Report on

Citronellol

Under supervision of staff members
of Pharmacognosy department

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Introduction

Citronellol, or dihydrogeraniol, is a natural acyclic monoterpenoid. Both enantiomers occur in nature is the primary component of rose oil (15–55%) and Pelargonium geraniums (20-40%), (+)-Citronellol, is also found in citronella oils (5-50%), peaking in Cymbopogon nardus (50%) and is the more common isomer.

Citronellol is a naturally occurring ingredient used in skincare and cosmetic formulations to improve the scent of products. It is also often used in perfume formulations to help enhance the intensity of other scent ingredients.

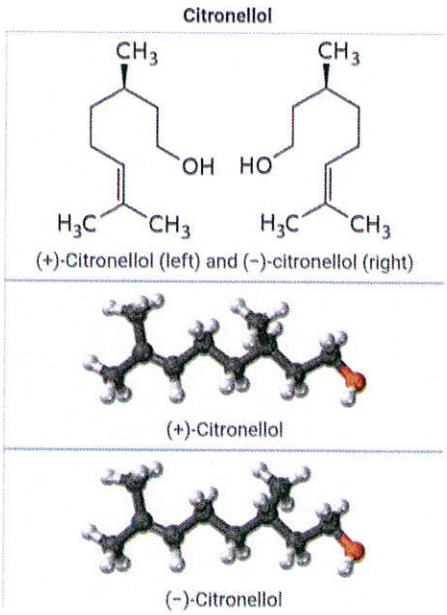
Unlike many essential oil components, citronellol doesn't cause irritation. While this is the case, it can cause sensitivity and irritation in some skin types. The low likelihood of irritation makes citronellol an ideal ingredient to improve the scent of products, such as shampoos, lotions, creams, shaving creams, and bath products.

Citronellol is known to be one of the active components of citronella oil insect repellents and citronella candles. In fact, citronella has been registered as a gentle, plant-based insect repellent. Citronella insect repellents have even been shown to repel dangerous Aedes aegypti mosquitoes, which are capable of spreading dengue fever and the Zika virus.

Structure and chemistry

-Description:

Citronellol is a monoterpenoid that is oct-6-ene substituted by a hydroxy group at position 1 and methyl groups at positions 3 and 7. It has a role as a plant metabolite.



-Properties:

- Chemical formula: $C_{10}H_{20}O$
- IUPAC: 3,7-dimethyloct-6-en-1-ol
- Molar mass: 156.269 $g \cdot mol^{-1}$
- Density: 0.855 g/cm_3
- Boiling point: 225°C (437°F; 498K)
- Melting Point: <-20°C
- Viscosity: 11.1 mPa
- Physical description: colourless oily liquid; rose-like aroma
- Taste: Bitter taste /d-Citronellol/; sweet, peach-like flavour /l-citronellol/
- Taste characteristics at 20 ppm: floral, rose, sweet and green with fruity citrus nuances
- Solubility: -In water, 200 mg/L at 25 °C
  -In water, 307 mg/L at 25 °C; 300 mg/L at 20 °C
  -Soluble in fixed oils, propylene glycol; insoluble in glycerine
- Stability/Shelf Life: Stable under recommended storage conditions.

-Occurrence:

• d-form (Cymopogon nardus).

• l-form (b-rhodinol or levocitrol) from oil of geranium (35 - 40 %) and oil of rose (20 - 35 %). in oil of citronella,

• dl-form (dihydrogeraniol) is probably formed an artifact during hydrodistillation

-Preparation:

Citronellol can be prepared by hydrogenation of geraniol or nerol

-Identification:

1- By preparation of the liquid acid phthalate which yield crystalline silver salt "m.p. 125- 126°C".

2- Upon oxidation with chromic acid it gives citronellal (aldehyde form), in which citronellal semi-carbazone derivative has a specific melting point

-Isolation:

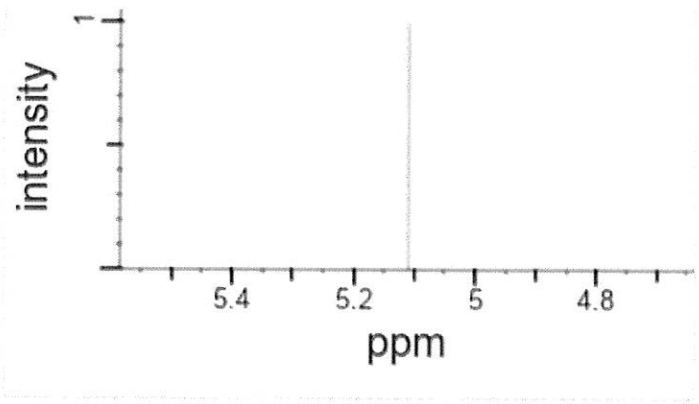
1- By fractional distillation.

2- Preparation with phthalate derivative:

- heating with phthalic anhydride at 200°C and formation of the (acid phthalate).

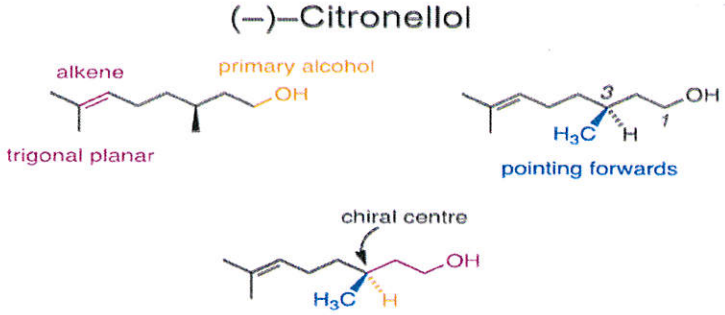
- The citronellol acid phthalate is purified with alc. KOH and extraction with ether.

-NMR spectrum:



Structure-activity relationship (SAR)

Citronellol ($C_{10}H_{20}O$), which has a fresh, floral and clean rose smell and is used in perfumes and insect repellents. From the skeletal structure we can see that (-)- citronellol contains both alkene and primary alcohol functional groups. All the carbon atoms have a tetrahedral shape apart from the two carbon atoms in the carbon- carbon double bond, which, like an aldehyde, are trigonal planar, or flat.

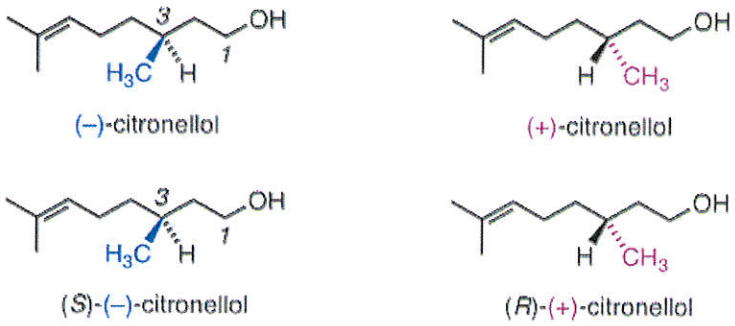


It is also interesting to look at the methyl group bonded to the tetrahedral carbon at position 3 of the carbon chain. You will see it is pointing towards us. The carbon atom at position 3 is distinctive because it is the only carbon bonded to four different groups – we call this an asymmetric or chiral centre.

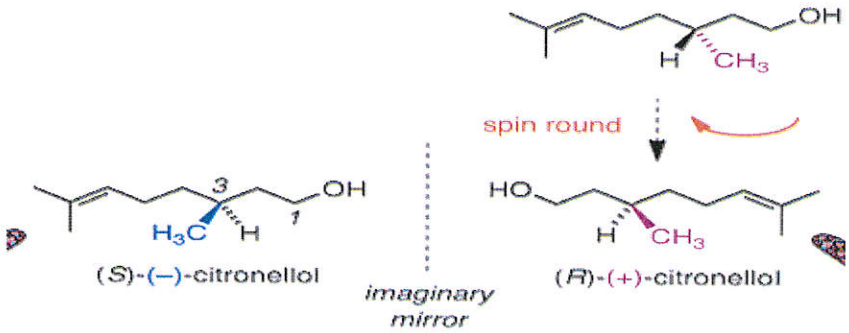
Interestingly, there are two possible ways of arranging the four different groups at a chiral centre.

In (-)- citronellol, we have seen that the methyl group at position 3 points towards us, and the hydrogen points away from us. If these groups are arranged differently, and the $-CH_3$ is pointing away from us, the molecules are non-identical structures with different shapes – indeed, when the methyl group is pointing away from us, we call it (+)-citronellol.

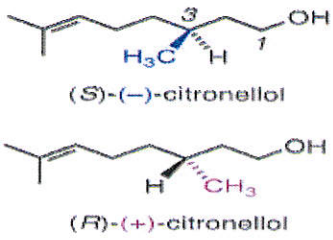
The use of (+) and (-) indicates that they rotate a special form of light, called plane- polarized light, in different directions. Often *R* or *S*, in brackets, is also included at the start of a name to indicate how the groups are arranged around a chiral centre – assignment of R *or* S is taught in our first year degree course.



If you spin around (***R***)- (+)- citronellol and compare this to (***S***)- (-)- citronellol you will see that they are non- identical mirror images – the mirror images cannot be arranged in space so that all of the atoms in both structures overlap – we say they are non- superimposable and we call them enantiomers (also known as optical isomers).



This can be tricky to visualise, so, to help, let's think of our hands. Hands are chiral – a left hand is a mirror image of a right hand. Left and right hands are not superimposable – when placed side by side the fingers and thumbs point in opposite directions. This is just the same for (***S***)- (-)- citronellol and (***R***)- (+)- citronellol, where the methyl groups point in opposite directions.



As enantiomers have different shapes they can have different biological properties – for example, the two different enantiomers of citronellol have different smells. So, it is very important to draw the structure of an enantiomer correctly.

Uses of citronellol:

Citronellol is used:

- as an insect repellent
- as an antifungal agent
- to treat parasitic infections
- to promote wound healing
- to lift mood or fight fatigue
- in perfumes or as a flavour additive in food
- as a fragrance enhancer

Pharmacological action

Citronellol disrupts membrane integrity by inducing free radical generation Citronellol, an oxygenated monoterpene, is found naturally in the essential oils of several aromatic plants and has been reported to exhibit growth inhibitory and pesticidal activities. We investigated the effect of citronellol, which is lipophilic in nature on membrane integrity in terms of lipid peroxidation, conjugated dienes content, membrane permeability, cell death, and activity of the enzyme lipoxygenase in roots of hydroponically grown wheat. Citronellol (50-250 microM) caused a significant inhibition of root and shoot growth. Furthermore, exposure to citronellol enhanced the solute leakage, increased the malondialdehyde content and lipoxygenase activity, and decreased the conjugated diene content. This indicates that citronellol induces generation of reactive oxygen species (ROS) resulting in lipid peroxidation and membrane damage. This was confirmed by in situ histochemical studies indicating cell death and disruption of membrane integrity. We conclude from this study that citronellol inhibits the root growth by ROS-mediated membrane disruption.

β-Citronellol is an alcoholic monoterpene found in essential oils such Cymbopogon citratus (a plant with antihypertensive properties). β-Citronellol can act against pathogenic microorganisms that affect airways and, in virtue of the popular use of β-citronellol-enriched essential oils in aromatherapy, we assessed its pharmacologic effects on the contractility of rat trachea. Contractions of isolated tracheal rings were recorded isometrically through a force transducer connected to a data-acquisition device. β-Citronellol relaxed sustained contractions induced by acetylcholine or high extracellular potassium, but half-maximal inhibitory concentrations (IC50) for K^+-elicited stimuli were smaller than those for cholinergic contractions. It also inhibited contractions induced by electrical field stimulation or sodium orthovanadate with pharmacologic potency equivalent to that seen against acetylcholine-induced contractions. When contractions were evoked by selective recruitment of Ca^{2+} from the extracellular medium, β-citronellol preferentially inhibited contractions that involved voltage-operated (but not receptor-operated) pathways. β-Citronellol (but not verapamil) inhibited contractions induced by restoration of external Ca^{2+} levels after depleting internal Ca^{2+} stores with the concomitant

presence of thapsigargin and recurrent challenge with acetylcholine. Treatment of tracheal rings with L-NAME, indomethacin or tetraethylammonium did not change the relaxing effects of β-citronellol. Inhibition of transient receptor potential vanilloid subtype 1 (TRPV1) or transient receptor potential ankyrin 1 (TRPA1) receptors with selective antagonists caused no change in the effects of β-citronellol. In conclusion, β-citronellol exerted inhibitory effects on rat tracheal rings, with predominant effects on contractions that recruit Ca^{2+} inflow towards the cytosol by voltage-gated pathways, whereas it appears less active against contractions elicited by receptor-operated Ca^{2+} channels.

Pharmaceutical preparations

Citronellol is found as an active constituent in the following pharmaceutical preparations:

1)Product name: Citronella Essential Oil

Although this essential oil has been typecast as an insect repellent (especially for malaria carrying mosquitoes), it also has great benefit in clearing the mind, refreshing rooms and for softening skin, while combating oily skin and sweaty feet. Furthermore, Citronella oil is a popular ingredient in wax candles and is also widely used in perfumes, soaps, skin lotions and deodorants.

2) Name of product: OstroVit Citronella Natural Essential Oil (30 ml)

OstroVit citronella oil has a pleasant citrusy aroma, while its active ingredients help you soothe pain and fight gastric problems. OstroVit Citronella Oil is obtained from the plant called West Indian lemon grass. Its composition is 100% natural and contains highly concentrated active ingredients. This is the essential oil which you can also use in aromatherapy.

3) Name of product: Pharmaid - Citronella Insect Repellant Spray (100ml)

Pharmaid-Protection Lotion Citronella (100ml)

is a natural face and body lotion with

insect repellant factors.

Citronella Insect Repellant Spray (100ml)

protects effectively against Mosquito bites,

providing a pleasant sleep.

It contains Citronella Oil, Tea Tree Oil,

soothing Chamomile Extract and Allantoin.

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Report on

Hesperidin

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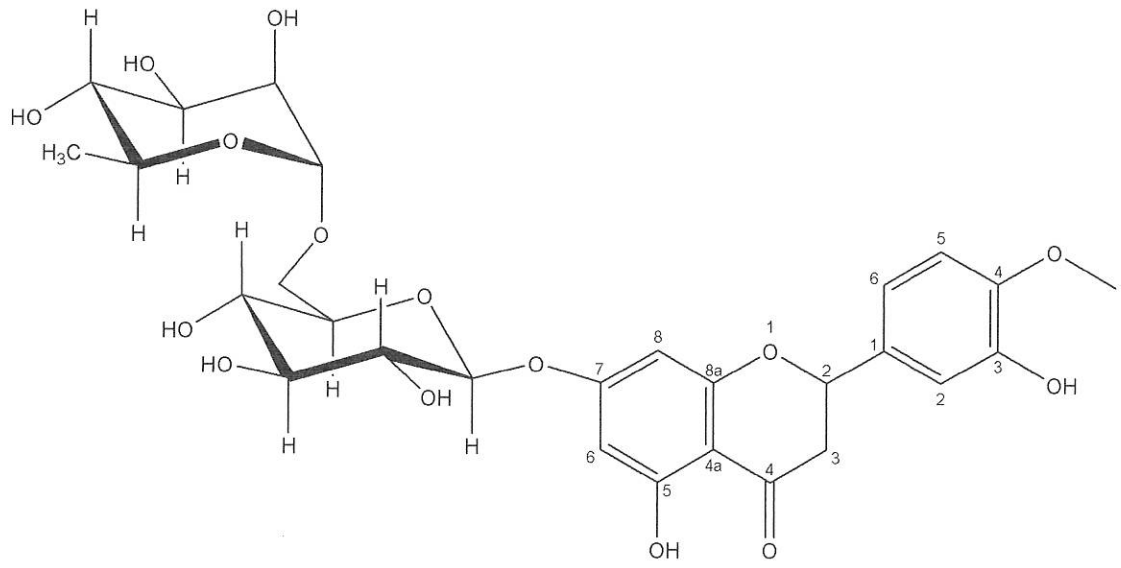
Introduction

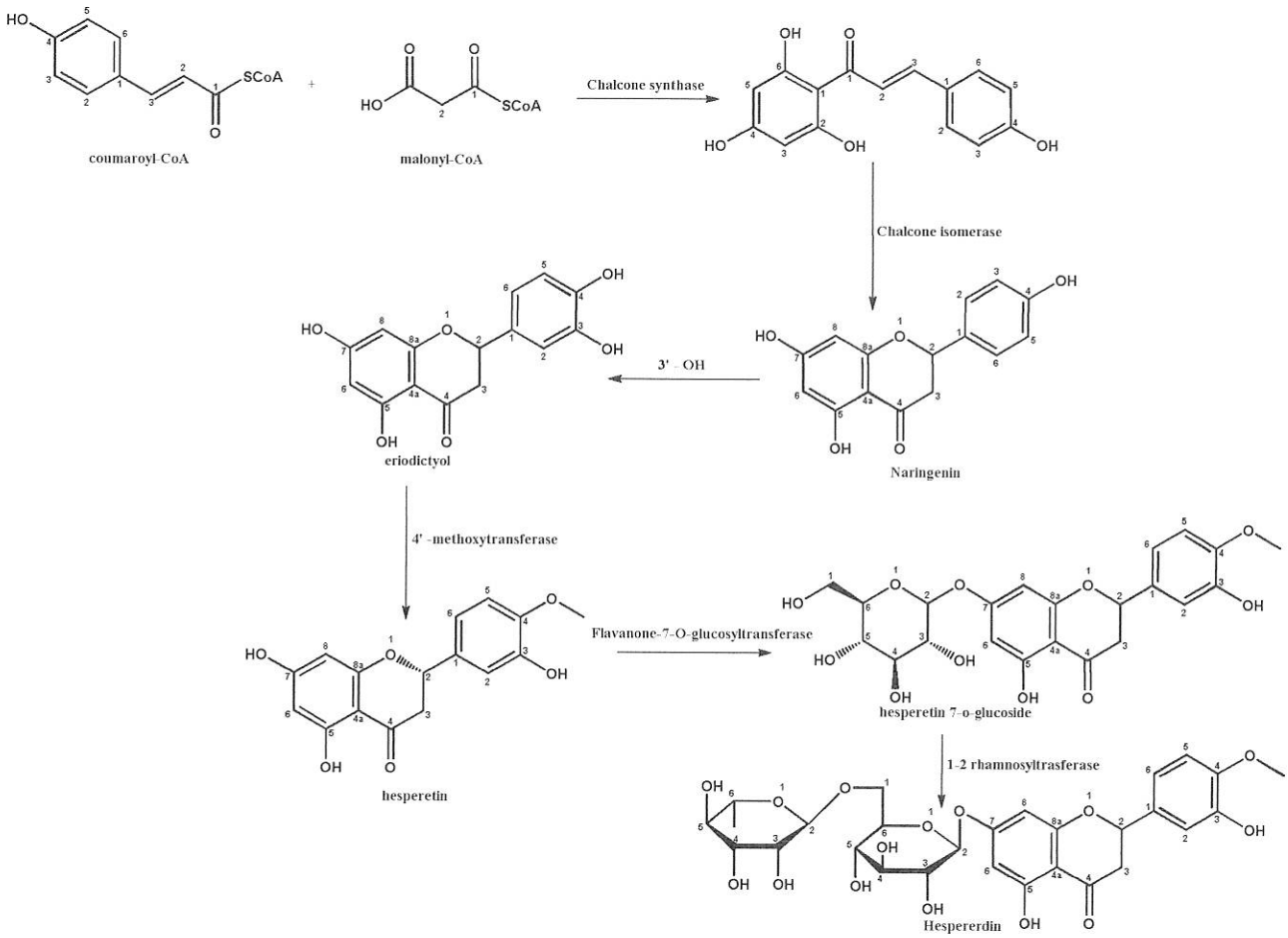
Hesperidin, a member of the flavanone group of flavonoids, can be isolated in large amounts from the rinds of some citrus species [e.g., *Citrus aurantium* L. (bitter orange), *Citrus sinensis* L. (sweet orange), and *Citrus unshiu* Marcov. (Satsuma mandarin)]. It was considered to possess a vitamin-like activity identical with or closely related to vitamin P (citrin). Hesperidin is necessary for absorption and retention of vitamin C. Hesperidin deficiency has since been linked with abnormal capillary leakiness as well as pain in the extremities causing aches, weakness, and night leg cramps. Its aglycone form is called hesperetin (1).

Hesperidin **(1)** is a disaccharide derivative that consists of hesperetin substituted by a 6-O-(alpha-L-rhamnopyranosyl)-beta-D-glucopyranosyl moiety at position 7 via a glycosidic linkage.



(1)

Biosynthesis (2, 3)



Uses of Hesperidin

Hesperidin and its antioxidant and anticancer activities (4)

Hesperidin and hesperetin have shown promising results in the suppression of various types of cancer (colon, prostate, hepatic, bladder, and lung cancer), as either a drug or as a pro-drug and co-adjuvant. Experimental findings from numerous studies have demonstrated the anticancer effects of hesperidin (Hesp) to be associated with antioxidant and anti-inflammatory activities along with its potential role in inhibiting the tumor cell metastasis and angiogenesis. Additionally, Hesperidin can also reverse drug resistance of

cancer cells, which make it a promising candidate to be used in combination with existing anti-cancer drugs.

Hesperidin and obesity (5)

Obesity refers to the pathological state in which the intake of energy is greater than the consumption, causing excessive body fat and making the body weight more than 20% of the standard body weight.

Hesperidin has anti-obesity activity. Hesperidin and hesperitin can stimulate the release of cholecystokinin (CCK), an appetite-regulating hormone, in enteroendocrine STC-1 cells, which is ultimately used to treat obesity by suppressing appetite.

Hesperidin can improve lipid metabolism. Adipose tissue stores lipids in the form of triglycerides, which secrete and regulate a variety of adipokines and cytokines. During obesity, in order to compensate for excessive lipid load, adipose tissue rapidly expands. Hesperidin (0.08%) reduces hepatic steatosis (fatty liver), adipose tissue and liver weight, and decreases serum total cholesterol and retinol binding protein (RBP). Therefore, hesperidin can treat obesity to a certain extent by regulating adipokines, cytokines, genes, and the like in lipid metabolism.

Hesperidin and chronic venous insufficiency (6)

Poor circulation, which can swell the legs (chronic venous insufficiency or CVI) - The taking by the mouth of a specific substance containing hesperidin methyl chalcone, butcher's broom, and vitamin C tends to alleviate the symptoms of poor leg circulation. It also appears to improve CVI symptoms by taking another product containing hesperidin and diosmin (Daflon) by mouth for 2-6 months.

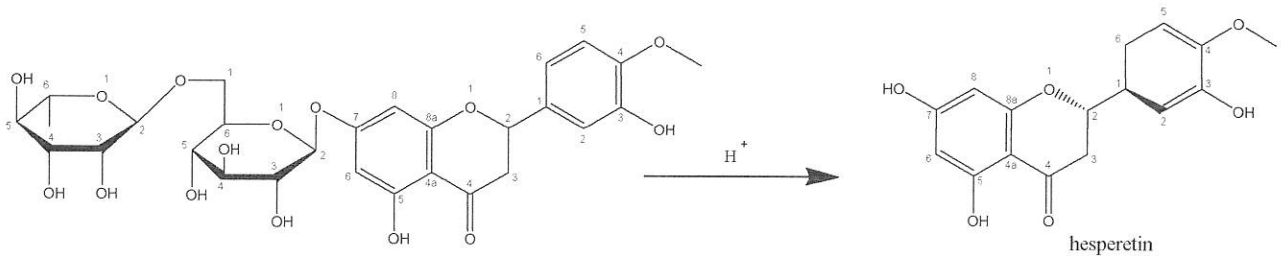
Hesperidin and SARS-CoV-2 (7)

Hesperidin may bind to multiple regions of severe acute respiratory syndrome coronavirus 2 (SARS-CoV-2) (spike protein, angiotensin-converting enzyme 2, and proteases). Hesperidin has a low binding energy, both with the SARS-CoV-2 "spike" protein responsible for internalization, and with the "PL^{pro}" and "M^{pro}" responsible for transforming the early proteins of the virus into the complex responsible for viral replication. This suggests that these flavonoids could act as prophylactic agents by blocking several mechanisms of viral infection and replication, and thus helping the host cell to resist viral attack.

Hesperidin and antimicrobial activity (7)

Hesperidin has been found to have protective effects in mice infected with encephalomyocarditis virus and Staphylococcus aureus when given before either single or combined viral-bacterial infections. Hesperidin was found to be effective against human rotavirus, which is the causative agent of diarrhea in infants and young children, and inhibited replication of influenza virus in vitro, as well as decreasing the number of infected cells. Hesperidin was also found to inhibit various sexually transmitted pathogens including Neisseria gonorrhoeae, Chlamydia trachomatis, herpes simplex virus-2, and human immunodeficiency virus, but had no toxic effects either on the host cells or on the growth of normal vaginal lactobacilli.

Hydrolysis of hesperidin (8)



Hespererdin

hesperetin

+ rhamnose + glucose

Structure Activity Relationship of Hesperidin (9)

The structural requirement considered essential for effective radical scavenging by flavonoids is the presence of 3"–o-hydroxy, 4"-o-methoxy system in the B ring, which possesses electron donating properties and is a radical target. Also, the C4'-C8' double bond conjugated with a 4-keto group, which is responsible for electron delocalization from the B ring, further enhances the radical-scavenging capacity, with saturation of the 4', 8'-double bond believed to cause a loss of potential activity. The presence of both 3"-OH and 5-OH groups, in combination with a 4-carbonyl function and C4'-C8' double bond, increases the radical scavenging activity. In the absence of a 3"–o-hydroxy, 4"-o-methoxy structure of the B ring, hydroxyl substituents in a catechol structure on the A-ring were able to compensate and become a larger determinant of hesperidin antiradical activity. Flavones possess the same basic structure as hesperidin. Flavonols that contain more hydroxyl groups (one to six OH groups) have higher free radical and superoxide anion radical scavenging abilities, such as hesperidin. hesperidin not only have a 2-hydroxyl group on the A and B-rings and on methoxy group on the B ring, but also possess the 4', 8'-double bond in conjugation with a 4-oxo function on the C-ring, which are essential structural elements in the potent radical scavenging activities of hesperidin.

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Gas chromatography

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Gas Chromatography

Introduction

Chromatography is an excellent and extremely powerful separation technique. It is fast, simple, sensitive and usually requires little sample.

Chromatography is carried out to separate an often complex sample mixture into its individual components and to obtain information in terms of:

- Qualitative analysis:

  Which components are present in the sample? A parameter that provides information for the identification of a sample component is the retention time.

- Quantitative analysis:

  How much of each compound is present? Concentrations can be determined from the peak area or the peak height in the chromatogram.

Chromatography is widely used in all kinds of applications areas, such as medical, environmental, food analysis, pharmaceutical, toxicology, quality testing and many more.

In general chromatography is based on the selective distribution of the different components between two phases. One of the phases, the stationary phase, is held immobilized inside the column while the other phase, the mobile phase travels through the column, flowing through the stationary phase. Compounds that exhibit a higher affinity for the stationary phase will travel more slowly than compounds exhibiting a lower affinity. The different sample components travel at different speed through the column and are eluted from the column at different times. The column should be sufficiently long to obtain the desired degree of separation.

Principles

A gas chromatograph is made of a narrow tube, known as the column, through which the vaporized sample passes, carried along by a continuous flow of inert or nonreactive gas. Components of the sample pass through the column at different rates, depending on their chemical and physical properties and the resulting interactions with the column lining or

filling, called the stationary phase. The column is typically enclosed within a temperature controlled oven. As the chemicals exit the end of the column, they are detected and identified electronically.

In contrast, gas chromatography uses a gaseous mobile phase to transport sample components through either packed columns or hollow capillary columns containing a polymeric liquid stationary phase. In most cases, GC columns have smaller internal diameter and are longer than HPLC columns.

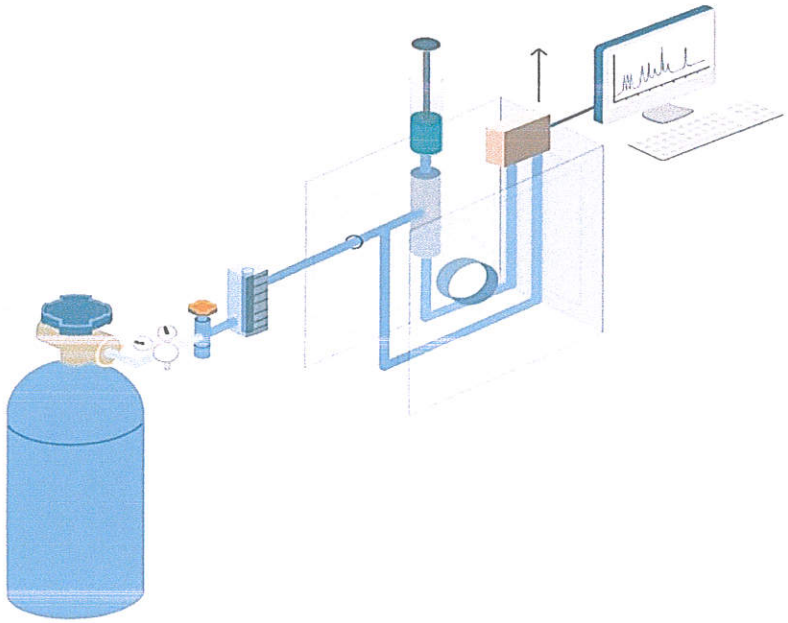
Types of Gas chromatography

It is a process of separating component (s) from the given crude drug by using a gaseous mobile phase.

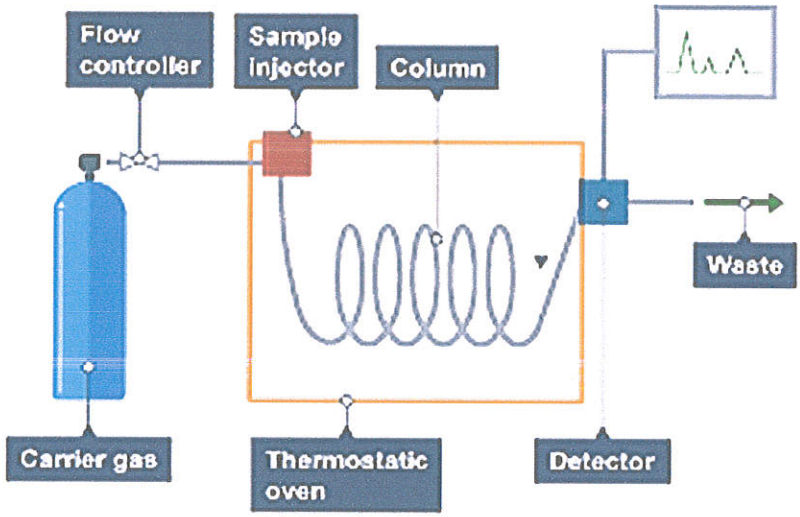
It involves a sample being vaporized and injected onto the head of the chromatographic column. The sample is transported through the column by the flow of inert, gaseous mobile phase. The column itself contains a liquid stationary phase which is adsorbed onto the surface of an inert solid.

Two major types :

- Gas-solid chromatography (stationary phase: solid)
- Gas-liquid chromatography (stationary phase: immobilized liquid)



Components of Gas chromatography



- Carrier gas
  - He (common), N2, H2, Argon
- Sample injection port
  - Micro syringe
- Columns
  - 2-50 m coiled stainless steel/glass/Teflon
- Detectors
  - Flame ionization (FID)
  - Thermal conductivity (TCD)
  - Electron capture (ECD)
  - Nitrogen-phosphorus
  - Flame photometric (FPD)
  - Photo-ionization (PID)

Carrier gas

- The carrier gas must be chemically inert.
- Commonly used gases include nitrogen, helium, argon, and carbon dioxide.
- The choice of carrier gas is often dependent upon the type of detector which is used.

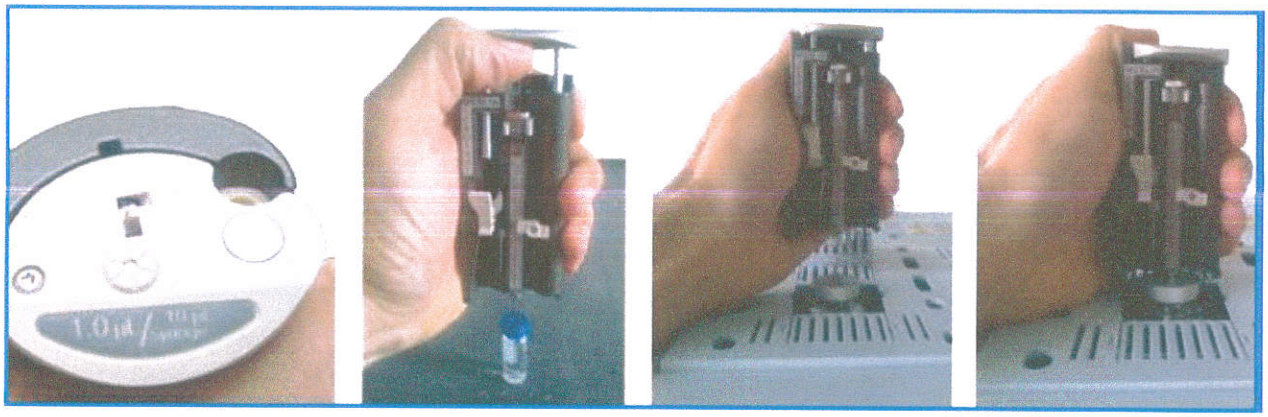
 The carrier gas system also contains a molecular sieve to remove water and other impurities

Sample injection

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

A calibrated micro syringe is used to deliver system a sample volume in the range of a few microliters into the vaporization chamber.

The sample must be of a suitable size and introduced instantaneously as a PLUG OF VAPOR Slow injection/oversize causes peak broadening and poor resolution.

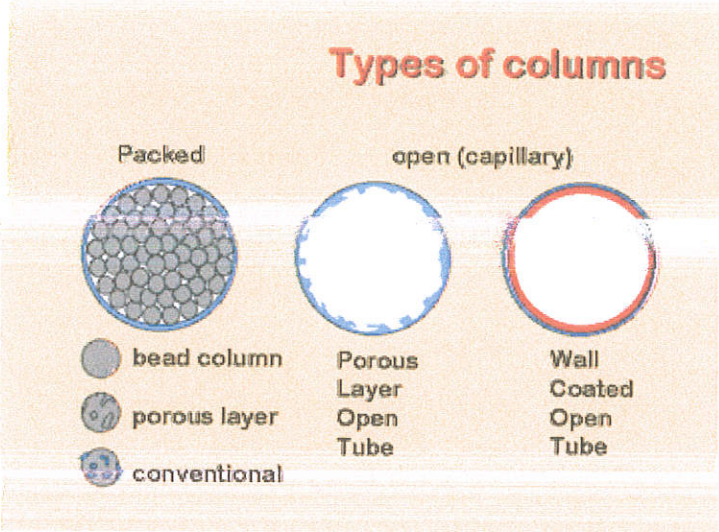


Columns

 There are two general types of column, packed and capillary (also known as open tubular).

 Packed columns contain a finely divided, inert, solid support material (diatomaceous earth) coated with liquid stationary phase. Most packed columns are 1.5 - 10m in length and have an internal diameter of 2 - 4mm.

 Capillary columns have an internal diameter of a few tenths of a millimeter. They can be one of two types; wall-coated open tubular (WCOT) or support-coated open tubular (SCOT).



DETECTORS

 There are many detectors which can be used in gas chromatography.

 Different detectors will give different types of selectivity.

IDEAL DETECTORS

 Sensitive (10-8 -10-15 g solute/s)

 Operate at high T (0-400 °C)

 Stable and reproducible

 linear response

 wide dynamic range

 Fast response

 Simple (reliable)

 Nondestructive

 Uniform response to all analytes

Advantages of Gas Chromatography

Sensitivity

Small sample is needed.

Economic

□ The cost of instrument is relatively low and its life is generally long.

Resolution

□ The technique has strong separation power and even complex mixture can be resolved into constituents.

The separation of some compounds such as methyl esters of stearic, oleic and linoleic acids by other techniques is extremely difficult or impossible. The boiling point differences are negligible in that the compounds vary only in degree of unsaturation. By using selective solvents, however, GC can provide resolution impossible by distillation or other techniques.

Speed

The entire analysis is completed less than half an hours

Analysis

□ The technique is relatively suitable for routine analysis

Two types of gas chromatography

1- gas-solid chromatography (GSC)

2 - gas-liquid chromatography (GLC).

1- gas-solid chromatography (GSC)

Gas-solid chromatography is based upon a solid stationary phase on which retention of analytes is the consequence of physical adsorption.

The stationary phase comprising of an active solid adsorbent powder is filled in an open tube. The active solid support provides an adsorbent surface on which selective adsorption and desorption of the volatile components takes place. Generally the packed columns are up to 10 m in length with internal diameters ranging from 2-4mm..

Conventionally columns are packed with porous polymers or materials such as activated carbon, molecular sieves, silica and alumina powder.

Advantages:

- Long column lifetimes
- Ability to retain and separate some compounds not easily resolved by other GC methods
- Geometrical isomers
- Permanent gases

Disadvantage:

- very strong retention of low volatility or polar solutes
- catalytic changes that can occur on GSC supports

2 - Gas-liquid chromatography (GLC).

Gas Liquid Chromatography a technique used to separate and identify the components of chemical compounds using a gaseous mobile phase.

In GLC the components of vaporize samples are fractionated due to partition between a gaseous mobile phase and a liquid stationary phase held in column.

Principle: It works based on the principle of partition.

(Gas Liquid chromatography is often synonymously referred to as Gas chromatography (GC). In this technique a nonvolatile liquid is coated as a thin layer on a powdered inactive solid support or on the inside wall of the capillary tubing. The thin liquid film serves to partition the sample components between the liquid film and the carrier gas. The inert support serves to increase the surface area of the liquid film for greater interaction with sample components.

The solid supports used include materials such as diatomaceous earths, crushed firebricks, glass powder, powdered teflon, carbon black,. Liquid phases applied generally have low volatility and high decomposition temperatures. Some typical examples are dimethyl silicone, polyethylene glycol, diethylene glycol succinate (DEGS), 50% phenyl methyl silicone.

GLC has several advantages over GSC:

- Wide range of liquid coatings afford a large range of separations
- Afford large concentration ranges to be evaluated
- Good resolution between peaks in shorter analysis time.

However, GSC can be used at higher temperatures due to limitations of volatility and instability of liquid coatings at higher temperatures. GSC is more often used for analysis of gases having no active functional groups which interact with the adsorbent surface.

Disadvantage:

- Liquid may slowly *bleed* off with time
- Contribute to background
- Change characteristics of the column with time

Similarities between gas liquid and gas solid chromatography :-

- Both are gas chromatographic techniques.
- Both uses gaseous mobile phase
- Both are used for the separation of volatile mixtures.
- Heat labile compounds cannot be separated.
- Both types used the same type of detectors.

	Gas solid chromatography	**Gas liquid chromatography**
1-the stationary phase	Solid state	Liquid state
2-mehanism	adsorption	partition
3-distribution coefficients of compounds	Much higher	Much smaller
4retention time	long	short
5-type of column	Packed column	Capillary column
6-temperature	Since liquid phase is abscent in gsc ,higher temperature can be used	The higher temperature in glc is determined by the nature of liquid phase in the glc
7-conc.of sample	Comparatively very small conc	Higher conc can be used

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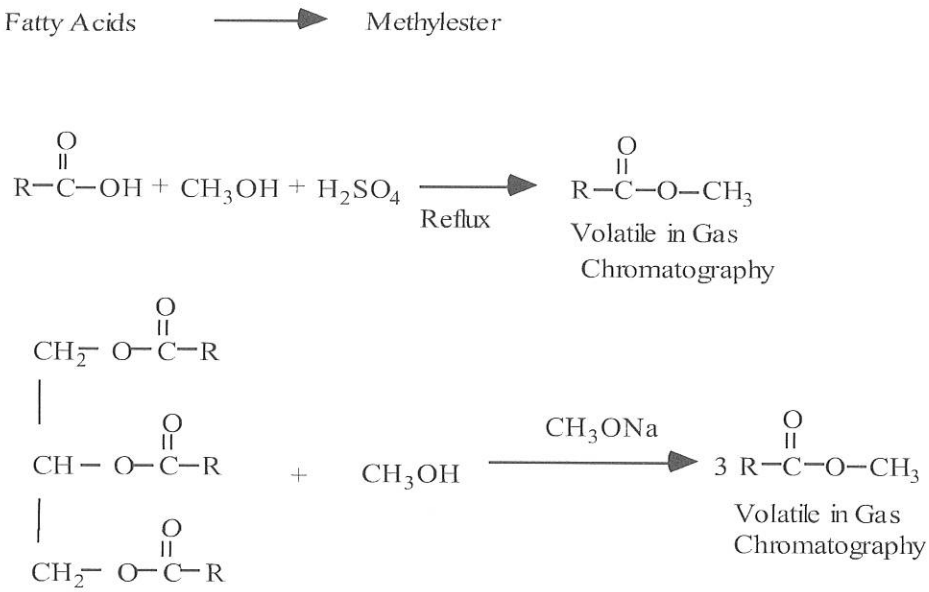
The disadvantage of GC

1- The components of the sample must be volatile at temperature at which they will not decompose

2- As there are more involatile materials than there are volatile, places a serious limitation on the field of application.

Material has to be volatilized at 250C without decomposition



Application

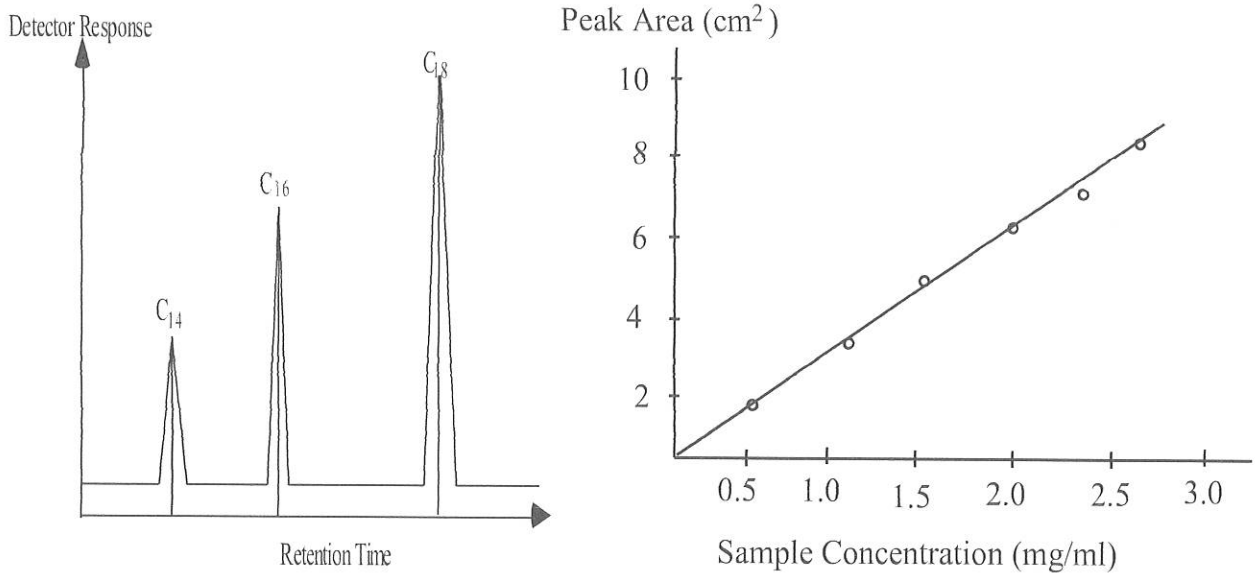
In general, substances that vaporize below 300°C (and therefore are stable up to that temperature) can be measured quantitatively. The samples are also required to be salt-free; they should not contain ions. Very minute amounts of a substance can be measured, but it is often required that the sample must be measured in comparison to a sample containing the pure, suspected substance known as a reference standard.

Various temperature programs can be used to make the readings more meaningful; for example to differentiate between substances that behave similarly during the GC process.

Professionals working with GC analyze the content of a chemical product, for example in assuring the quality of products in the chemical industry; or measuring chemicals in soil, air or water, such as soil gases. GC is very accurate if used properly and can

measure picomoles of a substance in a 1 ml liquid sample, or parts-per-billion concentrations in gaseous samples.

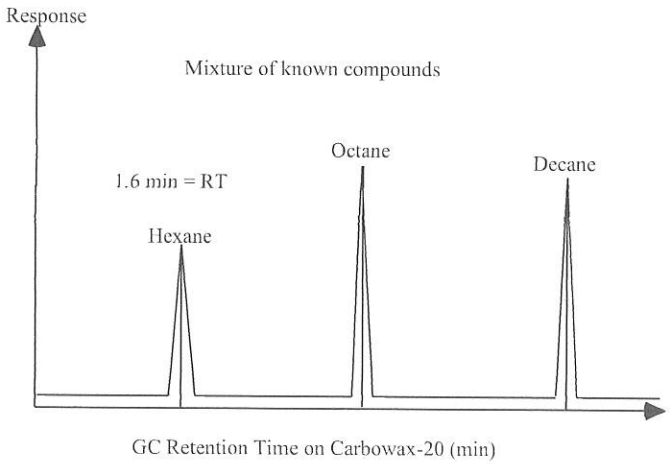
SEMI- QUANTITATIVE ANALYSIS OF FATTY ACIDS

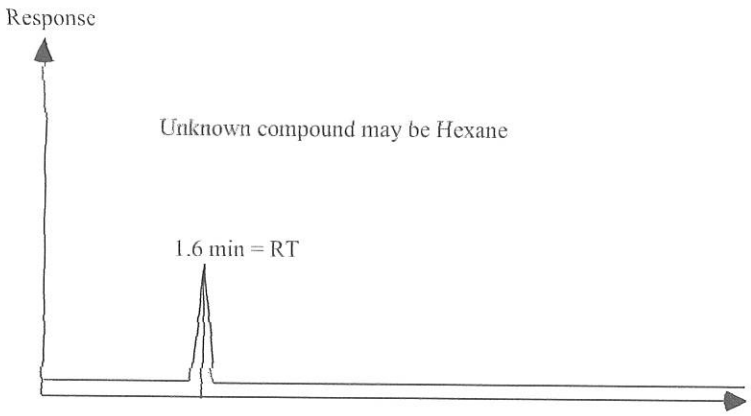


$$\text{The content \% of } C_{14} \text{ fatty acids} = \frac{C_{14}}{C_{14} + C_{16} + C_{18}} * 100$$

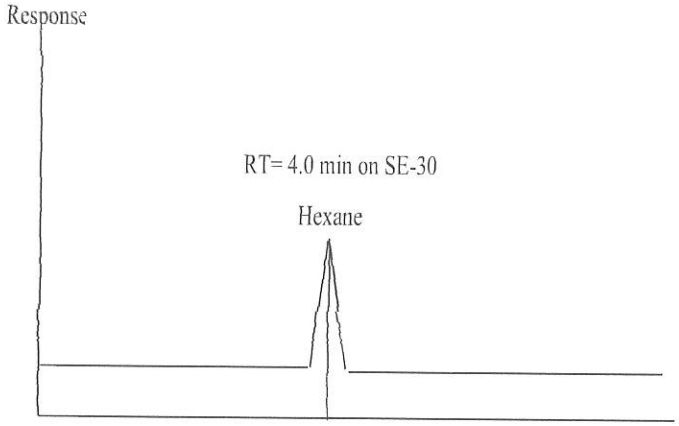
= the content % of C_4 fatty acids

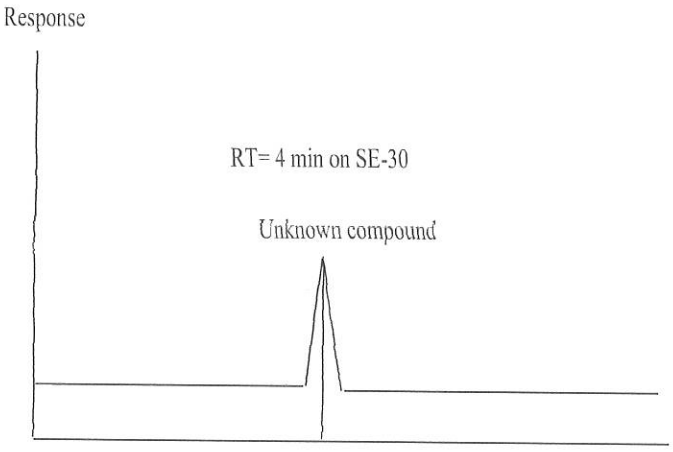
TENTATIVE IDENTIFICATION OF UNKNOWN COMPOUNDS





Retention Times





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