

A SHORT REVIEW ON GAS CHROMATOGRAPHY**Vishal Chavan^{1*}, Dr. Hemant V. Kamble² and Prof. Santosh A. Waghmare³**

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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, Gas Chromatograph autosamplers, Inlets, Detectors, Photo-ionization Detector.

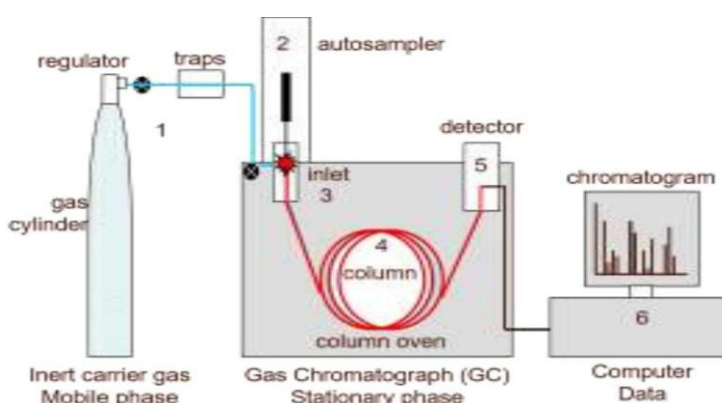
INTRODUCTION

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.

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. Gasliquid 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.



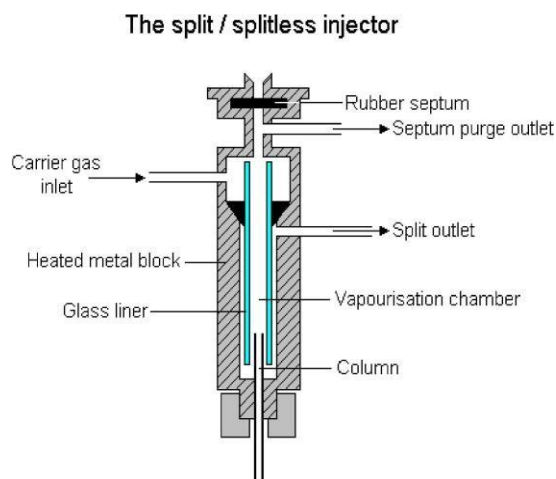
INSTRUMENTATION

Generally, all the chromatographs (GSC or GLC) consists of six basic components.

1. Sample injection system

A sample port is necessary for introducing the sample at the head of the column. A calibrated

microsyringe is used to transfer a volume of sample through a rubber septum and thus into the vaporization chamber. Most of the separations require only a small fraction of the initial sample volume and a sample splitter is used to direct excess sample to waste. Commercial gas chromatographs involves the use of both split and splitless injections when alternating between packed columns and capillary columns. The vaporization chamber is typically heated 50 °C above the lowest boiling point of the sample and subsequently mixed with the carrier gas to transport the sample into the column.



2. Carrier Gas

A carrier gas plays a vital role in GC. It should be inert, dry & free of oxygen. Helium, Nitrogen, argon & hydrogen gases are used as carrier gas depending upon the desired performance & detector being used. Carrier gas is supplied at high pressure & is passed to instrument at a rapid & reproducible rate.



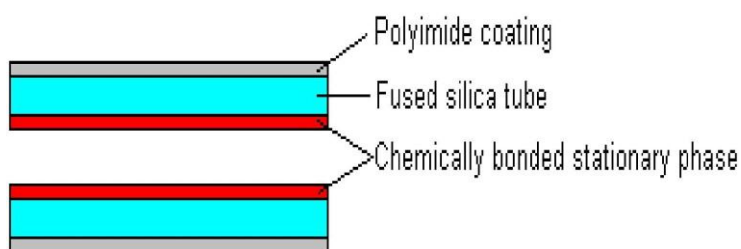
3. Separation Column

Open tubular columns or capillary columns & packed columns are used in GC. The first type

of capillary column is a wall-coated open tubular (WCOT) column and the second type is a support-coated open tubular (SCOT) column.

WCOT columns have a thin layer of the stationary phase coated along the column walls. In SCOT columns, the column walls are first coated with a thin layer of adsorbent solid, such as diatomaceous earth, a material which consists of single-celled, sea-plant skeletons. The adsorbent solid is then treated with the liquid stationary phase. While SCOT columns are capable of holding a greater volume of stationary phase than a WCOT column due to its greater sample capacity, WCOT columns still have greater column efficiencies. One of the most popular types of capillary columns is called the coated Fused Silica open tubular column.

Cross section of a Fused Silica Open Tubular Column



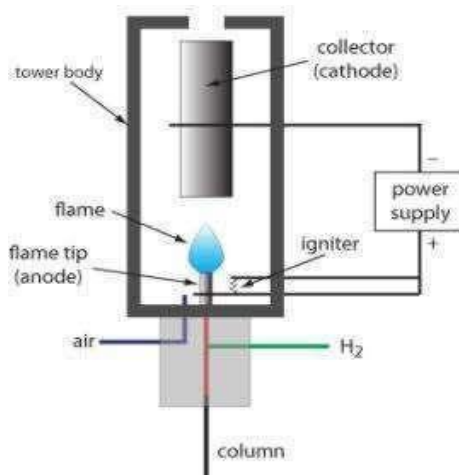
4. Column Oven or Thermostat chambers

The thermostat oven is there to control the temperature of the column to conduct precise work. The oven can be operated in two manners: isothermal programming or temperature programming. In isothermal programming, the temperature of the column is held constant throughout the whole separation. In the temperature programming method, the column temperature is either increased continuously or in steps as the separation progresses.

5. Detectors

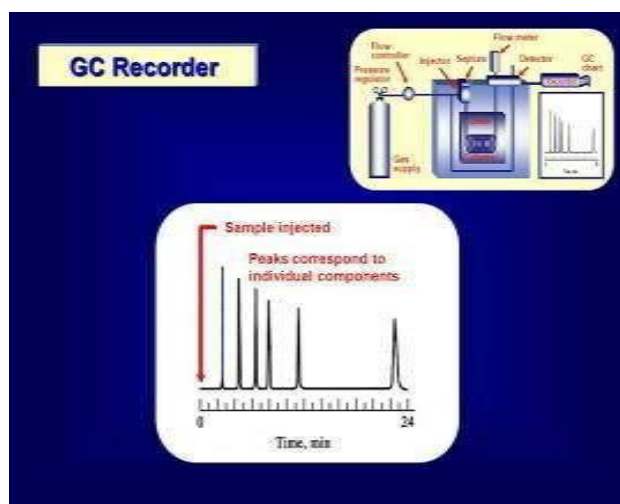
There are numerous detectors which can be utilized as a part of gas chromatography. Distinctive detectors will give different sorts of selectivity. A non-selective detector reacts to all mixtures aside from the carrier gas, a particular indicator reacts to a range of compounds with a typical physical or chemical property and a particular detector reacts to a one chemical compound. Detectors can likewise be gathered into concentration dependant detectors and mass flow dependant detectors. The signal from a concentration dependant detector is identified with the grouping of solute in the detector, and does not generally crush the sample

Dilution of with make-up gas will bring down the detectors reaction. Mass flow dependant detectors ordinarily decimate the sample, and the sign is identified with the rate at which solute particles enter the detector. The reaction of a mass flow dependant detector is unaffected by make-up gas.



6. Amplification & Recorder system

These are the last & final components of GC instrumentation. These are meant to record the signals that come from the detector. These use special electronic circuits the process & amplify the signals so as to display in an understandable graphical format that represents several peaks of the constituents of the sample under analysis.



Physical Components of Gas Chromatography

- Autosamplers
- Inlets
- Detectors

Autosamplers

The autosampler gives the way to bring a sample automatically into the channels. Manual insertion of the sample is possible but no more common. Programmed insertion gives good reproducibility and time-improvement.

Inlets

The column inlet (or injector) gives the way to bring a sample into a continuous stream of carrier gas. The inlet is a piece of equipment appended to the column head. The common inlet sorts are: split injector, on-column inlet, PTV injector, and Gas source inlet or gas switching valve, P/T (Purge-and-Trap) system.

The decision of carrier gas (portable stage) is very important. The carrier gas must be chemically inert. Generally utilized gasses include nitrogen, helium, argon, and carbon dioxide. The decision of carrier gas is regularly depend upon the sort of indicator which is utilized. The carrier gas framework likewise contains an molecular sieve to expel water and different other impurities. So, helium might be more efficient and give the best separation if flow rates are optimized. Helium is non-combustible and works with a more prominent number of detectors. Thus, helium is the most well-known carrier gas utilized. In any case, the cost of helium has gone up significantly over recent years, causing an expanding number of chromatographers to change to hydrogen gas.

Detectors

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Various types of detectors used in GC are.

- Mass Spectrometer (GC/MS)
- Flame Ionization Detector (FID)
- Thermal Conductivity Detector (TCD)
- Electron Capture Detector (ECD)
- Nitrogen-phosphorus
- Flame photometric (FPD)
- Photo-ionization (PID)

Mass spectrometer (GC/MS)

Numerous GC instruments are combined with a mass spectrometer, which is a very good blend. The GC isolates the compounds from each other, while the mass spectrometer distinguishes them in view of their fragmentation pattern.

Flame ionization detector (FID)

This detector is extremely sensitive towards organic atoms (10^{-12} g/s = 1 pg/s, linear range: 10^6 - 10^7), yet relative insensitive for a couple of small molecules i.e., N₂, NO_x, H₂S, CO, CO₂, H₂O. In the event that appropriate measures of hydrogen/air are blended, the burning does not bear the cost of any or not very many particles bringing about a low background signal. In the event that other carbon containing compounds are introduced with this stream, cations will be created in the profluent stream. The more carbon atoms are in the molecule, the more fragments are framed and the more delicate the detector is for this compound. Unfortunately, there is no relationship between the number of carbon molecules and the size of the signal.^[70] Subsequently, the individual reaction components for every compound must be experimentally decided for every instrument. Because of the fact that the sample is burnt (pyrolysis), this procedure is not appropriate for preparative GC. Furthermore, a few gasses are typically required to work a FID: hydrogen, oxygen (or compressed air), and a carrier gas.^[71-73]

Thermal conductivity detector

Thermal Conductivity Detector is less sensitive than the FID (10^{-5} - 10^{-6} g/s, straight range: 10³- 10⁴), yet is fitting for preparative applications, in light of the way that the example is not annihilated. The acknowledgment relies on upon the relationship between the two gas streams, one containing only the carrier gas, the other one containing the transporter gas and

the compound. Really, a carrier gas with a high warm conductivity i.e., helium or hydrogen is used to amplify the temperature distinction (and along these lines the distinction in resistance) between two fibers (=thin tungsten wires). The broad surface-to-mass extent permits a fast equilibration to a relentless state. The temperature distinction between the reference and the specimen cell fibers is seen by a Wheatstone bridge circuit.

Electron capture detector (ECD)

This detector comprises of a depression that contains two terminals and a radiation source that transmits - radiation (i.e., ^{63}Ni , ^3H). The impact amongst electrons and the carrier gas (methane in addition to an inert gas) creates a plasma-containing electrons and positive ions. On the off chance that a compound is available that contains electronegative molecules, those electrons will be "caught" to frame negative particles and the rate of electron accumulation will diminish. The identifier is to a great degree particular for mixes with particles of high electron liking (10-14 g/s), yet has a generally little straight range (~10²-10³). This indicator is every now and again utilized as a part of the investigation of chlorinated mixes i.e., pesticides (herbicides, insecticides), polychlorinated biphenyls, and so forth for which it shows a high sensitivity.

Nitrogen-Phosphorus

A type of thermionic detector where nitrogen and phosphorus change the work capacity on an uncommonly coated bead and a subsequent current is measured. Alkali Flame Detector, AFD or Alkali Flame Ionization Detector, AFID. AFD has high affectability to nitrogen and phosphorus, like NPD. Nonetheless, the alkaline metal particles are supplied with the hydrogen gas, instead of a bead over the fire. Consequently AFD does not endure the "fatigue" of he NPD, but rather gives a steady sensitivity over drawn out stretch of time. What's more, when alkaline ions are not added to the fire, AFD works like a standard FID.

Flame photometric (FPD)

Flame photometric (FPD) which utilizes a photomultiplier tube to identify spectral lines of the mixes as they are burned in a fire. Compounds eluting off the column are conveyed into a hydrogen energized fire which excites particular components in the molecule, and the excited components (P,S, Halogens, Some Metals) radiate light of particular characteristic wavelengths. The emitted light is separated and detected by a photomultiplier tube. Specifically, phosphorus emission is around 510-536 nm and sulfur discharge os at 39 4 nm.

Photo-ionization detector (PID)

The Polyarc reactor is an additional to new or existing GC-FID instruments that progressions over each natural compound to methane atoms going before their recognition by the FID. This framework can be used to upgrade the reaction of the FID and think about the recognition of various more carbon-containing mixes. The complete change of mixes to methane and the now indistinguishable reaction in the indicator moreover it additionally disposes of the prerequisite for alignments and gauges since response variables are all equivalent to those of methane. This checks the fast examination of complex blends that contain atoms where standards are not open. The successive reactor is sold economically as the Polyarc reactor, available online from the Activated Research Company.

How GC Works

In GC, firstly vaporized sample is injected into the chromatographic column and then sample moves through the column with the flow of inert gas & results in the separation of the components of sample which are recorded as a sequence of peaks as they leave the column. The different components of the sample separated & eluted at different & particular time which is called retention time. Retention time is determined by each component reaching the component at a characteristic time.

Applications

GC has wide range of applications in various fields .It has a medicinal & pharmaceutical applications. It is used in food, beverage, flavor & fragrance analysis. It is also helpful in environmental analysis and monitoring. It is used to detect doping of drugs In forensics, it is used in cases of arson, detection of body fluids, for the testing of fiber, blood alcohol, detection of poisons, pesticides & also to detect explosives residues. It is also useful in Security and chemical warfare agent detection.

The application of gas chromatography to environmental analysis

GC has significant role in the identification & quantification of pollutants of environment. Capillary GC is used in the analysis of various classes of persistent organic contaminants in air, water, soils, sediments and biota. The organic pollutant groups like volatile organic compounds (VOCs); polycyclic aromatic hydrocarbons (PAHs); pesticides; and halogenated compounds such as polychlorinated dibenzo-p-dioxins and dibenzofurans, polychlorinated biphenyl, terphenyls, naphthalenes and alkanes, organochlorine pesticides, and the brominated flame retardants, polybrominated biphenyls and polybrominated diphenylethers are analysed

by GC.

Application of gas chromatography in food analysis

Gas chromatography (GC) is widely used in food analysis. Quantitative and qualitative analysis of food composition, natural products, food additives, flavor and aroma components, a variety of transformation products and contaminants, such as pesticides, fumigants, environmental pollutants, natural toxins, veterinary drugs, and packaging materials are done through GC.

Application of GC in catalysis

Determination of the physicochemical properties of solid catalysts and adsorbents, catalyst evaluation and kinetics of catalytic reactions, and study of catalytic reactions are done under chromatographic conditions. GC is no longer to be regarded merely as an analytical tool for the quick (and, if necessary, continuous) determination of product composition, but as an essential part of an integrated program of kinetic analysis, including the determination of reaction parameters as well as diffusional constants. GC can be used in the study of catalysis in two ways. In the first, the catalyst under study is packed in a chromatographic column, and the properties are estimated by the chromatographic parameters such as retention time, retention volume, band width and shape, and behavior of the chromatographic peak; while in the second, a micro reactor, in which a catalytic reaction or certain measurements on the catalyst are carried out, is directly connected to the chromatographic system whose function is to provide a rapid analysis of feed and products of the catalytic process.

Application of GC to the qualitative & quantitative Copolyamide analysis

The previous techniques used for the analysis of copolyamide are time consuming & are unable to give both qualitative as well as quantitative analysis. The gas chromatographic separation of the diacids recovered from hydrolyzed copolyamides prepared from hexamethylenediamine gives both qualitative & quantitative results. The method requires only less than or 0.2 gm samples. The percent 6 nylon in copolyamide is determined, by difference & with copolyamide made from more than diamine, a calibration curve for each diamine then be prepared as well as for diacids. This method involves the gas chromatographic resolution of the polymer hydrolyzate. the liberated diacids in the hydrolylate are esterified with the boron trifluoride – methanol & the diesters are recovered in the diethyl ether, dried, gas chromatographed & the retention time is measured to identify the corresponding diacid. A second hydrolyzed used is made made caustic, extracted with-

butanol which is then removed by atmospheric distillation & thus the residue is gas chromatographed to identify diamines.

GC analysis of xylene isomer

Xylene isomers are precursors to many chemicals. o-xylene is a precursor for phthalic anhydride, m- xylene is a precursor for isophthalic acid, p- xylene is a precursor for terephthalic acid & dimethyl terephthalate. The cresol isomers are precursors to many chemicals. The chromatogram of a mixture of aromatic & methyl phenol compounds was generated using an SLB-IL60 ionic liquid column. Its interaction mechanisms allows the separation of all three xylene isomers & all three cresol isomers.

GC analysis of petroleum products

The petroleum products such as jet fuel petrol, diesel, kerosene are also analysed through GC. Test parameters involves column- supeul –Q PLOT, oven-35 degree celsius, 16 degree per min. to 250 degree Celsius, detector – TCD, carrier gas – He, sample-jet fuel. GC analysis of water in gasoline is also done.

Other common applications

- Identification of hazardous compounds in waste dumps
- Quantification of drugs & their metabolites in blood & urine for both pharmacological & forensic applications.
- Identification of reaction products.
- Quantification of pollutants in drinking & waste water.
- Analysis of industrial products for quality control.
- Skin sample analysis.
- RNA isolation.
- Astro chemistry & geochemical search.

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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