

Gas Chromatography – A Brief Review

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Abstract

Gas chromatography is a term used to describe the group of analytical separation techniques used to analyze volatile substances in the gas phase. In gas chromatography, the components of a sample are dissolved in a solvent and vaporized in order to separate the analytes by distributing the sample between two phases: a stationary phase and a mobile phase. The mobile phase is a chemically inert gas that serves to carry the molecules of the analyte through the heated column. Gas chromatography is one of the sole forms of chromatography that does not utilize the mobile phase for interacting with the analyte. The stationary phase is either a solid adsorbant, termed gas-solid chromatography (GSC), or a liquid on an inert support, termed gas-liquid chromatography (GLC). Gas chromatography is an instrumental technique used forensically in drug analysis, arson, toxicology analyses of other organic compounds.

Introduction

Gas Chromatography is a widely used analytical technique used to separate & analyze the gaseous & volatile compounds. In 1952, Modern Gas Chromatography was invented by James & Martin. Since early 1950's this technique was first used for the separation of amino acids now GC has large number of applications as this technique is rapid & has a great sensitivity. Both qualitative & quantitative analysis can be done through GC. Even minute quantity sample can be analyzed through GC. In gas chromatography, the sample is dissolved in a solvent and vaporized in order to separate the analytes. The sample is distributed between two phases: a stationary phase and a mobile phase. The mobile phase is a chemically inert gas such as helium, nitrogen etc. Gas chromatography is one of the unique forms of chromatography that does not need the mobile phase for interacting with the analyte. The stationary phase is either a solid adsorbent, termed gas-solid chromatography (GSC), or a liquid on an inert support, termed gas-liquid

chromatography (GLC).The criteria for the compounds to be analyzed in GC is volatility & thermostability.

Principle

In gas-solid chromatography, solid adsorbent is used as a stationary phase & separation takes place through adsorption process while in gas- liquid chromatography, the stationary phase consists of thin layer of non-volatile liquid bound to solid support & separation takes place through the process of partition.Gas-liquid chromatography is most commonly used technique. The sample which is to be separated is first converted into vapours & thus mixed with gaseous mobile phase. Components of a sample that are more soluble in stationary phase travels slower & the components that are less soluble in stationary phase travels faster. The components are thus separated according to their partition co-efficient.

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 are have a thin later of the stationary phase coated along the column walls. In SCOT columns, the column walls are first coated with a thin layer of adsorbant solid, such as diatomaceous earth, a material which consists of single-celled, sea-plant skeletons. The adsorbant 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.
4. Column Oven or Thermostat chambers: The thermostat oven are 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: Most common types of detectors used in GC are: Mass Spectrometer , Flame ionization detector (FID), Electron capture detector (ECD), Thermal conductivity detector (TCD), Atomic emission detector (AED), Photoionization detector (PID), Chemilumnescence detector. Detector is present at the end of the column & gives the quantitative measurement of the components of the mixture as they elute in combination with the carrier 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.

Flow regulators & flow meters are also there in GC to deliver the carrier gas with uniform pressure & flow rate.

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.

Chromatographic analysis

The number of peaks determines the number of components present in the given sample, the identity of the components are determined by their characteristic retention times & the quantity of the component in a given sample is determined by the area under the peaks.[1]

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.[2]

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.[3]

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.[4]

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 hydrolyzate 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 n-butanol which is then removed by atmospheric distillation & thus the residue is gas chromatographed to identify diamines.[5]

GC analysis of xylene isomers: Xylene isomers are precursors to many chemicals . o-xylene is a precursor for pthalic anhydride , m- xylene is a precursor for isophthalic acid , p- xylene is a precursor for tetrapthalic 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. It's interation mechanisms allows the separation of all three xylene isomers & all three cresol isomers.[6]

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 ib gasoline is also done.[6]

Other common applications

- Identification of hazardous compounds in waste damp.
- 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.[7-9]

Conclusion:

Thus , it can be concluded that at present, GC is the most widely used analytical technique available for separations & identifications of compounds or complex mixtures.The factors that

makes GC most widely used technique are its speed, good resolving power, sensitivity with few mg of sample, good precision & accuracy.

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