School of Basic and Applied Science

Course Code : MEV303 Course Name: Techniques in Environmental Sciences

GC
Gas Chromatography

Theory and Instrumentation

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Prerequisites

- Knowledge of different types of mobile and stationary phases
- Concept of Hydrophobicity and Hydrophilicity
- Concepts of diffusion of analyte in between liquid and gas

Objectives

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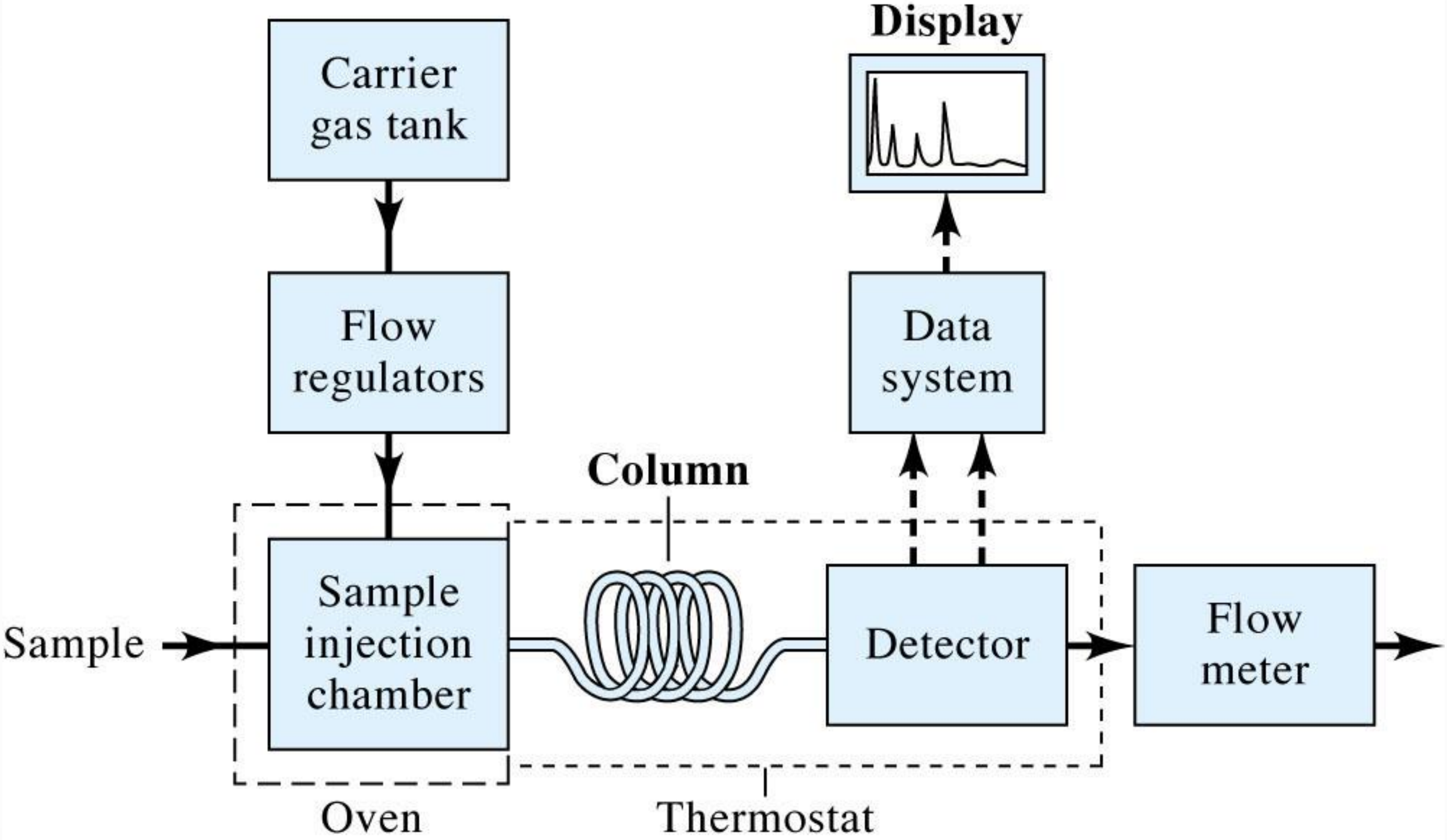
- ***<u>At the end of this Section you should be able to:</u>***
- *Understand the instrumentation and theory of Gas Chromatography*
- *Explain the types and applicability of columns used in GC*
- *Explain the types and applicability of detectors used in GC*

Gas Chromatography

- In gas chromatography (GC), the sample is vaporized and injected onto the head of a chromatographic column. Elution is brought about by the flow of an inert gaseous mobile phase.
- The mobile phase does not interact with molecule of the analyte; its only function is to transport the analyte through the column.
- Gas-liquid chromatography is based upon the partition of the analyte between a gaseous mobile phase and a liquid phase immobilized on the surface of an inert solid.


Display
Carrier gas tank
Flow regulators
Data system
Column
Sample
Sample injection chamber
Detector
Flow meter
Oven
Thermostat

INSTRUMENTS FOR GC

Carrier Gas-Supply

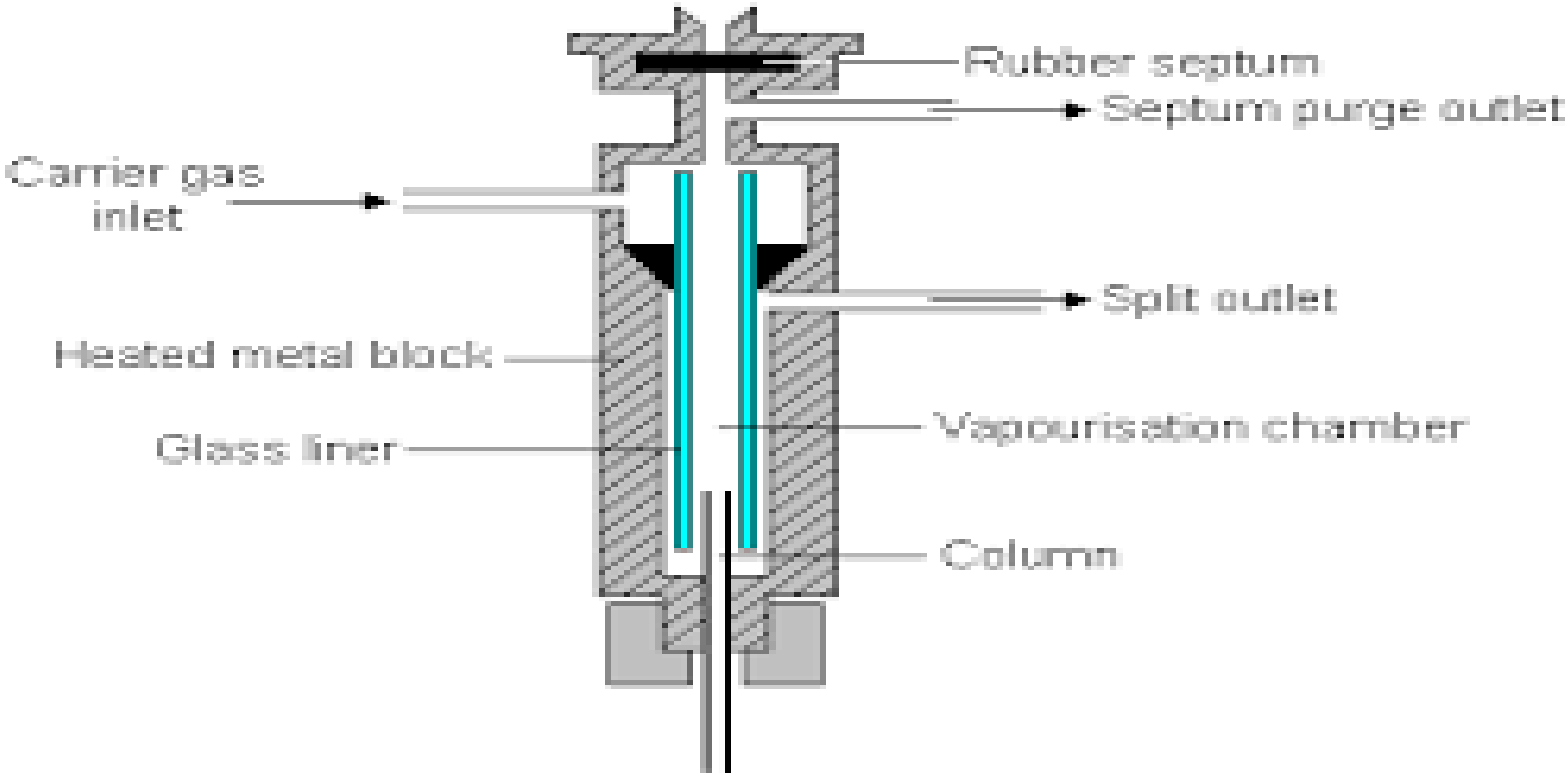
Carrier gases, which must be chemically inert, include helium, nitrogen, and hydrogen.

In addition, the carrier gas system often contains a molecular sieve to remove water or other impurities.

Sample Injection System

- Column efficiency requires that the sample be of suitable size and be introduced as a “plug” of vapor; slow injection of oversized samples causes band spreading and poor resolution.
- The most common method of sample injection involves the use of microsyringe to inject a liquid or gaseous sample through a self-sealing, silicone-rubber diaphragm or septum into a flash vaporizer port located at the head of the column (the sample port is ordinarily about 50^{o}C above the boiling point of the least volatile component of the sample).

The split / splitless injector



columns

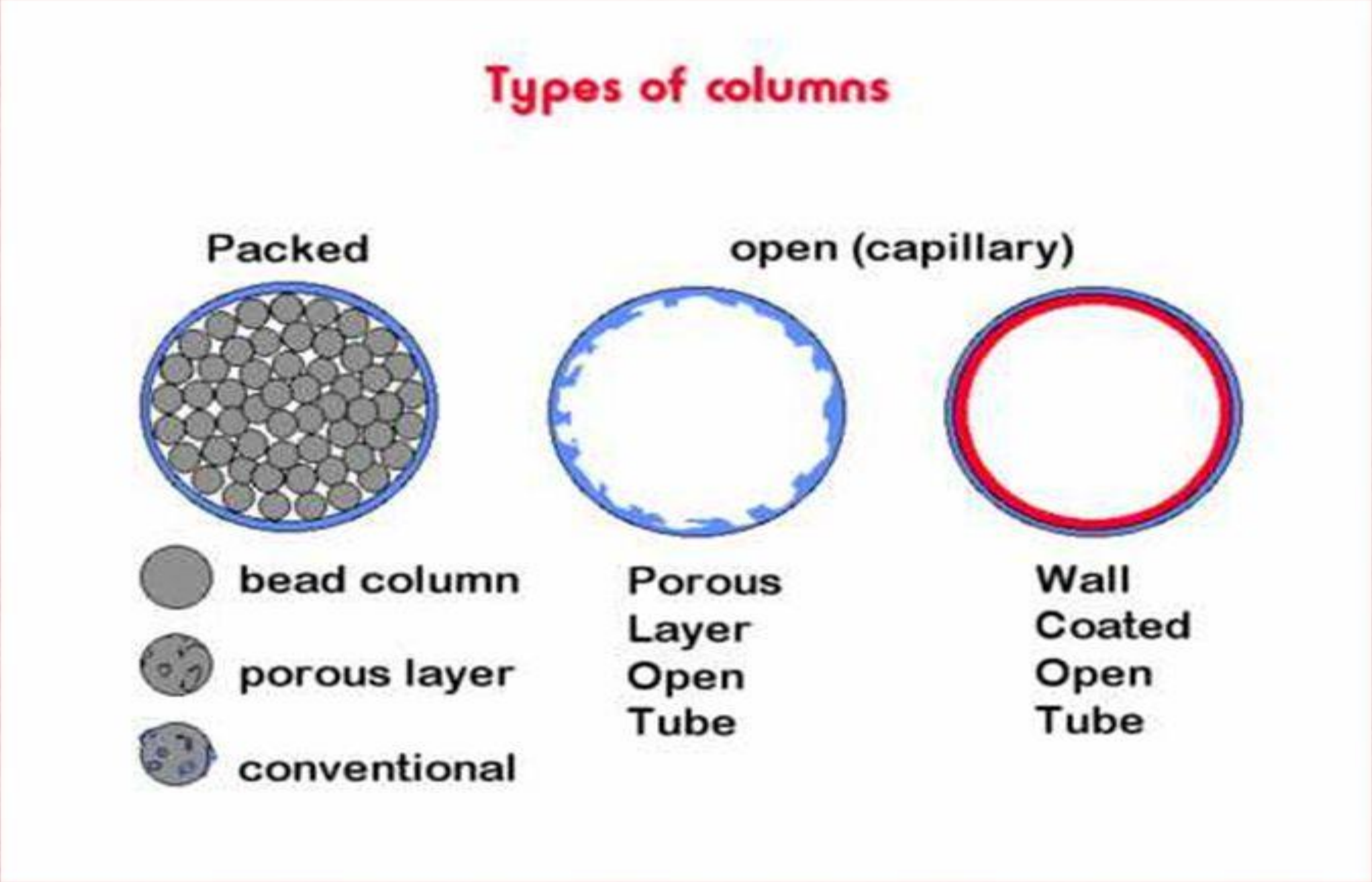

Types of columns
Packed
open (capillary)
bead column
porous layer
conventional
Porous Layer Open Tube
Wall Coated Open Tube

Table 1.1: Types of columns employed in gas chromatography

Packed	Gas Solid Chromatography (GSC)	Solid stationary phase
	Gas Liquid Chromatography (GLC)	Liquid stationary phase (Gel)
Open Tubular (OT)	Wall Coated Open Tubular (WCOT)	Stationary phase supoorted by inner wall
	Support Coated Open Tubular (SCOT)	Stationary phase supported by a layer of adsorbent material on inner wall
	Porous Layer Open Tubular (PLOT)	Contains layer of porous solid adsorbent in inner wall

Column Configurations

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- Chromatographic columns vary in length from less than 2 m to 50 m or more. They are constructed of stainless steel, glass, fused silica, or Teflon. In order to fit into an oven for thermostating, they are usually formed as coils having diameters of 10 to 30 cm.

GAS CHROMATOGRAPHIC COLUMNS

Open tubular Columns

Wall-coated columns are simply capillary tubes coated with a thin layer of the stationary phase. In support-coated open tubular columns, the inner surface of the capillary is lined with a thin film (~30 μm) of a support material, such as diatomaceous earth. This type of column holds several times as much stationary phase as does a wall-coated column and thus has a greater sample capacity.

Packed Columns

Packed columns are fabricated from glass, metal (stainless steel, copper, aluminum), or Teflon tubes that typically have lengths of 2 to 3 m and inside diameters of 2 to 4 mm. These tubes are densely packed with a uniform, finely divided packing material, or solid support, that is coated with a thin layer of the stationary liquid phase. In order to fit in a thermostating oven, the tubes are formed as coils having diameters of roughly 15 cm.

Detection Systems

Characteristics of the Ideal Detector: The ideal detector for gas chromatography has the following characteristics:

1. Adequate sensitivity

2. Good stability and reproducibility.

3. A linear response to solutes that extends over several orders of magnitude.

4. A temperature range from room temperature to at least 400^{o}C.

Characteristics of the Ideal Detector

5. A short response time that is independent of flow rate.

6. High reliability and ease of use.

7. Similarity in response toward all solutes or a highly selective response toward one or more classes of solutes.

8. Nondestructive of sample.

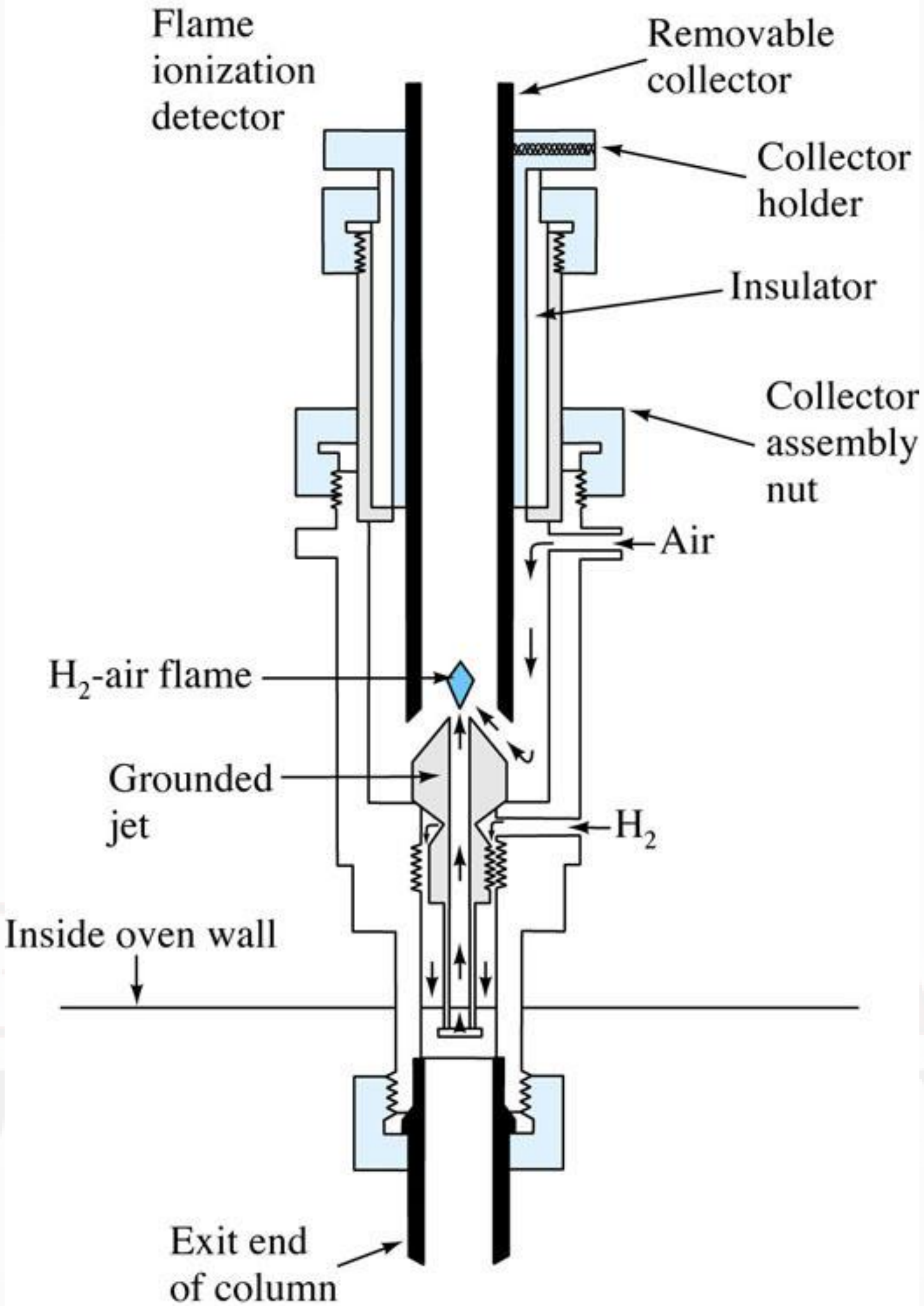
Flame Ionization Detectors (FID)

The flame ionization detector is the most widely used and generally applicable detector for gas chromatography.

- The effluent from the column is mixed with hydrogen and air and then ignited electrically.
- Most organic compounds, when pyrolyzed at the temperature of a hydrogen/air flame, produce ions and electrons that can conduct electricity through the flame.

Flame Ionization Detectors (FID)

- A potential of a few hundred volts is applied.
- The resulting current is then measured.
- The flame ionization detector exhibits a high sensitivity large linear response range, and low noise.
- A disadvantage of the flame ionization detector is that it is destructive of sample.


Flame
ionization
detector
Removable
collector
Collector
holder
Insulator
Collector
assembly
nut
Air
H_2-air flame
Grounded
jet
H_2
Inside oven wall
Exit end
of column

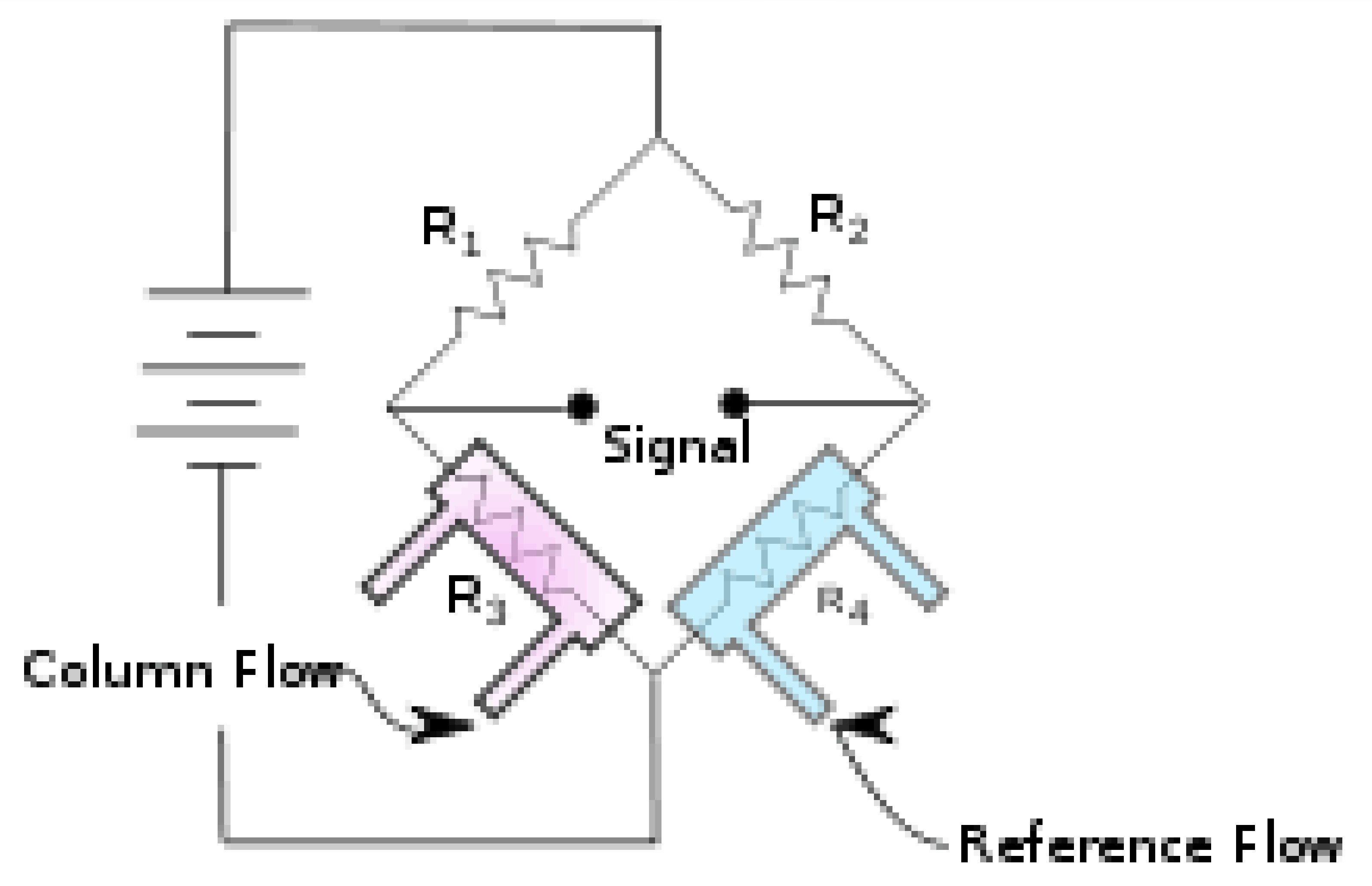
Thermal Conductivity Detectors(TCD)

A very early detector for gas chromatography, and one that still finds wide application, is based upon changes in the thermal conductivity of the gas stream brought about by the presence of analyte molecules.

- The sensing element of TCD is an electrically heated element whose temperature at constant electrical power depends upon the thermal conductivity of the surrounding gas.
- The heated element may be a fine platinum, gold, or tungsten wire or a semiconducting thermistor.

Thermal Conductivity Detectors(TCD)

- The advantage of the thermal conductivity detector is its simplicity, its large linear dynamic range(~10^5), its general response to both organic and inorganic species, and its nondestructive character, which permits collection of solutes after detection.
- A limitation of the katharometer is its relatively low sensitivity (~10^{-8} g solute/mL carrier gas).
- Other detectors exceed this sensitivity by factors as large as 10^4 to 10^7.



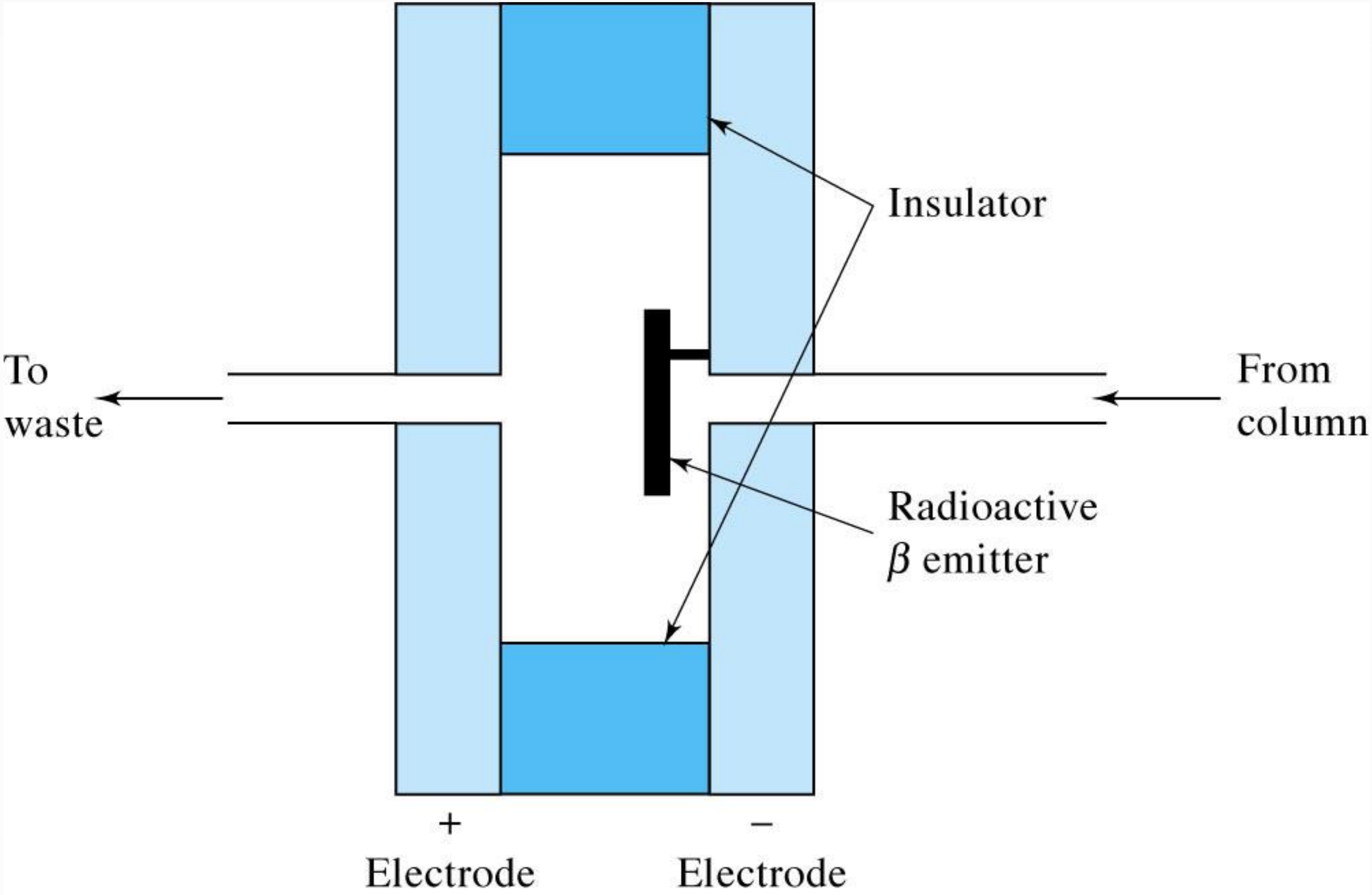
R1
R2
Signal
R3
R4
Column Flow
Reference Flow

Electron-Capture Detectors(ECD)

- The electron-capture detector has become one of the most widely used detectors for environmental samples because this detector selectivity detects halogen containing compounds, such as pesticides and polychlorinated biphenyls.
- The effluent from the column is passed over a β emitter, usually nickel-63. An electron from the emitter causes ionization of the carrier gas and the production of a burst of electrons. In the absence of organic species, a constant standing current between a pair of electrodes results from this ionization process. The current decreases markedly, however, in the presence of those organic molecules that tend to capture electrons.

Electron-Capture Detectors(ECD)

- The electron-capture detector is selective in its response being highly sensitive to molecules containing electronegative functional groups such as halogens, peroxides, quinones, and nitro groups.
- It is insensitive to functional groups such as amines, alcohols, and hydrocarbons.
- An important application of the electron-capture detector has been for the detection and determination of chlorinated insecticides.


Insulator
To
waste
From
column
Radioactive
β emitter
+
Electrode
−
Electrode

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