

## A Short Review on Gas Chromatography

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### ABSTRACT :-

Gas chromatography is the general term for a range of analytical separation techniques used to analyze volatiles in the gas phase. Gas chromatography separates the analytes by separating the sample into two phases, the stationary phase and the mobile phase, by dissolving the sample components in a solvent and evaporating them. The mobile phase is chemically inert gas, which transports the analyte molecules through the heated column. Gas Chromatography is one of the only chromatographies that interacts with analytes without the use of mobile phases. The stationary phase is either a solid adsorbent, called Gas-Solid Chromatography (GSC), or a liquid on an inert support, called Gas-Liquid Chromatography (GLC). Gas chromatography is an instrumental technique used forensically in drug analysis, arson, and toxicology analysis of other organic compounds.

**Keywords:-** Gas ,column, mobile phase, stationary Phase, pressure

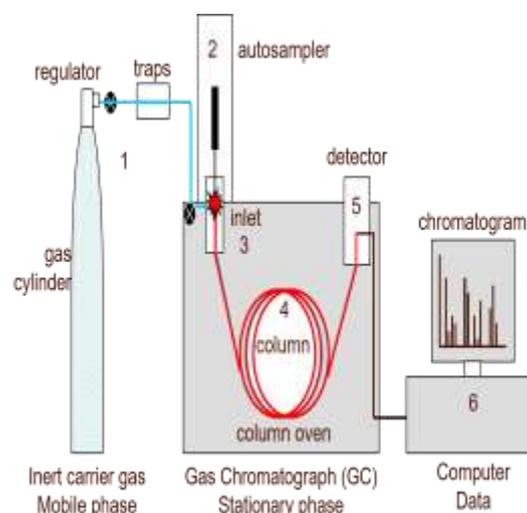
### I. INTRODUCTION:-

Gas chromatography is an analytical technique that is widely used to separate and analyze gaseous and volatile compounds. Modern gas chromatography was invented in 1952 by James and Martin. From the beginning of the 1950s, this method was first used to isolate amino acids. GC is a fast and very sensitive method. Both qualitative and quantitative analysis can be done by GC. It can also be the number of minutes analyzed by GC. In gas chromatography, the sample is dissolved in a solvent and evaporated. Split analytics. The sample is distributed between two phases: stationary phase and mobile phase. The liquid phase is helium or nitrogen etc. is a chemically inert gas such as gas chromatography is a unique type of solid phase chromatography that does not require interaction with the analyte [1]

### Principal:-

In gas-solid chromatography, a solid adsorbent is used as the stationary and separation

phase. In gas-liquid chromatography with a stationary phase adsorption process, solid consists of a thin layer of immobile liquid with support and separation. Through the process of division, gas-liquid chromatography is the most commonly used method. The separated sample is first vaporized and then mixed with the gas mobile stage. In the stationary phase, faster particles travel more slowly & in the stationary phase, the less soluble components travel faster. So are the components. The sample solution stored in the device, which is separated together for distribution, enters the gas stream that passes through the separator pipe called "column". (Helium or nitrogen is called carrier gas.) Various components are separated inside the column. The detector measures the amount of components leaving the column. To measure a sample with an unknown concentration, a standard sample with a known concentration is injected into the instrument. The peak retention time (outer form) and area of the standard sample are compared with the test sample to calculate the concentration [1,2].



## INSTRUMENTATION CHROMATOGRAPHY

A good gas chromatography machine contains the following important components,

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

### 1. Pressure Regulator :-

The cylinder regulator is directed directly to the gas cylinder valve and has the function of reducing the cylinder pressure to a value sufficient for the Gas Chromatograph. For example, there are two types of valves i.e. single stage and double stage.

- Single stage regulators



A single stage regulator is rarely used to provide constant pressure from the cylinder to the gas chromatograph. It is used in gas lines to control line pressure. However, the delivery pressure cannot be controlled as well as the dual-stage regulator. Such regulators are installed in the gas line and require constant pressure monitoring and adjustments.

- Dual stage regulators

A two-stage regulator uses two separate regulators in one housing and is attached to the gas cylinder valve. The first stage functions to reduce the cylinder pressure, while the second stage reduces the final pressure. A two-stage regulator has the advantage that while the cylinder pressure decreases due to gas consumption, the second stage provides a constant pressure regardless of the inlet pressure. Although a two-stage regulator is more

## GAS

expensive than a single-stage regulator with tolerance to drift, i.e. a gradual increase in delivery pressure when the tank is empty, a two-stage regulator is recommended when connecting to a Gas Chromatograph where the delivery pressure is constant. It is necessary.

- **CGA code**

The Compressed Gas Association (CGA) has introduced a foolproof coding system that prevents accidental mixing of cylinders and regulators, because not all regulators can be used in all cylinders. Each cylinder and regulator is identified by a CGA number code, and cylinders with matching numbers can be used together. CGA codes for some common gases:

Gas	CGA code
Nitrogen, Helium, Argon	580
Oxygen	540
Air	590
Hydrogen.	350

- **Regulators for specific gases:-**

### 1. Flammable gases :-

Combustible gases such as hydrogen have brass fittings that are reverse threaded. Tighten the nut by turning it counterclockwise. Reverse-threaded nuts can be identified by a line engraved around the circumference. If using acetylene gas, ensure that no copper tubing is used and no alloys containing copper or silver are used in the construction of the regulator.

### 2. Corrosive gases:-

Regulators must be stainless steel or monel. Install a suckback filter after the regulator to prevent corrosive gases from flowing back into the regulator and cylinder.

### 3. High purity gases :-

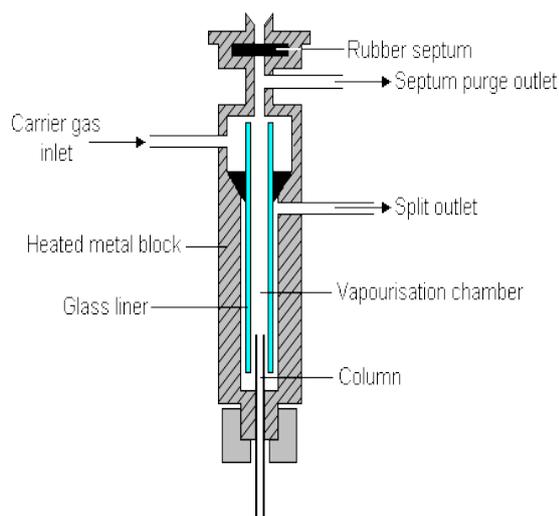
Contamination of high-purity gases can be caused by the presence of moisture, oxygen, or other gaseous vapors. Such contaminants can enter the system when the regulator is removed from the cylinder or when there is a leak or poor seal. It is recommended to use stainless steel diaphragm regulators instead of elastomeric diaphragm regulators as they do not adsorb and release contaminants. (3)

### 2. Sample injection port:-

For optimal column efficiency, the sample should not be too large and should be introduced

onto the column as a “slug”. Injecting large samples slowly results in band broadening and loss of resolution. The most common injection method uses a microsyringe to inject the sample through a rubber septum and into the flasher port at the top of the column. The sample port temperature is typically about 50 °C above the boiling point of the least volatile component of the sample. For packed columns, sample sizes range from tenths of a microliter to 20 microliters. Capillary columns, on the other hand, require much less sample, typically around 10-3 mL. Split/splitless injection is used for capillary GC. Check out this diagram for a split/splitless injector.

The split / splitless injector



Injector can be used in one of two modes. Split or splitless. The injector contains a heated chamber with a glass liner into which the sample is injected through a septum. Carrier gas enters the chamber and can be exhausted in three ways (if the injector is in split mode). The sample vaporizes to form a mixture of carrier gas, vaporized solvent, and vaporized solute. Some of this mixture enters the column, but most of it leaves the split outlet. The septum purge port prevents septum bleed components from entering the column.[4]

### 3. Columns :-

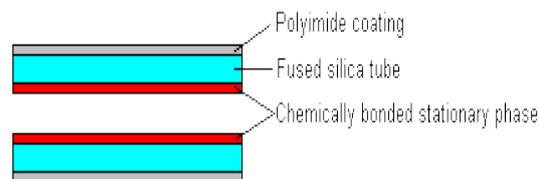
There are two common types of columns: packed columns and capillary columns (also called open tube columns). A packed column contains a finely divided inert solid support (usually based on diatomaceous earth) coated with a liquid stationary

phase. Most packed columns are 1.5 to 10 m long and 2 to 4 mm id.

Capillary columns have an internal diameter of a few tenths of a millimeter. They can be of one of two types. Wall Coated Open Tube (WCOT) or Carrier Coated Open Tube (SCOT). Wall-coated columns consist of a capillary tube whose walls are coated with a liquid stationary phase. In supported columns, the inner wall of the capillary is lined with a thin layer of support material such as diatomaceous earth onto which the stationary phase is adsorbed. In general, SCOT columns are less efficient than his WCOT columns. Both types of capillary columns are more efficient than packed columns.

In 1979 a new type of his WCOT column was developed – the fused silica open tubular (FSOT) column.

Cross section of a Fused Silica Open Tubular Column



These have much thinner walls than the glass capillary columns, and are given strength by the polyimide coating. These columns are flexible and can be wound into coils. They have the advantages of physical strength, flexibility and low reactivity.

### Classification of column:-

Columns can be classified into two types in Gas chromatography i.e.

1. Packed column
2. Open/capillary column

#### 1. Packed column

Column are made up of glass or tubes of stainless steel, copper or aluminum.

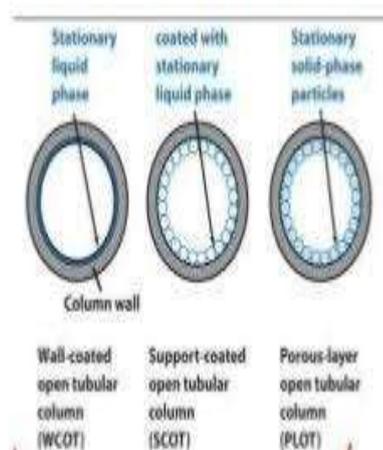
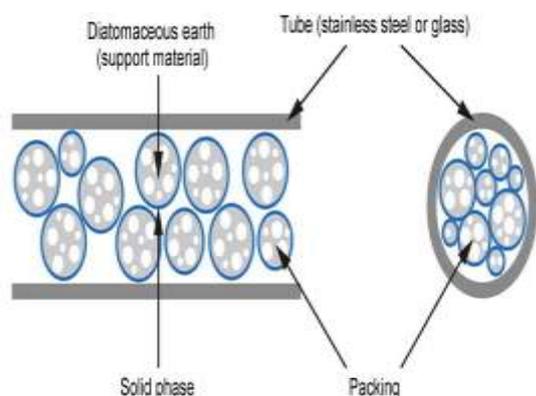
Column are available in packed manner.

Packed column contain a finely divided, inert, solid support material (commonly based on diatomaceous earth) which absorbs liquid used as stationary phase.

Most packed column are 2-20m in length and have an internal diameter of 1-8mm.

The ratio of volume of stationary phase to the mobile phase ( $V_s/V_m$ ) for the column ranges from 10-20.

The number of theoretical plates per foot length of the column is 100-1000.



#### Advantages

Larger sample capacity

#### Disadvantages

Less applicable: fixed gas analysis

Lower column efficiency than that of capillary column.

#### 2. Open/ capillary column :-

Long capillary tubes 10-100 m or more in length.

Inner diameter 0.3-0.5.

Uniform narrow D.M. from 0.025-0.075cm. The inner layer of the

Capillary is coated with a very thin film (approximately 1 nm) of liquid that acts as the stationary phase. The stationary phase to mobile phase volume ratio ( $V_s/V_m$ ) of the

Column ranges from 100 to 300, which is the main reason for its high efficiency.

Load capacity <0.01nl

Stainless steel, radiator shape (5)

#### 4. Stationary phase:-

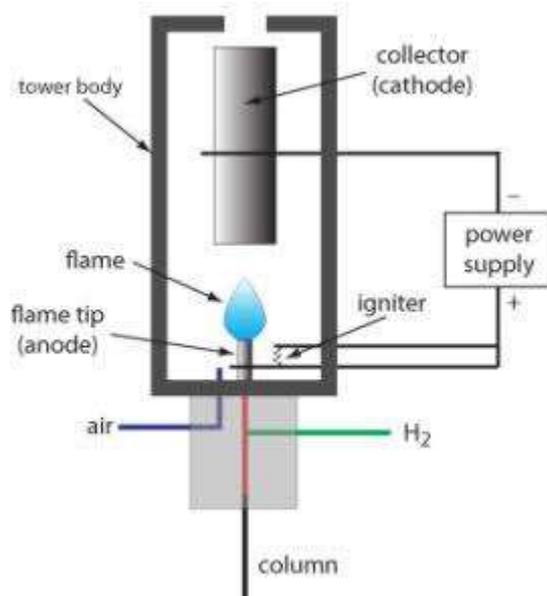
A stationary phase is a microscopic layer of viscous liquid on the surface of solid particles on an inert solid support in a piece of glass or metal tubing called a column. Some surfaces of solid particles act as stationary phases.

#### 5. Detector :-

Commonly used detectors are flame ionization detectors (FID) and thermal conductivity detectors (TCD). Although TCD has the advantage of being non-destructive, its low detection limits for most analytes preclude its widespread use. FID is primarily sensitive to hydrocarbons and is more sensitive than TCD. FID cannot detect water or carbon dioxide, making it ideal for the analysis of organic analytes in the environment. FID is two to three times more sensitive for analyte detection than TCD.

TCD relies on the thermal conductivity of a material flowing around a tungsten-rhenium wire through which an electric current flows. In this configuration, helium or nitrogen with relatively high thermal conductivity acts as a carrier gas to keep the filament cool and maintain uniform filament resistivity and electrical efficiency. As analyte molecules mix with the carrier gas and elute from the column, the temperature and resistivity of the filament increase while the thermal conductivity decreases, ultimately leading

to voltage fluctuations that trigger the detector response. Detector sensitivity is proportional to filament current, but inversely proportional to detector ambient temperature and carrier gas flow rate.

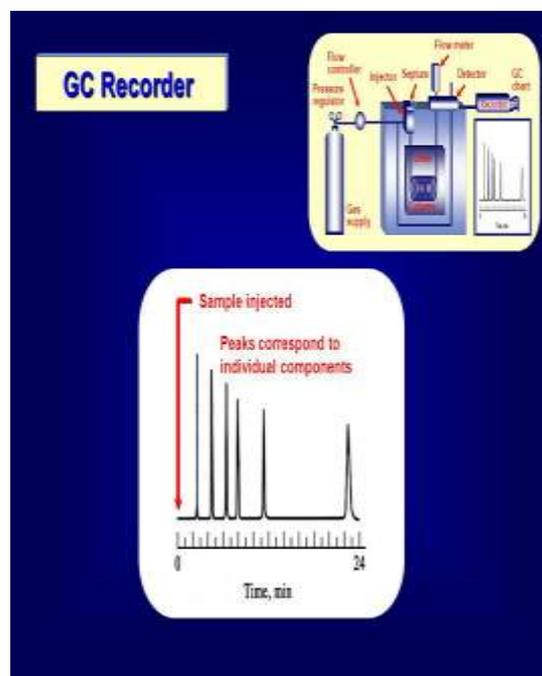


In a flame ionization detector (FID), electrodes are placed next to a hydrogen/air fuel flame near the column exit, and as carbon-containing compounds exit the column, they are pyrolyzed by the flame. This detector only works for organic/hydrocarbon compounds due to the ability of carbon to form cations and electrons during pyrolysis, creating an electric current between the electrodes. Current increases are transformed and displayed as peaks in the chromatogram. FID has a low detection limit (a few picograms per second), but cannot generate ions from carbonyl-containing carbons. FID compatible carrier gases include helium, hydrogen, nitrogen, and argon. Using a FID may change the current before it enters the detector. A methanizer converts carbon monoxide and carbon dioxide into methane for detection. Another technology is Activated Research Inc's Polyarc, which converts all compounds to methane (6).

### 6. Signal Recorder:-

A recorder is used to record the response obtained from the detector after amplification. Potentiometric detectors are commonly used in gas chromatography. In this type of recorder the input response is continuously compensated by the feedback response. Pens linked by this system

move proportionally along the width of the chart paper and record the signal. At the same time, the chart paper moves at a constant speed along its length. You must record that zero before operating the recorder. (7)



The recorder should be generally 10 mv (full scale) fitted with a fast response pen (1 sec or less). The recorder should be connected with a series of good quality resistances connected across the input to attenuate the large signals. An integrator may be a good addition.

### Application:-

- Quantification of pollutants in drinking and waste water using official U.S. Environmental Protection Agency (EPA) methods.
- Quantification of drugs and their metabolites in blood and urine for both pharmacological and forensic applications.
- Identification of unknown organic compounds in hazardous waste dumps.
- Identification of reaction products.
- Analysis of industrial products for quality control.
- ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN INDOOR AIR [8]

### Advantage:- [9]

1. Improved resolution – GC technology allows you to more easily separate closely related peaks in your data than other chromatographic methods such as thin layer chromatography (TLC).

Parameters can be adjusted in real time to better resolve peaks that occur. GC is suitable for very complex mixtures such as fumes that TLC can hardly resolve.

2. Increased Analysis Speed – Operating parameters can be easily changed (even during an experiment), allowing sample analysis to be completed in minutes. Optimum resolution can be achieved quickly with GC.

3. Wider sample choice – A wider choice of volatile samples can be analyzed with GC. The ability to control the temperature of the process allows for samples with high boiling points to be analyzed.

4. Fully Quantitative – The software used in gas chromatography provides more accurate data than other techniques, making it a fully quantitative technique. For example, TLC requires additional equipment such as densitometers and processing steps, increasing the cost of all experiments.

5. High Sensitivity – Dedicated detectors can detect target compounds at much lower limits than other technologies. In short, gas chromatography has a high level of sensitivity.

6. Non-destructive testing capabilities – detectors used in gas chromatographs such as: B. Flame photometric detectors and thermal conductivity detectors are non-destructive. This makes GC a suitable technique for non-destructive testing of samples.

7. Column Selection – Columns available in gas chromatographs come in a wide range of sizes and can be used for a variety of applications. GC experiments can also be performed on a variety of stationary and liquid supported phases.

8. Software Features – The GC has many advanced software features. Improved normalization, peak and baseline optimization improve real-time control and result reporting. This gives gas chromatography a distinct advantage over traditional techniques, as advanced data analysis capabilities enhance and improve results.

9. Column Reuse – Columns used in gas chromatography experiments can be reused, greatly reducing experimental running costs. However, it should be stored properly according to the manufacturer's instructions.

10. RESULTS AND RECORD STORAGE – With TLC, solid plates can degrade over time, so results have limited shelf life unless digitized.

## II. CONCLUSION:-

From this we can conclude that GC is currently the most widely used analytical technique

available for the separation and identification of compounds or complex mixtures. GC is the most widely used technique due to its speed, excellent resolution and sensitivity at several mg samples, and excellent accuracy and precision.

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