to the presence of water vapour air in the carrier gas
3. The detector response to all organic compounds except formic acid

4. As the composition of gas in the flame changes the number of ions and electro ions will also change.



THERMIONIC DETECTOR:

- The hot gas flows around a electrically heated rubidium silicate bed which maintain at 180^0 voltage with an applied current.
- As carrier gas containing solutes passes through the cell, **a change in the filament current occurs**



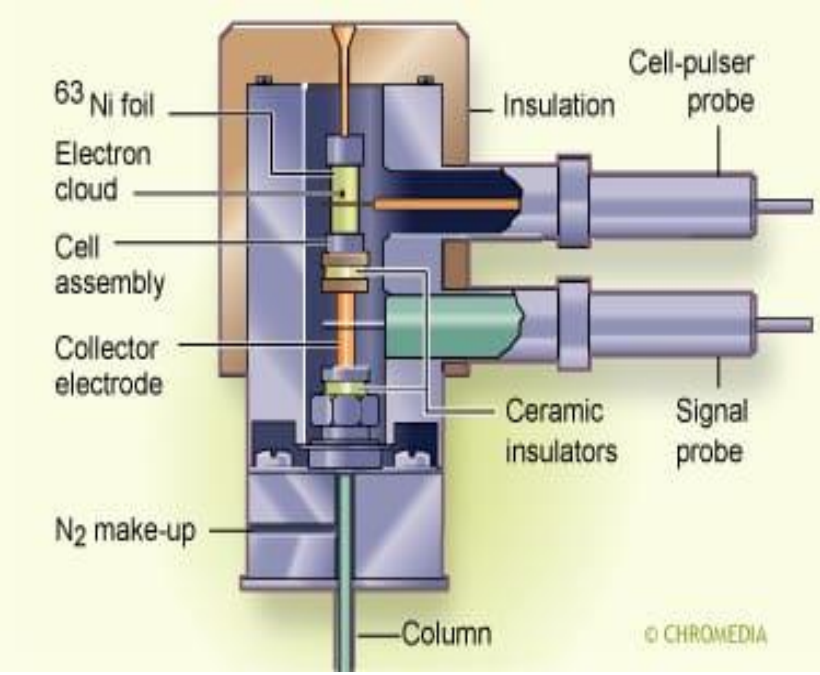
- Thermionic detector is selective towards organic compounds containing phosphorous and nitrogen.
- The column effluent is mixed with hydrogen and oxygen and pass through the flame tip and ignited.
- Then the hot gas flows around a electrically heated rubidium silicate bed which maintain at 1800 voltage.

ELECTRON CAPTURE DETECTOR:

1. Selectivity compound containing halogen, pesticides and polychlorinated diphenyl.
2. Detector is radioactive source which emits electron.
3. A cathode which repeats the electron.
4. A anode and wire screen with collect electron.

ADVANTAGES:

1. Highly sensitive
2. Good detective system
3. High resolution
4. Time fast analysis is possible
5. Destructive detector



DISADVANTAGES:

1. Destructive detector
2. Sample must be volatile
3. Lower column efficiency than capillary column
4. Carrier gas used.
5. Performance deteriorates with time.
6. Non-linear response unless potential across the detector is pulsed.

REFERENCES:

1. Gurdeep R. Chatwal page No. 2.677 to 2.691
2. Douglas A. Skoog page No.867
3. Kasture A.V. page No. 65 to 72
4. Hobart H. Willard, seventh edition, page no.552

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