



INTERNATIONAL JOURNAL OF CLINICAL PHARMACOKINETICS AND MEDICAL SCIENCES

Published by Pharma Springs Publication Journal Home Page: <https://pharmasprings.com/ijcpms/>

Review on Food Analysis by Using Gas Chromatography

Neelofar H¹, Karna Haripriya^{*1}, Kishore Bandarapalle², Chekurthi Swetha Reddy¹, Kanugonda Jaswanth Reddy¹, Koka Madhavi¹

¹Department of Pharmaceutical Analysis, Sri Padmavathi School of Pharmacy, Tiruchanoor, Tirupati-517503, Andhra Pradesh, India

²Department of Pharmaceutics, Sri Padmavathi School of Pharmacy, Tiruchanoor, Tirupati-517503, Andhra Pradesh, India

Article History:

Received on: 10 Aug 2022

Revised on: 02 Sep 2022

Accepted on: 04 Sep 2022

Keywords:

Stationary Phase,
Mobile Phase,
Carbohydrates,
Fatty Acids

ABSTRACT

Explains gas chromatography (G.C.), a crucial analytical method used in the food business. It makes it possible to swiftly and affordably separate and identify complicated organic compounds. The substances that G.C. will analyse must quickly move into the gas phase and be volatile. An inert carrier gas vaporizes the substance to be examined and transports it through a substantial column. A packing material wrapped in an involatile liquid fills the column. In a mixture, the molecules of each material are divided between the gas and the liquid. A substance will move with the carrier gas longer and escape from the column more quickly the more volatile it is. G.C. must remove certain chemicals from the food analysis.



*Corresponding Author

Name: Karna Haripriya

Phone: 8185903235

Email: kharipriya146@gmail.com

eISSN: 2583-0953

DOI: <https://doi.org/10.26452/ijcpms.v2i4.344>



Production and Hosted by

Pharmasprings.com

© 2022 | All rights reserved.

INTRODUCTION

Chromatography is the more precise term for color writing. It is a physical process of separation wherein a mixture of substances can be isolated, separated, and purified into various molecules that depend on multiple distribution rates depending on solubility, Affinity, and Interaction. Chromatography is a very efficient technique that separates ingredients' constituent parts according to their properties, structure, size, and other factors. To physically separate components, chromatography uses two stages: a stable phase and a moving phase travels by way of it in a specific direction (called a mobile phase).

Stationary Phase

The stationary phase is always made up of a layer of liquid that has been adsorbed onto the outside of a sturdy endorsement [1].

e.g: Glass, Silica, Alumina

Mobile Phase

A liquid or a gaseous component is always present in this phase.

e.g., N-Hexane, Petroleum ether, Cyclo hexane

Gas Chromatography

Vapor phases Other names of gas chromatography include chromatography (VPC) and gas-liquid partition chromatography (GLPC). Gas chromatography also referred to as fluid chromatography, is a method of sequentially separating components through partitioning stationary and mobile phases within a column. It segregates, organizes, and differentiates members in organic chemical mixtures. This approach is practical in separating compounds with high volatility, Thermal stability, and low molecular weights [2].

History of Gas Chromatography

Both A.J.P. Martin and R.L.M. Synge received Nobel awards in 1952 for their contributions to liquid/soil

chromatography. Martin proposed using vapor as the mobile phase in his award speech.

James and Martins utilized ethyl acetate years ago vapor, a combination with triglycerides which have been absorbed for dissipate attached to an absorbent and placed in a tube. The vapor steam eluting from such a capillary was passed through a mechanical titration apparatus, producing a graph depicting a set of procedures reflecting its progressive insertion of bases when they were individually drained were being used to neutralize through automated titration [3].

In the late 1940s, during World War II, Austrian chemist Erika Cremer developed gas chromatography just at universities in Innsbruck, a time when women, particularly in German-speaking nations, were anticipated to limit their activities to raising children, going to church, and working within the kitchen, according to a 2008 article by Leslie Meter. According to all reports, a bright female scientist, Professor Dr. Cremer, passed away in 1996.

Principles of Gas Chromatography

There are two phases towards the mishmash: a fixed phase and a mobile phase, sometimes known as this mixture, through to the stationary phase using only a carrier gas. Molecules in the mobile phase move through to the stationary phase and interact. A magnitude but every interaction's resonance towards the stationary phase change because of each component's attributes and structural peculiarities. As a result, depending on the driving force, Various elements possess varying columns of residence time and exit it in different sequences [4].

Instrumentation of Gas Chromatography

Components of Gas Chromatography

1. Mobile phase
2. Sample injector
3. The separation column
4. Supports
5. Detectors
6. Recorder [Figure 1].

Mobile Phase

Typically, three different Gas chromatography make use of various categories of gas:

Carrier gas: Transferring the injected sample to the separation column requires carrier gas. Furthermore, these are still in charge of transporting separated components toward the detectors after that. E.g : Helium, Hydrogen, Nitrogen

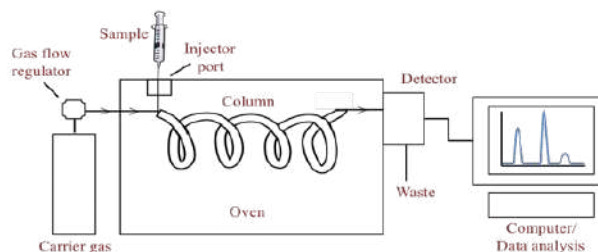


Figure 1: Schematic Diagram of Gas Chromatography

Fuel gas: They support the flame in a hydrogen-based flame ionization detector (FID).

Zero air: This is cleaned air that supports the combustion of the flame in the detector by acting as an oxidant. The abovementioned three are blended in the desired ratio before being introduced to the gas chromatographic apparatus [5].

Sample Injector

In gas chromatography, the sample that needs to be analyzed is injected using a sample injector. A representative has been infused utilizing such a calibrated syringe. A sampler and mobile phase should be in the same physical condition because they must travel together. Since the mobile phase in gas chromatography has been in a gaseous state, a sample must also be in a gaseous condition. A heater built inside the sample injector enables the vaporization of liquid samples. If the model is solid, it is crushed, ground, and transformed into a liquid state [6].

The Column of Separation

A metal column made up of bent metallic into a U shape, wound into an open spiral, or shaped like this fundamental basis in gas chromatography is just a plain pancake. Metals can be utilized up to 2500. Column insertion is made simple using Swege lock fittings, and several sizes of columns are employed based on the needs. **Liquid phase:** The only liquid-phase restrictions are their thermal stability, moisture-holding capability, and brittleness. **Fluid steps** No single phase could overcome the separating difficulties at every temperature. Silicone crude oils and apiezon L were nonpolar along with paraffin, squalane, and silicone gum rubber. Such compounds categorize all components according to the sequence in which they boil. **Transitional Polarity:** Such compounds have a polarity and polarizable component attached to such a protracted nonpolar skeleton, permitting these to absorb either nonpolar or polar solutes. **A case study.** Diethyl hexyl phthalate is applied to differentiate higher the boiling alcohols. Polarized carbowaxes were liquids phase composed primarily of polar functional groups. Molecules were divided into nonpolar and

opposing groups. Opposite liquid steps with strong hydrogen bonds, like glycol, are those having hydrogen bonds. A chemical reaction with a solute is used in specific purpose phases to achieve separations. Unsaturated hydrocarbons are separated, for instance, by AgNO₃ in glycol [7].

Supports

An efficacy established is the level of assistance and the degrees of partitioning, respectively, by the structure and surface properties like such supportive components. Support should remain inert while having the ability to create a thin film that can immobilize a significant amount of liquid phase on its surface.

When diatomaceous earth is heated to 9000 C and treated with Na₂CO₃, the particle fusion results in coarser aggregates. The support should be able to be packed into a uniform bed and should be sturdy enough to resist disintegration during handling. Up to 3% of stationary phases can be coated with glass beads with reduced permeability and a small surface area. Furthermore, the use of cross-linked polymeric pellets with varying degrees or sturdiness of styrene-alkyl-vinyl-benzene is employed. Examples: Glass, Silica, Alumina.

Detector

Detectors, which might be concentration-dependent or mass-dependent, detect the individual elements' arrival and emit a signal. The sensors should be near the column's outflow and at a suitable temperature to prevent decomposing. Crucial properties of chromatographic detectors are High reliability, Sensitivity, Good range of temperatures, Preferably Non-destructive, Small peak value to avert peak broadening, Low noise, Linear range, a short reaction period as well as a rate of flow that seem to be independent. A detector interacts well with solvents when they elute from the column. The sensor transforms this interaction into an electronic signal and transmits it to the data system. A chromatogram is produced after the signal's strength is plotted against time (starting from the injection time). While some detectors react to any solution that elutes through such a column, others exclusively react to solutes with particular structures, functional groups, or atoms. Selective sensors respond more favorably to a given class of solute. Most detectors need one or more gases to operate perfectly; ancillary, makeup, reagents, as well as products of combustion are all present. One gas may occasionally have numerous uses. The kind of detector gas depends on the particular detector but is also very standard among G.C. manufacturers. Each type of detector has a different flow rate depending on the G.C. manufacturer. It's

crucial to adhere to the advised flow rates to get a detector's optimum sensitivity, selectivity, and linear range [8].

Types of Detectors Used in Gas chromatography

1. Flame ionization detector
2. Nitrogen phosphorus detector
3. Electron capture detector
4. Thermal conductivity detector
5. Flame photometric detector
6. Photoionization detector
7. Electrolytic conductivity detector
8. Mass spectrometer

Flame Ionization Detector

The most popular detector in gas chromatography is the FID. Since practically all organic molecules contain carbon atoms (C), the FID is sensitive to and capable of identifying them. However, double-bonded carbon atoms by oxygenation, such as those in the carboxyl and carbonyl groups, are insensitive to the FID (C.O., CO₂, HCHO, HCOOH, CS₂, CCl₄, etc.)

Mechanism

Chemicals combust inside an amount of hydrogen as such process. Atoms generated via substances that contain CO₂ were taken towards the collection. A signal has been developed by measuring those electrons, which achieve collectors [9].

Selectivity: C-H bonding compounds. A subpar response for some organic materials without hydrogen.

Sensitivity: 0.1-10 ng

Linear range: 10⁵-10⁷

Gases: Hydrogen as well as oxygen as combustion; helium or nitrogen for makeup

Temperature: 250–300°C; for high thermal findings, 400–450°C.

The FID produces a hydrogen flame by burning air and hydrogen supplied from below. The hydrogen flame oxidizes the carbon in a sample that was brought into the detector on a carrier gas, which results in an ionization process.

A collector electrode draws the generated ions into an electric field, where the constituents are then measured [10].

Applications: Organic compound analysis.

Nitrogen Phosphorus Detector (NDP)**Mechanism**

Chemicals burn within a platelet that is powered via hydrocarbons and air surrounding a rubidium bead. Components possessing phosphorous but also nitrogen create ions that are drawn to the collector. A signal is produced, and the number of ions that hit the collector is counted.

Selectivity: Nitrogen as well as phosphorous consisting compounds

Sensitivity: 1-10 pg

Linear range: 10^4 - 10^{-6}

Gases: Combustion uses hydrogen and air, whereas helium is utilized for makeup.

Temperature: 250-300°C

Electron Capture Detector (ECD)

A highly sensitive, selective detector for electrophilic chemicals is the ECD. Metal-organic compounds, diketone cannabinoids, natural halogen cannabinoids, etc., can all be detected by the ECD. Education, Culture, Sports, Science, and Technological Ministries in Japan must receive notification of usage since the ECD is equipped with a radioactive isotope [11].

The ECD employs the following detecting principle. The voltage value change that keeps the collected ion current at collectors constant is what the ECD uses to detect ions.

Mechanism

Each detector's cell's ^{63}Ni alloy acts as a source of the electron. Electricity gets generated within the cells. Electronegative compounds' capability to capture electrons leads to a decrease in electricity. During indirect indicators of electrical losses, a signal is generated.

Selectivity: Nitrates, conjugated carbonyls, as well as halogens

Sensitivity: 1-100 pg (nitrogen), 0.1-10 pg (halogenated substances), but also 0.1-1 ng (carbonyls)

Linear range: 10^3 - 10^4

Gases: Nitrogen or argon/methane

Temperature: 300-400°C.

Applications

1. Environment analysis
2. Environmental organic mercury
3. Chlorinated VOCs in discharge water

4. Residual PCBs and chlorinated insecticides

Thermal Conductivity Detectors (TCD)

Except for the carrier gas, all chemicals are detectable by the TCD. Inorganic gases and other components that the FID is not sensitive to are mainly detected using the TCD [12].

The gas helium is frequently employed as a carrier. (He and H₂ are examined using N₂ and Ar.)

Mechanism

An applied current heats a filament inside a detector cell. The filament current alters as carrier gas-carrying solutes move through the cell. A reference cell's current and the current change are compared. A signal is produced once the difference is measured.

Selectivity: every combination, excluding the carrier gas

Sensitivity: 5-20 ng

Linear range: 10^5 - 10^6

Gases: Makeup - same as the carrier gas

Temperature: 150-250°C

Applications: Water, formaldehyde, formic acid, etc.

Flame Photometric Detectors (FPD)

The FPD is a sensitive, selective detector for organic tin (Sn), sulfur, and phosphorus (P) molecules. The FPD is exceptionally demanding as it only picks up element-specific light generated within a hydrogen flame [13].

Mechanism

Compounds burn in a hydrogen-air flame as the mechanism. Light-emitting species are produced by chemicals that contain sulfur and phosphorus (sulfur at 394 nm and phosphorous at 526 nm). Only one of the wavelengths can pass through a monochromatic filter. A signal is produced after measuring the amount of light with a photomultiplier tube. Each detection mode requires a distinct filter.

Selectivity: Compounds containing phosphorus or sulfur. One at a time only.

Sensitivity: 10-100 pg (sulfur); 1-10 pg (phosphorous)

Linear range: Non-linear (sulfur); 10^3 - 10^5 (phosphorous)

Gases: Combustion - hydrogen and air; Makeup - nitrogen

Temperature: 250-300°C [14].

Food

Definition

An essential element that the body must have and also be healthy is food. Proper nutrition is crucial for overall well-being all through lifetime and good development throughout infancy, childhood, adolescence, and adulthood [15]. The human diet is not limited to any particular type of food. Man consumes a range of plant and animal-based foods because no single item can satisfy all of our nutritional needs. The adage "We are what we eat" is accurate. Of course, most of us do not turn into bananas if we eat a banana, but whether for good or bad, our bodies must assimilate, modify, and excrete the chemicals we ingest. Food is a necessary component of life, and access to it frequently determines the size of a population. There is no doubting the significance of food, even while there is some debate among friends over whether we "eat to live" or "live to eat" (and some individuals "are dying to eat" or "eat themselves to death"). Chemical analysis is the only way to determine which chemicals are present in food and how many there are. The nutritional requirements for the various substances or their impacts on health can then be determined. Chromatography, in particular, has been crucial for separating numerous organic compounds in food. Analytical chemistry, in general, has played a significant role in human development through its ability to identify and quantify components in food [Table 1] [Figure 2] [16].

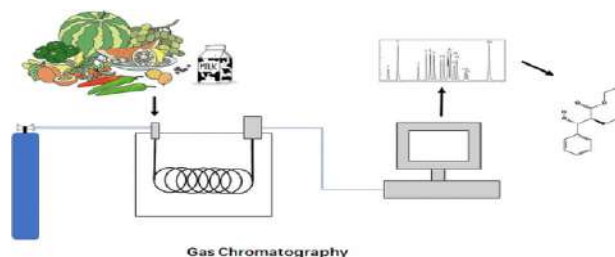


Figure 2: Food Analysis by Using Gas Chromatography

Need for Food Analysis

Food analysis is done to verify the nutritional value and safety of food. Foods and raw materials shouldn't include any toxic ingredients or microbe concentrations that are unsafe. Eating out and purchasing food from the grocery store should be safe. An approved laboratory must analyze the food producers under official food control. Accredited laboratories also carry out numerous additional kinds of food analysis.

Meeting Government and Industry Regulations

Government and business organizations impose strict regulations on the food and beverage industry.

For instance, peanut butter in the USA must include at least 90% peanuts, a total fat level of no more than 55%, and a maximum of 10% other components like salt and sugar [17]. Everyone can meet and maintain these requirements with the help of food analysis, from product makers to ingredient suppliers.

Protecting Consumers

Consumer safety is a top priority in the food and beverage industry. Before they are placed on store shelves for customer purchase, analytical procedures are employed to screen materials and completed goods. For instance, foodborne bacteria and viruses can be found in items like lettuce using polymerase chain reaction (PCR) assays. Toxins left behind by pesticides and herbicides, as well as traces of metal, wood, glass, and other pollutants, are also found using laboratory tests.

Preventing Food Fraud

Food fraud is a global problem that impacts customers, product makers, and ingredient suppliers. One of the most well-known instances of food fraud occurred in 2013 when it was discovered that frozen ready meals and burgers purportedly made with beef included horse DNA.

Nutritional Labeling

Consumers can get detailed information about the nature and composition of food through nutritional labeling.

Most nations mandate that food producers display uniform nutritional labels that include information on fat, cholesterol, carbs, salt, sugars, and protein. Consumers can make informed decisions using this information.

Consistency and Quality Control

While consistency can differ for small-batch items, large-scale producers place a high premium on keeping the same general features.

Regular testing enables manufacturers to match consumer expectations, eventually boosting revenues and expanding their market share.

Testing is done to keep track of everything from starch and sugar content to flavor and texture, which might vary in products like potatoes.

Research and Development

Food analysis is essential to corporate research and development. For instance, the American business Beyond Meat uses food analysis methods to create novel plant-based beef products.

The company has become the market leader and formed alliances with well-known companies like KFC, Pizza Hut, and Panda Express [18].

Table 1: Chromatographic Techniques Used for Food Analysis

Components of Food	The Chromatographic Method Used for the Analysis
Carbohydrates	High-performance liquid chromatography (HPLC) Gas chromatography (G.C.) Thin layer chromatography (TLC)
Proteins	Column chromatography High-performance liquid chromatography (HPLC)
Fats	High-performance liquid chromatography (HPLC) Gas chromatography (G.C.) Thin layer chromatography (TLC)
Vitamins & Minerals	High-performance liquid chromatography (HPLC) Gas chromatography (G.C.)
Dietary fibers	High-performance liquid chromatography (HPLC)

Sample Preparation for Gas Chromatography

Generally, a food product cannot be injected straight into a G.C. without undergoing sample preparation. Degradation of non-volatile contents will occur due to the injection port's high temperatures, and several erroneous G.C. peaks will be produced corresponding to the resulting volatile degradation products.

Additionally, it is frequently necessary to isolate the target ingredient from the food matrix to allow concentration to G.C. detectable limits or to segregate it from most food.

As a result, sample pretreatment, component isolation, and concentration are usually required before G.C. analysis. Grinding, homogenization, or other methods of reducing particle size are frequently used in sample preparation.

Many foods have active enzyme systems that change the food product's composition. This is particularly clear in the field of flavor work.

It may be necessary to inactivate enzyme systems using high temperatures, fast thermal processing, sample storage under freezing conditions, sample drying, or homogenization with alcohol.

During the production of the sample, the food may experience microbial growth or chemical reactions. Certain chemicals (such as sodium fluoride), thermal processing, drying, or freezing storage frequently suppress microorganisms [19].

False peaks on the G.C. often occur from chemical interactions. As a result, the model needs to be kept in settings that prevent degradation.

Analysis of Carbohydrates

1. Carbohydrates are a crucial source of energy metabolism for animals and plants that depend upon plants for food.

2. In addition to serving as a crucial component of nourishment, sugars and starches also act as a structural substance (cellulose), one of the three essential elements in RNA and DNA, a constituent of such power component ATP, recognition sites onto cell surface membrane. Saccharides are another name for carbohydrates, or sugars, if they are tiny.
3. Like HPLC, G.C. (gas-liquid chromatography, or GLC) offers a qualitative and quantitative examination of carbohydrates.
4. Sugars must be transformed into volatile derivatives for G.C. The alditol peracetates are the derivatives that are utilized the most frequently. These derivatives are made in the manner described in the D galactose illustration.
5. The most popular type of detector for hyperacetylated carbohydrate derivatives is a flame ionization detector (FID), but mass spectrometers are increasingly being employed.
6. The detection limits are lowered using a mass spectrometric (M.S.) detector and further reduced by an MS/MS detector.
7. The origins and adulterations of food and additives have been identified using gas chromatography combustion-isotope ratio mass spectrometry.
8. The two preparation steps required for G.C. for carbohydrate analysis—reducing aldehydic groups to primary hydroxyl groups and converting reduced sugars (alditol) into volatile peracetate esters—pose the biggest challenge. Of course, each of these steps must be 100% finished for the analysis to be successful.

Neutral Sugars

A surplus of sodium or potassium borohydride dissolved in Balanced diluted glucose from 80% ethanolic extracts or hydrolysis of such polysaccharides over 40 °C was decreased by utilizing ammonia hypochlorite. Inclusion of glacial acetic acid after the process to remove any extra borohydride. The acidified solution is dried by evaporation. There may be an issue: Fructose will be converted to a mixture of d-glucitol (sorbitol) and d-mannitol if it is available, whether it is a naturally occurring sugar, the result of the hydrolysis Reduction to Alditolsof inulin, or an additive [from a high-fructose syrup (HFS), invert sugar, or honey].

Acetylation of Alditols

A dry mixture of alditols is mixed with acetic anhydride and a catalyst. About 10 min at room temperature, add water, then dichloromethane. The alditolperacetate residue is dissolved in a polar organic solvent (often acetone) for chromatography. Following mixing, the dichloromethane layer is cleaned with water before being dried by evaporation.

GC of Alditol Peracetates

If inositol is added as an internal standard before acetylation, alditol peracetates can be chromatographed isothermally and recognized by their retention durations compared to inositol hexaacetate. To assess elution durations and relative responses, it is crucial to run standards of the alditol peracetates of the sugars being measured with inositol hexaacetate as an internal standard [20].

Fatty Acid Composition and Fatty Acid Methyl Esters (FAMES)

By measuring the types and amounts of fatty acids present, typically, The rich acid concentration, or fatty acid profile, of such packaged foods could be determined by separating those triglycerides as well as evaluating those using capillary G.C. can be established.

Principle

Triacylglycerols are often transesterified to create fatty acid methyl esters to boost volatility before G.C. analysis. Acyl lipids are easily transesterified using a base like sodium hydroxide or methanol. This mixture's sodium methoxide will quickly convert acyl lipids into FAMES but won't interact with free fatty acids. Acidic substances like methanolic HCL or boron trifluoride react with FFAs quickly but with acyl lipids more slowly. Two-step methylation is used in procedures like the AOCS Method Ce 1b-89, which involves treating the lipid with 0.5 N NaOH

and too much BF₃/methanol. FFAs, acyl lipids, and phospholipids can all be quickly methylated because of this. The stage using sodium hydroxide is not a saponification process. Direct transmethylation is what it is [21].

Procedure

By homogenizing the meal with a suitable solvent, such as hexane-isopropanol (3:2, vol/vol), and then letting the solvent evaporate, the lipid can be extracted from the food, for instance. The extracted lipid is mixed with sodium hydroxide methanol, an internal standard in isooctane, and then heated at 100 °C for five minutes to create the FAMES. After the sample has cooled, further BF₃-methanol is added, and the mixture is heated at 100 °C for 30 minutes. The upper isooctane solution containing the FAMES is withdrawn, dried with anhydrous Na₂SO₄, and then diluted to a concentration of 5–10% for injection onto the G.C. This is done after adding saturated aqueous sodium chloride, more isooctane, and mixing. Several techniques outline steps and circumstances for using G.C. to determine the composition of fatty acids. AOCS Method Ce 1f-96 is most suited for determining trans-isomer fatty acids, while AOCS Method Ce 1b-89 specializes in marine oils.

Cholesterol and Phytosterols

There are numerous techniques for measuring cholesterol and phytosterols in diverse matrices. A review of the scientific literature will provide information on existing procedures and methods that may be simplified or tailored for usage with certain foods.

Principle

It is saponified to remove the fat from the food. Acyl lipids are hydrolyzed during the saponification process to produce water-soluble FFA salts. Following hydrolysis, specific components (unsaponifiable or nonsaponifiable matter) remain soluble in organic solvents because their solubility does not change. Trimethylsilyl (TMS) ethers or acetate esters are created by extracting and derivatizing cholesterol (from the unsaponifiable fraction). This raises their volatility and lessens chromatographic peak tailing issues. Capillary G.C. is used to accomplish quantification.

Procedure

The food's lipids are taken out, saponified, and the extractable portion is taken out. To do this, a part of the chloroform layer is filtered through anhydrous sodium sulfate and dried in a water bath with a stream of nitrogen gas. The mixture is then refluxed while concentrated potassium hydroxide

and ethanol are added. Shaking is done after adding aliquots of benzene and 1 N potassium hydroxide. After removing the aqueous layer, the procedure is repeated using 0.5 N potassium hydroxide. The benzene layer is dried with anhydrous sodium sulfate, and an aliquot is evaporated to dryness on a rotary evaporator after being repeatedly washed with water. The leftovers are absorbed by dimethylformamide. Trimethylchlorosilane and hexamethyldisilazane (HMDS) are added to an aliquot of this sample to be derivatized. After including water (to react with and deactivate excess reagent) and an internal standard in heptane, the mixture is centrifuged. A GC with a nonpolar column receives a partial injection of the heptane layer. The HMDS and TMCS reagents are quickly inactivated by water. Hence the reaction environment must remain anhydrous [22].

Measurement of Vitamin D

Like steroids, vitamin D and its metabolites are relatively non-volatile substances that require G.C. oven temperatures of 200–3500 to separate. When one considers their shared chemical basis, it may not be a surprise that there are numerous similarities between the issues related to the G.C. analysis of steroid hormones and vitamin D. Both categories of steroids can be analyzed by G.C. without the creation of derivatives, even though there is significant peak broadening, which is likely due to adsorption into the column's "inert" support. The selection of suitable products can improve peak shape and separation. Because C21-corticosteroids with an 17-hydroxyl group are thermally unstable, high oven temperatures can cause side chain cleavage, which results in the formation of C19 steroids unless appropriate derivatives are generated before G.C. Vitamin D and its metabolites also experience temperature changes G.C. The B ring is closed when vitamin D is injected into a G.C. column, resulting in the formation of the isomers pyro- (9 α H3' loan) and pyro- (9SCH3, 10SH) calciferous.

Two peaks are produced when a single vitamin D metabolite is added to a G.C. column; these peaks are typically created in a constant ratio. At all temperatures over 25 °C, similar cyclization occurs in the test tube, and the balance of the isomers to one another remains constant. When a "profile" is required, those interested in G.C. prefer to use methods in which a single compound results in a single peak. A single molecule can produce many mountains, leading to severe interpretational issues. Since one metabolite can only make one peak, much work has been done to discover vitamin D derivatives and its metabolites that would withstand ther-

mal cyclization during G.C. analysis. Isotachysterols are the only derivatives successfully applied in this situation thus far [23].

Monitoring Pesticide Residues in Greenhouse Tomato

One of the most popularly cultivated vegetables in the world is the tomato (*Lycopersicon esculentum*, also known as *Solanum lycopersicum* or *Lycopersicon lycopersicum*), which is a member of the Solanaceae family [Figure 3].

Materials and Reagents

A mixture of pesticides (600 mg/ml of azoxystrobin, difenoconazole, fludioxonil, pirimicarb, as well as tebuconazole; 300 lg/ml of captan, chlorpyrifos, cyprodinil, but rather 100 lg/ml of Sigma-Aldrich provided the internal standard (I.S.) triphenylphosphate (TPP), which was 99.0% pure. TPP individual stock I.S. solution was created in Mecn at a concentration of 20 g/ml. The relevant concentrations of working mixes were made, stored in the dark, and kept chilled at 4⁰c [24].



Figure 3: Image of Tomato

Sampling and Sample Preparation

A DLLME approach and a modified Quenchers method were used to remove the pesticides from the tomato. Fresh tomato samples that had not been washed were minced using a Silvercrest SSMS 600 B2 Kompennass hand blender. The following steps make up the extraction process:

Weigh and transfer 10 0.1 g of the sample to a 50 ml centrifuge tube. Add the solution and vortex for one minute to thoroughly mix it with the selection. Add 10 ml of mean to the models, close the lines, and violently shake them by hand for 30 seconds. Add 4 g anhydrous mgso4 and 1 g NaCl to the sample tubes.

Close the tubes, then violently shake them by hand for one minute. Centrifuge the tubes for five minutes at 5000 rpm. Transfer 8 to 15 ml of the upper layer of the men extract into an amber container. Pour 1 ml of the finished extract into the 15 ml conical SarstedttubesAdd 100 ll of carbon tetrachloride and 4 ml of ultrapure water to this extract. Shaking the test tube forcefully by hand for one minute causes tiny droplets of carbon tetrachloride in an aqueous

solution to disperse, forming a hazy solution. Use a pipette to remove the upper aqueous phase from conical tubes centrifuged at 5000 rpm for 4 min (dispersive particles of silt are at the bottom of the centrifuge tube) (A vial containing 100 microlites of the settled sediment is filled with one ll, which is then injected into the GC-ms system [25].

Apparatus and G.C.-M.S. Conditions

A DB-5MS column underwent G.C. separation. The carrier gas was helium, and the pressure was scaled from 150 kpa to 367.1 kpa while increasing at a rate of 2.2 kpa/min. The injector temperature was 280 C, and the injection was performed in the splitless mode for 0.5 minutes. The glass liner was equipped with a Restekcarbofrit plug. The following was the oven temperature program: After holding at 80 C for 2.0 min, the temperature ramped up to 180 C at 20 C/min, then to 230 C at 5 C/min, to 280 C at 20 C/min, and ultimately to 300 C at 40 C/min, where it remained for 3.0 min. The MS transfer line was maintained at a temperature of 280 C. A 23-minute runtime was used. The following mass spectrometric settings were set: electron impact ionization with a 70eV energy, 230 C for the ion source, and 150 C for the M.S. quadrupole. Each molecule was measured based on peak area utilizing one target and one or two qualifier ions, with the M.S. instrument regularly set in selective ion monitoring (SIM) mode [26].

Fatty Acids in Walnuts and Peanuts

Nuts are considered nutrient-rich foods, and eating them has been linked to a lower risk of coronary heart disease. Due in part to their high quantity of unsaturated fatty acids, nuts provide many health benefits. The fatty acid composition of nuts can be ascertained using validated techniques from AOCS® and AOAC®. This work illustrates the application of some of these approaches to determine the fatty acid composition of walnuts and peanuts. We will present data demonstrating analysis on three distinct selectivity G.C. capillary columns: Omegawax®, SPTM-2560, and SLB®-IL111 [Figure 4] [27].

Column Selectivity for Fatty Acid Methyl Esters

The polyethylene glycol (PEG)-based phase creates the moderately polar Omegawax. Fatty acid methyl esters (FAMES) are eluted according to the degree of unsaturation with little overlap across various carbon chain lengths. Applications requiring the examination of saturated, monounsaturated, and polyunsaturated fatty acids employ it. It cannot, however, offer the best resolution of the cis and trans isomer groups. It is necessary to use a more polar col-

umn to study cis/trans-FAMES. An extremely opposite cyano silicone column is the SP-2560. This phase's selectivity allows for the resolution of cis and trans isomers and positional geometric isomer separations [28]. The polarity of the SLB-IL111, an ionic liquid column, is greater than that of the SP-2560. The SLB-IL111 has shown elution patterns that work well with the SP-2560 for analyzing cis/trans-FAMES. To profile the fatty acids (including PUFAs) found in peanuts and walnuts, we employed Omegawax. The C18:1 cis/trans isomers were then determined using the SP-2560 and SLB-IL111 columns [29].



Figure 4: Image of Walnuts and Peanuts

Experimental

According to AOCS® Official Method Ce 1k-093, 1 g samples of dry-roasted peanuts and shelled, chopped walnuts were prepared using acid digestion/alkali hydrolysis, followed by methylation. Antioxidant BHT was added before extraction. Before G.C. analysis, all samples were concentrated to 1 mL. The following G.C. columns were used in the analysis:

1. Omegawax®, 30 m x 0.25 mm I.D., 0.25 μ M
2. SP™-2560, 100 m x 0.25 mm I.D., 0.20 μ M
3. SLB®-IL111, 100 m x 0.25 mm I.D., 0.20 μ M [30]

CONCLUSION

Gas chromatography is one of the oldest chromatographic techniques with numerous applications. Gas chromatography provides plenty of contribution to food analysis, gives meaningful results, and may Analysis many other food products in the future. Gas chromatography in food analysis is effectively implemented in the processes of authenticating as well as preventing crime involving a wide range of beverages and food items, including olives as well as other edible veggies, oils, honey as well as other bee products, etc. primarily using gas chromatography through connection with such a mass spectrometer as well as a flame ionization detection was applied.

ACKNOWLEDGEMENT

The corresponding author desires to explicit utmost gratitude to the Management and Prof. Dr. D. Ranganayakulu, M. Pharm., PhD, Principal, Sri Padmavati School of Pharmacy, Tiruchanoor, Andhra Pradesh, India, for presenting all the necessary laboratory demands of the research and constant support.

Conflict of Interest

The authors declare no conflict of interest, financial or otherwise.

Funding Support

The authors declare that they have no funding for this study.

REFERENCES

- [1] P Oliveri and G Downey. Multivariate class modelling for the verification of food-authenticity claims. *TrAC Trends in Analytical Chemistry*, 35:74–86, 2012.
- [2] G P Danezis, A S Tsagkaris, F Camin, V Brusic, and C A Georgiou. Food authentication: Techniques, trends & emerging approaches. *TrAC Trends in Analytical Chemistry*, 85:123–132, 2016.
- [3] L Manning and J M Soon. Developing systems to control food adulteration. *Food Policy*, 49:23–32, 2014.
- [4] J Spink and D C Moyer. Defining the public health threat of food fraud. *Journal of food science*, 76(9):157–163, 2011.
- [5] V M Wheatley and J Spink. Defining the public health threat of dietary supplement fraud. *Comprehensive Reviews in Food Science and Food Safety*, 12(6):599–613, 2013.
- [6] E Borràs, J Ferré, R Boqué, M Mestres, L Aceña, and O Busto. Data fusion methodologies for food and beverage authentication and quality assessment-A review. *Analytica Chimica Acta*, 891:1–14, 2015.
- [7] L Cuadros-Rodríguez, C Ruiz-Samblás, L Valverde-Som, E Pérez-Castaño, and A González-Casado. Chromatographic fingerprinting: An innovative approach for food 'identification' and food authentication-A tutorial. *Analytica Chimica Acta*, 909:9–23, 2016.
- [8] G P Danezis, A S Tsagkaris, V Brusic, and C A Georgiou. Food authentication: state of the art and prospects. *Current Opinion in Food Science*, 10:22–31, 2016.
- [9] D S Wishart. Metabolomics: applications to food science and nutrition research. *Trends in food science and technology*, 19(9):482–493, 2008.
- [10] J C Moore, J Spink, and M Lipp. Development and application of a database of food ingredient fraud and economically motivated adulteration from 1980 to 2010. *Journal of food science*, 77(4):118–126, 2012.
- [11] L Manning. Food fraud: Policy and food chain. *Current Opinion in Food Science*, 10:16–21, 2016.
- [12] EUR-Lex. Regulation (EC) No 178/2002 of the European Parliament and of the Council of 28 January 2002 laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety. *Official Journal of the European Communities*, 31:1–24, 2002.
- [13] L M Reid, C P O'donnell, and G Downey. Recent technological advances for the determination of food authenticity. *Trends in Food Science and Technology*, 17(7):344–353, 2006.
- [14] G Corrado. Advances in DNA typing in the agro-food supply chain. *Trends in Food Science and Technology*, 52:80–89, 2016.
- [15] M Formisano and G Corrado. The Evolution of DNA Typing in Agri-Food Chain. *OBM Genetics*, 3(3):1–12, 2019.
- [16] M Gallo and P Ferranti. The evolution of analytical chemistry methods in foodomics. *Journal of Chromatography A*, 1428:3–15, 2016.
- [17] L Torres Vaz-Freire, MDR Gomes Da Silva, and A M Costa Freitas. Comprehensive two-dimensional gas chromatography for fingerprint pattern recognition in olive oils produced by two different techniques in Portuguese olive varieties Galega Vulgar, Cobranc, OSA e Carrasquenha. *Analytica Chimica Acta*, 633(2):263–270, 2009.
- [18] C Ruiz-Samblás, L Cuadros-Rodríguez, A González-Casado, De Paula Rodríguez, F García, P De La Mata-Espinosa, and J M Bosque-Sendra. Multivariate analysis of HT/GC-(IT) MS chromatographic profiles of triacylglycerol for classification of olive oil varieties. *Analytical and bioanalytical chemistry*, 399(6):2093–2103, 2011.
- [19] T Cecchi and B Alfei. Volatile profiles of Italian monovarietal extra virgin olive oils via HS-SPME-GC-MS: Newly identified compounds, flavors molecular markers, and terpenic pro-

- file. *Food Chemistry*, 141(3):2025–2035, 2013.
- [20] A Bajoub, T Pacchiarotta, E Hurtado-Fernández, L Olmo-García, R García-Villalba, A Fernández-Gutiérrez, and A Carrasco-Pancorbo. Comparing two metabolic profiling approaches (liquid chromatography and gas chromatography coupled to mass spectrometry) for extra-virgin olive oil phenolic compounds analysis: A botanical classification perspective. *Journal of Chromatography A*, pages 267–279, 1428.
- [21] A Lioupi, N Nenadis, and G Theodoridis. Virgin olive oil metabolomics: A review. *Journal of Chromatography B*, 1150:122161, 2020.
- [22] M Paolini, L Bontempo, and F Camin. Compound-specific $\delta^{13}\text{C}$ and $\delta^{2}\text{H}$ analysis of olive oil fatty acids. *Talanta*, 174:38–43, 2017.
- [23] E K Richter, J E Spangenberg, M Kreuzer, and F Leiber. Characterization of rapeseed (*Brassica napus*) oils by bulk C, O, H, and fatty acid C stable isotope analyses. *Journal of Agricultural and Food Chemistry*, 58(13):8048–8055, 2010.
- [24] Y Yamini, N Bahramifar, F Sefidkon, M J Saharkhiz, and E Salamifar. Extraction of essential oil from *Pimpinella anisum* using supercritical carbon dioxide and comparison with hydrodistillation. *Natural Product Research*, 22(3):212–218, 2008.
- [25] G P Blanch, M D M Caja, M L Ruiz Del Castillo, and M Herraiz. Comparison of different methods for the evaluation of the authenticity of olive oil and hazelnut oil. *Journal of agricultural and food chemistry*, 46(8):3153–3157, 1998.
- [26] M L Ruiz Del Castillo, M Herraiz, and G P Blanch. Determination of the enantiomeric composition of γ -lactones in edible oils by on-line coupled high performance liquid chromatography and gas chromatography. *Journal of agricultural and food chemistry*, 48(4):1186–1190, 2000.
- [27] V G Dourtoglou, T Dourtoglou, A Antonopoulos, E Stefanou, S Lalas, and C Poulos. Detection of olive oil adulteration using principal component analysis applied on total and regio FA content. *Journal of the American Oil Chemists' Society*, 80(3):203–208, 2003.
- [28] F Sakouhi, C Absalon, G Flamini, P L Cioni, H Kallel, and S Boukhchina. Lipid components of olive oil from Tunisian Cv. Sayali: Characterization and authenticity. *Comptes Rendus Biologies*, 333(9):642–648, 2010.
- [29] A Rohman and Y B Che Man. Authentication of extra virgin olive oil from sesame oil using FTIR spectroscopy and gas chromatography. *International Journal of Food Properties*, 15(6):1309–1318, 2012.

Copyright: This is an open access article distributed under the terms of the Creative Commons Attribution-NonCommercial-ShareAlike 4.0 License, which allows others to remix, tweak, and build upon the work non-commercially, as long as the author is credited and the new creations are licensed under the identical terms.

Cite this article: Neelofar H, Karna Haripriya, Kishore Bandarapalle, Chekurthi Swetha Reddy, Kanugonda Jaswanth Reddy, Koka Madhavi. **Review on Food Analysis by Using Gas Chromatography.** Int. J. of Clin. Pharm. Med. Sci. 2022; 2(4): 125-135.



© 2022 Pharma Springs Publication.