Chapter 7

Lemon Grass (*Cymbopogon citratus*)

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Additional information is available at the end of the chapter

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Abstract

Lemon grass is, also called fever grass, a perennial plant with thin, long leaves that is indigenous to many Asian countries. Lemon grass contains citral, which is an essential oil, having medicinal and other useful significance. In the present work, essential oil 3,7-dimethyl-2,6-octadienal (citral) is hydrodistilled from lemon grass in the laboratory, 3,7-dimethyl-2,6-octadienal acetals (citral acetals) are synthesized from citral along with para-toluene sulfonic acid as a catalyst, which are used in perfumery, flavor, for fortifying lemon oil and has strong antimicrobial qualities. Infrared spectroscopy (IR) and gas chromatography (GC) were conducted for verification of chemical constitution present in essential oil and acetals of lemon grass. Nutritionally, lemon grass is a good source of vitamins A and C, folic acid, magnesium, zinc, copper, iron, potassium, calcium and manganese. Lemon grass oil (citral) is hydrodistilled and IR and GC are conducted to verify its constituents.

Keywords: lemon grass, essential oil, citral, ionons, vitamin E

1. Introduction

Lemon grass belongs to Cymbopogon, a genus of about 55 species of grasses, native to temperate and tropical regions. It is a lofty perennial grass. Common names of Cymbopogon include lemon grass, silky heads, citronella fever grass and barbed wire grass amongst many others. Essential oil called as citral or 3,7-dimethyl-2,6-octadienal is present in leaves and twigs of lemon grass which can be extracted easily by hydrodistillation. The essential oil of lemon grass has many important chemical constituents, which are helpful for many applications. It has cis and trans citral, myrcene, geranial, etc. Citral after distillation can be used for the synthesis of ionones, vitamin A, different types of citral acetals, these acetals has a wide range of applications in perfumery and helpful to reduce antibacterial activities.

1.1. Lemon grass oil

Lemon grass is a tropical herb of 3–6 feet length, leaves and twigs of this grass have essential oil, which has insect repellent activity. Leaves of this grass dried and stored for making tea, helps to cure many problems of stomach and anesthetic problems. Lemon grass leaves from local garden of PCSIR Laboratories were collected, dried under shade to deactivate starch, and cut into small pieces of 1–2 inches. Essential oil having lemon-like aroma was extracted by steam distillation, which can be used as scent and flavoring agents in medicine. It can help in fever reduction, helpful to improve digestion, reduce diarrhea, and stomachaches. As diluted oil, it is used to ease pain and arthritis, sterile stimulating, antispasmodic, and pain reliever. Lemon grass plant is shown in **Figure 1**.

1.2. Physical characteristics of citral

Name: 3,7-dimethyl-2,6-octadienal

Molar mass: 152.24 g/mol

Appearance: pale yellow liquid

Figure 1. Lemon grass plant.

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Odor: lemon like

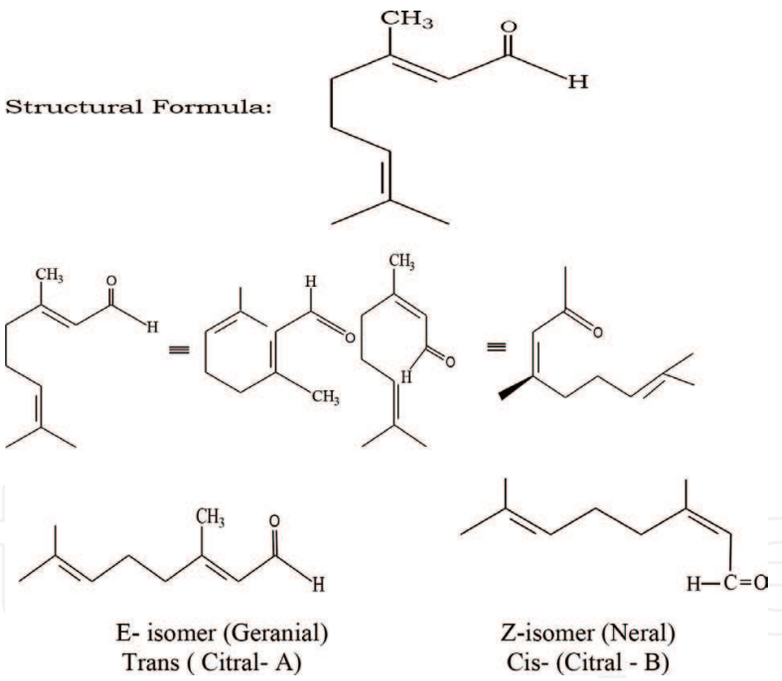
Density: 0.893 g/cm^3

Boiling point: 229°C

Refractive index: 1.484–1.490

Lemon grass essential oil (citral, 3,7-dimethyl-2,6-octadienal) or lemonal belongs to monoterpenoids having a formula $C_{10}H_{16}O$. It is a diastereoisomer of E-isomer (geranial or citral A). The Z-isomer is known as neral or citral B.

Commercial citral, is as mixture of two isomers due to cis-trans isomerism at the C=C bond nearly the aldehyde group obtained from oils of plant sources. The geranial has strong lemon-like odor while neral has less. Citral can be used in cosmetic, medicine and food industries. It also has strong antimicrobial activities [1] and pheromonal effects in insects [2]. Citral is a basic intermediate for the synthesis of flavoring and fragrance components such as ionones, methyl ionones, and vitamins A [3] and E [4]. While the essential oil from lemon grass contains 70–75% of citral in addition to myrcene, geraniol, and nerol, is used in a cheap type of soap and cosmetics.



1.3. The stereochemistry of geometrical isomers of citral

Ciral has two isomers, cis and trans. E-isomer (geranial) is trans and also known as citral A and Z-isomer (neral) is cis and known as citral B.

1.4. Sources of citral

There are two major sources of citral, which are explained in below.

1.4.1. Essential oils rich in citral

Percentage of oil (citral) varies according to plant species, about 90–98% oil is present in lemon myrtle, 70–80% in litsea cubeba, 65–85% in lemon grass, 30–35% in lemon verbena, 26% in ironbark lemon, 11% in lemon balm, 6–9% in lime, and about 2–5% in lemon and oranges [5].

1.4.2. Important plant sources of citral

Lemon myrtle contains chemotypes of two essential oils.

Lemon myrtle oil has typically 90–98% citral and oil yield 1–3% from fresh leaf. It is the highest natural source of citral.

The citronellal chemotype is uncommon and can be used as an insect repellant [6].

1.4.3. Litsea cubeba

Litsea cubeba fruits yields 3–5% essential oil. The 70–83% oil is obtained by oil's primary isolation. In China, oil production estimated between 500 and 1500 tonnes of oil per annum [7].

1.4.3.1. Lemon grass (Cymbopogon flexuosus)

Lemon grass (*Cymbopogon flexuosus*) [8], also called Cochin grass or Malabar grass, is native to India, Sri Lanka, Cambodia, Thailand, and Burma. *Cymbopogon citratus*, also known as serai in Malay, is assumed to have origin in Malaysia. While both can be used interchangeably, *C. citratus* is more used in cooking. In India, *C. citratus* is used both as a medical herb and perfumes.

1.4.4. Chemical synthesis of citral from pinene

Citral is also commercially produced from pinene [9]. The starting material is β-pinene (1) which is separated from turpentine oil by efficient fractional distillation of turpentine oil under vacuum. The β-pinene obtained in this way is passed through a heated tube with a short contact time at a temperature of about 600°C to furnish myrcene (2). The pyrolysis is thermal reaction and no catalyst is required.

Fortunately, the yield of myrcene under favorable condition can be as high as 90%. Myrcene is converted to the desired terpene mixture of cis and trans-alcohols, that is, geraniol and nerol by hydration of the double bond. This is not as easy as it would appear, to obtain a useful yield it is necessary to proceed via the hydrochloride, resulting a product, containing as much as 80% of linalyl chloride (3). The dry hydrochloric is passed through myrcene below −10°C in the presence of cuprous chloride. Linalyl chloride obtained in this way isomerizes into two isomers: geranyl chloride (4) and neryl chloride (5).

Either of the allylic chlorides can be converted to linayl or geranyl and neryl acetate by reaction with sodium acetate under suitable conditions. Thus if the sodium acetate is reacted in the presence of cuprous copper, the major product is linalyl acetate; whereas in the complete absence of copper, geranyl acetate (5) and neryl acetates (6) are predominate. The mixed terpene acetates undergo saponification to produce geraniol and nerol. These terpene alcohols are selectively oxidized to citral (7). As shown in **Scheme 1**.

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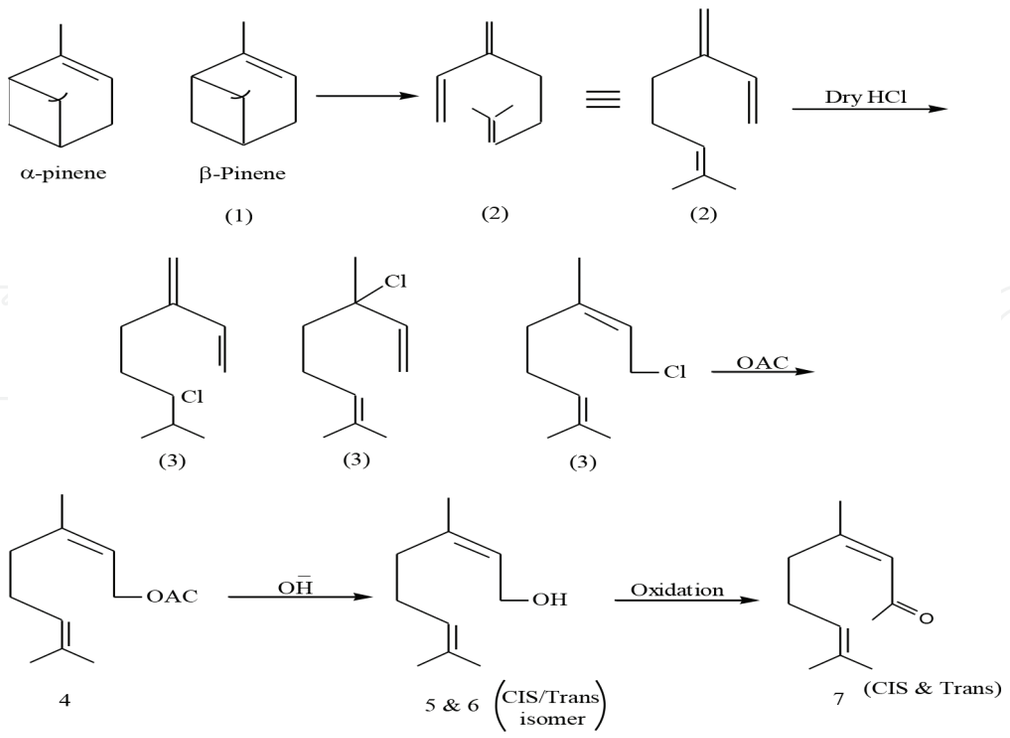


Scheme 1. Synthesis of citral from pinene.

1.5. Total synthesis of citral

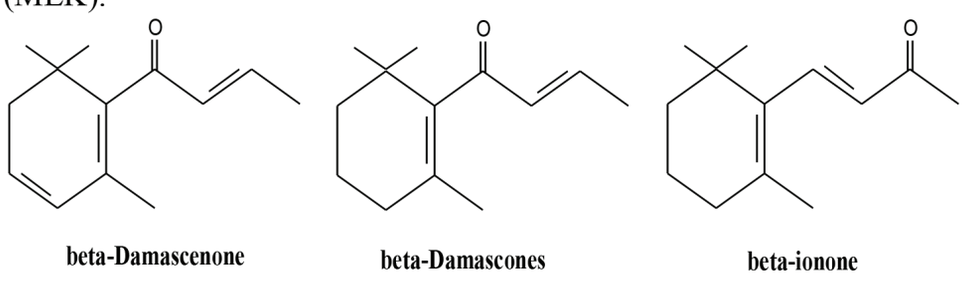
BASF opened new continuous production of citral from 2-methyl- 4-hydoxy-but-1-ene in 2004 [10]. The annual production of citral is 40,000 tons per annum, which is shown in **Scheme 2**.

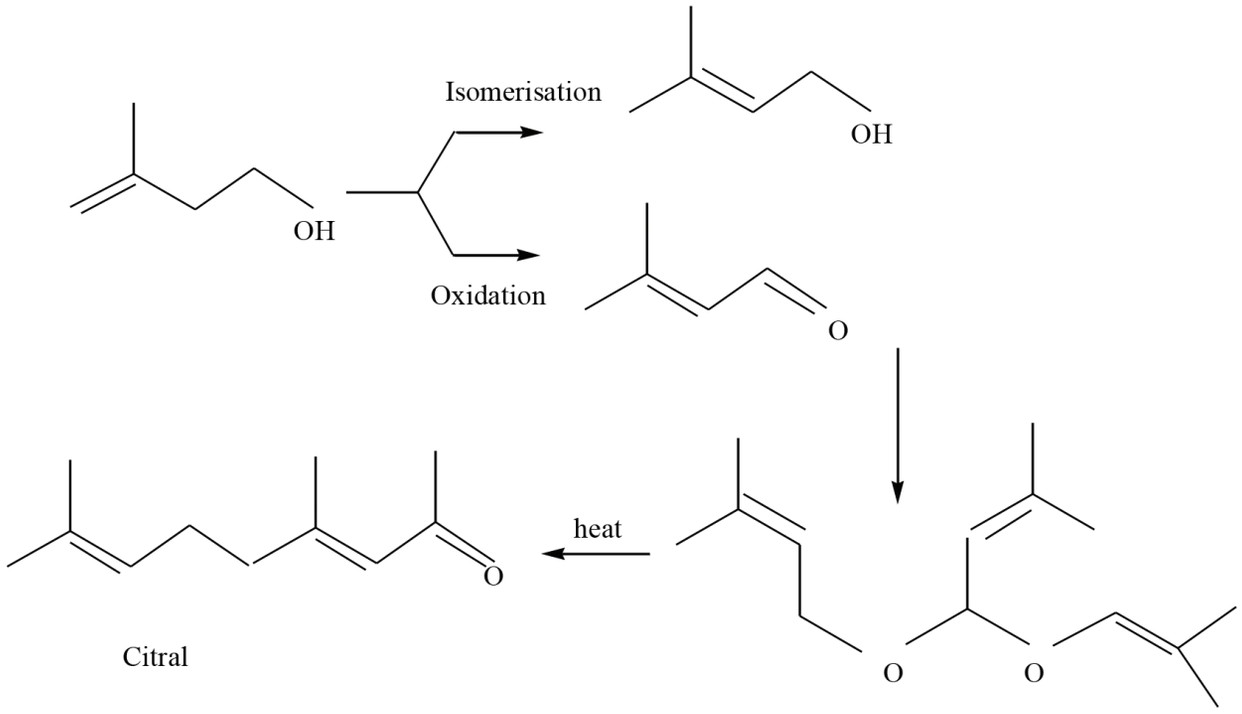
1.6. Synthetic applications of citral

1.6.1. Synthesis of ionones and methyl ionones from citral

The ionones belong to rose ketones having closely related series of chemical substances including damascones. A variety of essential oils contain these aroma compounds, the ionones, for example, β-ionone has rose aroma to some extent and it is used as a raw material for the production of retinol. Methyl-ionones are not found in the essential oils, but these are synthesized by aldol condensation of citral with methyl ethyl ketone (MEK).

(MEK).





Scheme 2. Total citral synthesis.

1.6.2. Synthesis of ionones through pseudoionones

Ionone can be synthesized from citral though pseudoionone (PS). Citral and acetone by basic homogeneous or heterogeneous catalysis give PS, and serves as an example of aldol condensation. This is followed by a rearrangement reaction in acidic media to ionones. The nucleophilic addition of the carbanion (3) of acetone (1) to the carbonyl group on citral (4) is a base catalyzed. The aldol condensation product (5) eliminates water through the enolate ion (6) to the pseudoionone (7). The cyclisation reaction of PS to ionone proceeds by acid catalysis where the double bond in (7) opens to form the carbocation (8). A rearrangement reaction of the carbocation follows with ring closure to (10, 11). Finally, a hydrogen atom can be abstracted from (9) to form either (10) (extended conjugated system) or (11) as shown in **Scheme 3**.

Besides the pseudoionones, which are a mixture of two isomers as mentioned above, several side reactions can take place too, especially self-condensation of citral leading to dimerization and polymerization. Secondary reactions are potential problems. This is shown in **Scheme 4**.

1.6.3. Synthesis of vitamin A from ionone

Ionone is a key intermediate for the production of vitamin A. Ionone (C_{13} compound) on hydroformation gives C_{14} compound (2), which is treated with acetylene compound (a C_6 compound) to vitamin A having $C_{20,}$ by series of catalytic and isomerization reactions (see **Scheme 5**).

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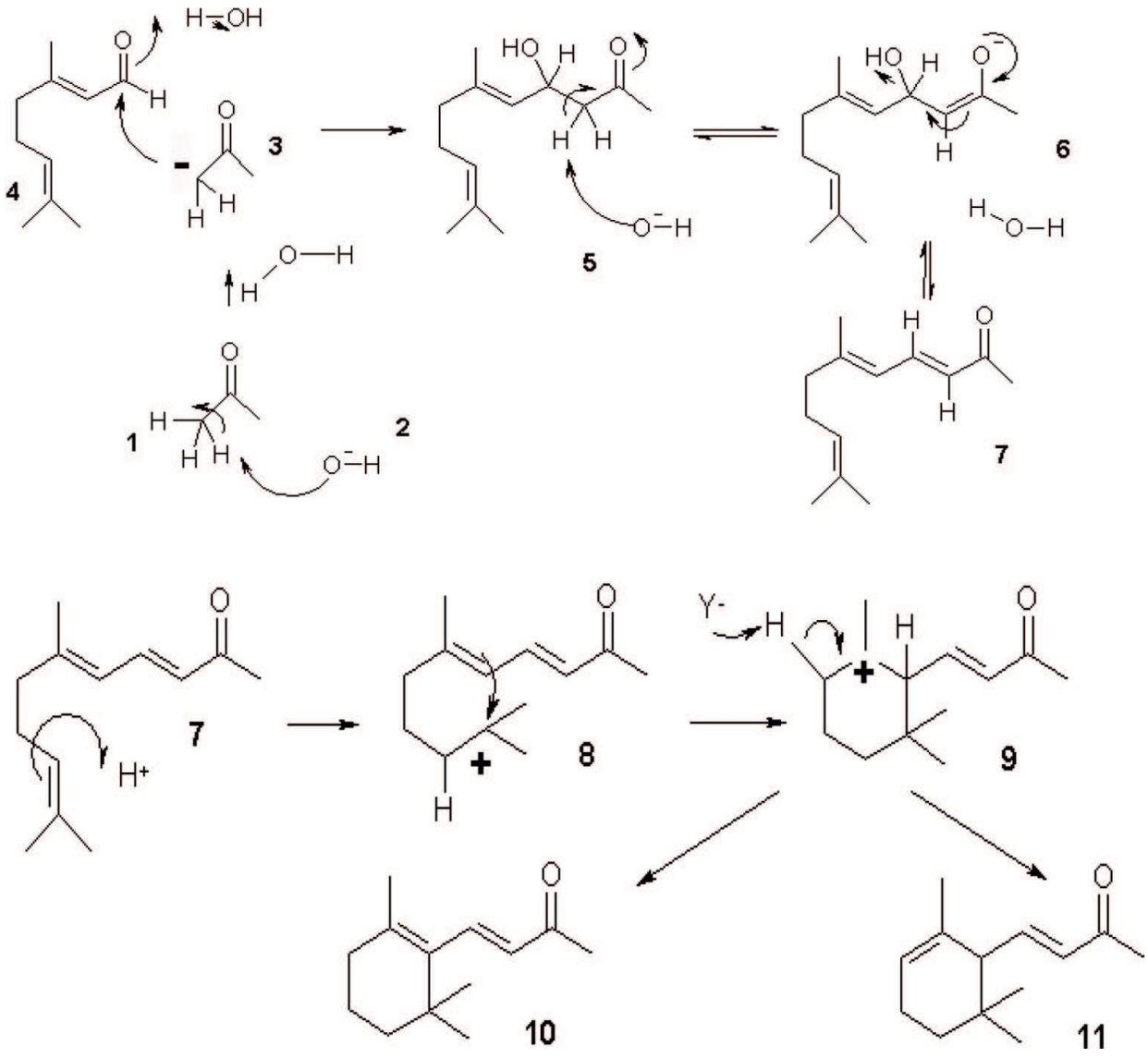


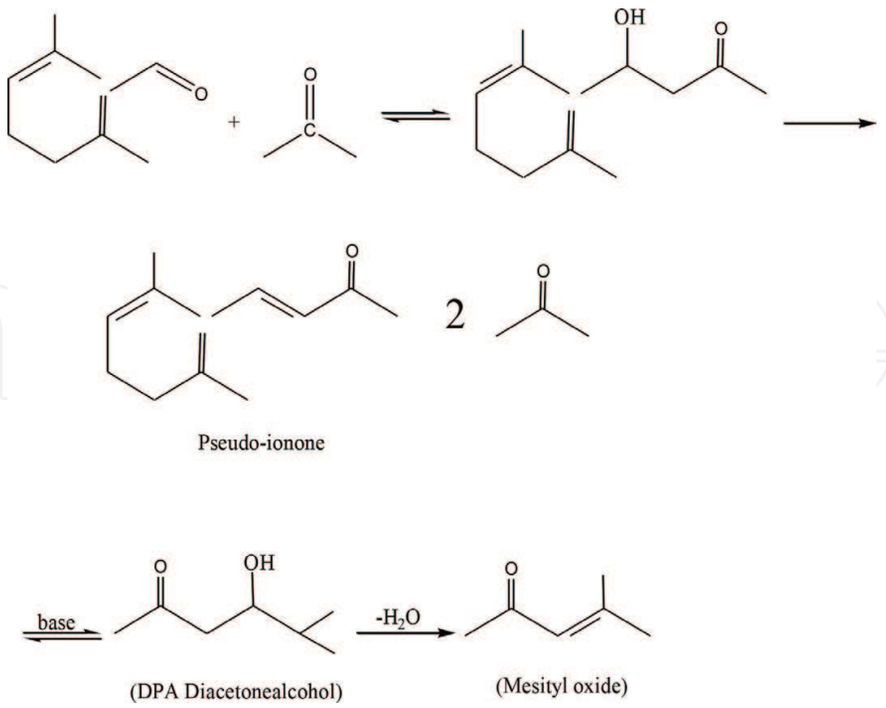
Scheme 3. Synthesis of ionones pseudoionones from citral.

1.6.4. Synthesis of vitamin E (tocopherol)

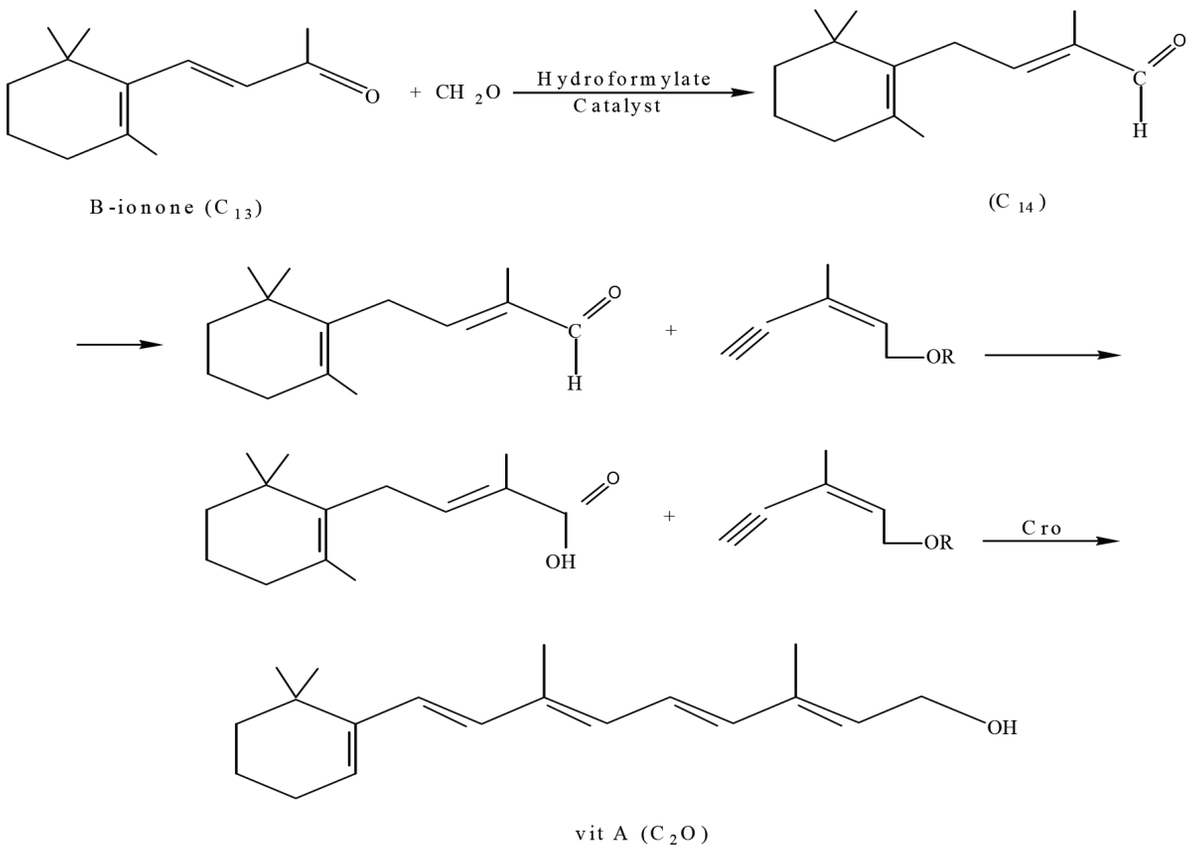
Vitamin E can be synthesized from pseudoionones and tri-methyl hydroquinone (TMHQ) as shown in **Scheme 6**.

1.7. Citral acetals and their importance in cosmetics and toiletries

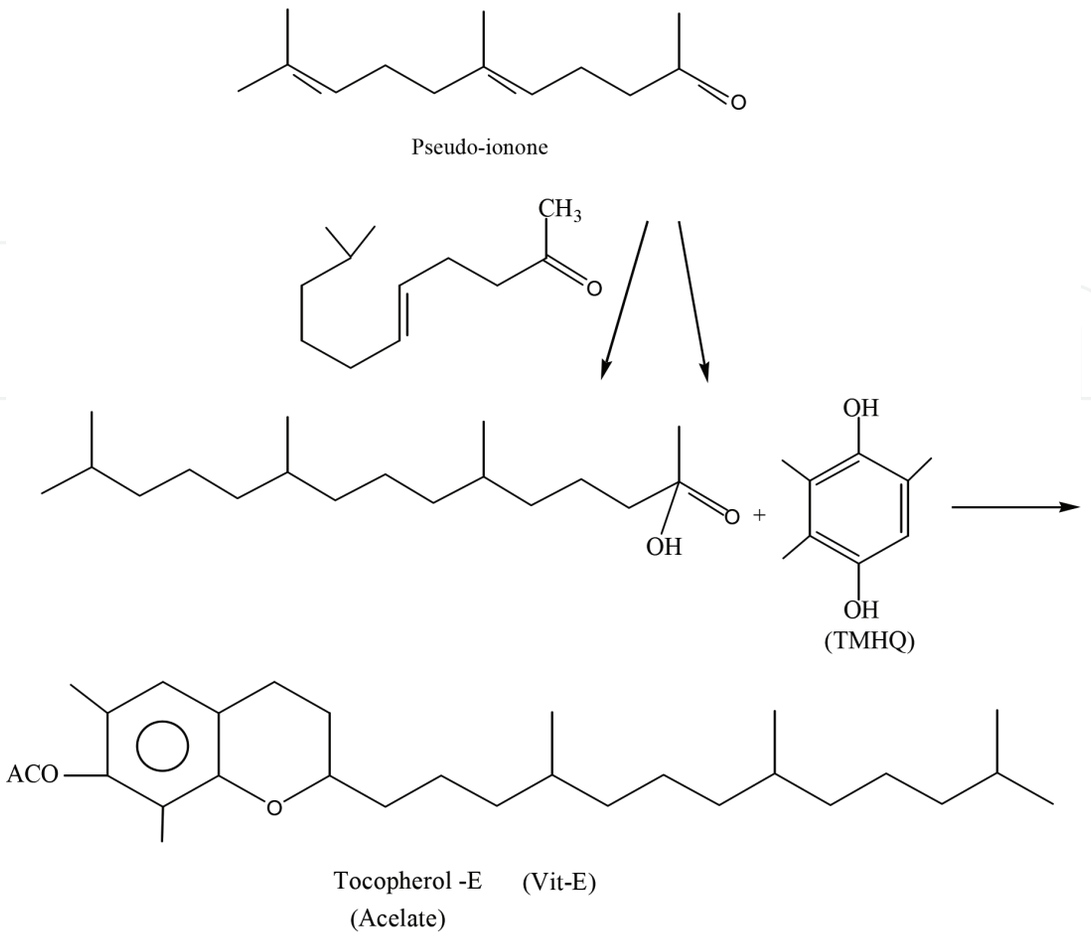
Citral has strong lemon-like aroma but it is highly volatile and instable to sunrays and alkalis, thus hardly sustaining its aroma [11]. To resolve this problem, citral dimethyl acetal and citral diethyl acetal have been used these compound have neroli-like citrus green



Scheme 4. Condensation of citral.



Scheme 5. Catalytic isomerization and synthesis of vitamin A.



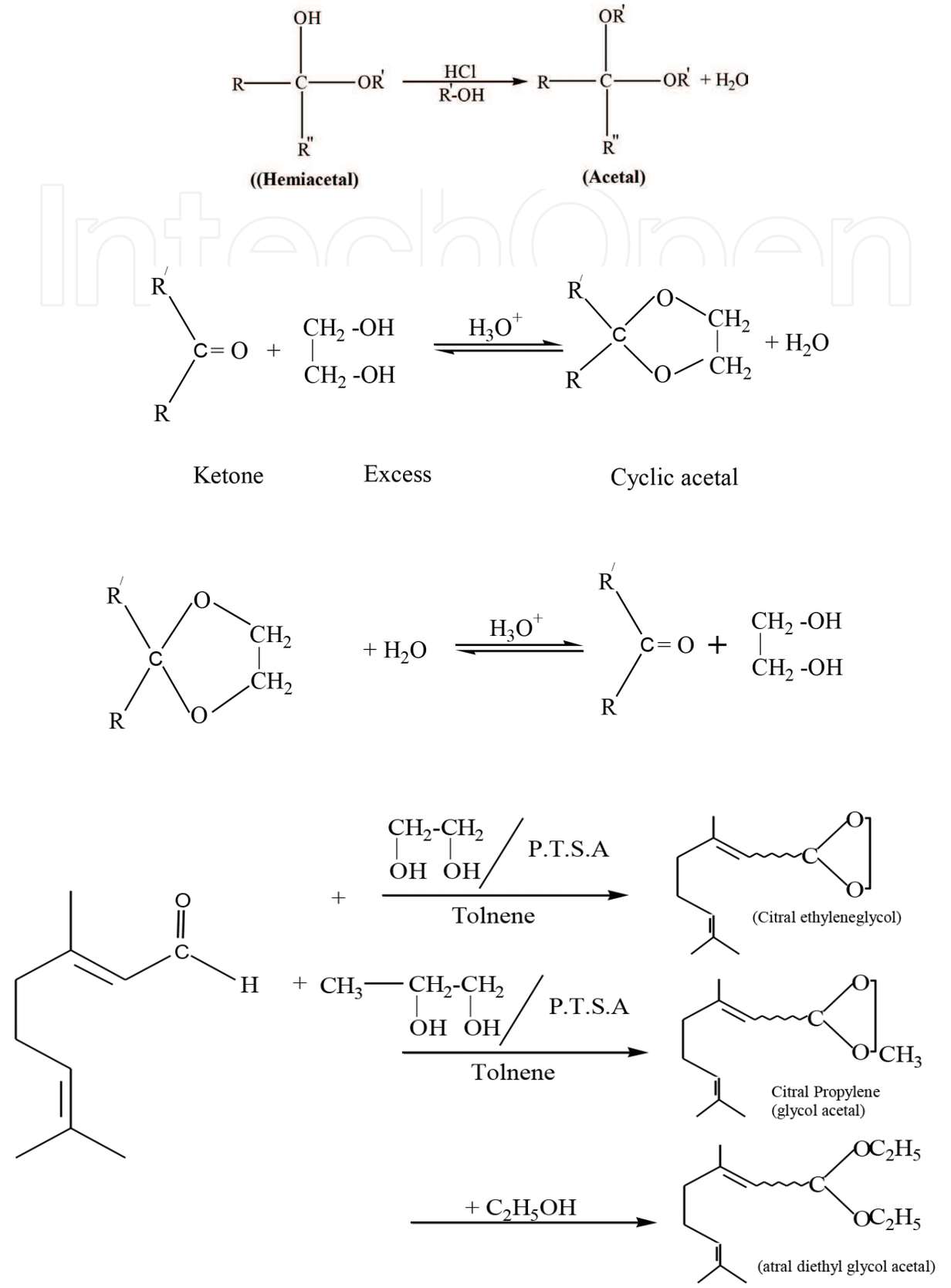
Scheme 6. Synthesis of vitamin E.

aromas. Citral propylene acetals and mono-ether glyceryl acetals are also synthesized and used in cosmetic.

These citral acetals are used in cosmetics and toiletries to suppress body smell produced by bacteria instead of antibacterial agents. Citral acetals in cosmetic formulation slowly release citral, which is antibacterial and also relative save to use.

1.8. Synthesis of citral acetals

When aldehydes or ketones are treated with alcohols in the presence of acids, it produces an acetal sometimes called ketal [12, 13]. The acetal group has two OR groups attached to the same carbon atom. The formula of acetals is not favored when ketones are treated with simple alcohols and gaseous hydrogen chloride. The formation of cyclic acetals is favored with an excess of 1,2-diol and a trace of acid. The reaction can be reversed by treating the acetal with aqueous acid. Citral ethylene glycol and propylene glycol acetals produced by azeotropic acid catalyzed reaction as shown in **Scheme 7**.



Scheme 7. Synthesis of citral acetal.

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2. Material and methods

Lemon grass leaves were harvested in the month of July, allowed to dry at room temperature for 2 days and cut into pieces of 2 inches in length then distilled in an appropriate stainless steel equipment having a capacity of 15 L along with a condenser having inlet and outlet for water, a tank is heated with a gas burner to continue and complete distillation. **Figure 2** shows a stainless steel tank for distillation.

The azeotropic mixture of essential oil is collected in a separating funnel and the essential oil of lemon grass (5.7 g) was obtained, dried over anhydrous sodium sulfate, and kept in an air tight stoppered bottle. The percentage yield of oil was determined by infrared spectroscopy (IR) and gas chromatography (GC), and obtained data lemon grass oil were plotted on graphs.

2.1. Hydrodistillation of essential oil of lemon grass in Dean-Stark apparatus

Citral (lemon grass oil) was hydrodistilled by using Dean-Stark apparatus, which consists of a round bottomed flask with the capacity of 2 L. Three-fourth of the flask was filled with 500 g of crushed dried leaves of lemon grass along with water and hydrodistilled. The oil was separated and dried over anhydrous sodium sulfate. It yields 0.38% oil. **Figure 3** shows Dean-Stark apparatus.

2.2. Analytical equipment used

Infrared spectrophotometer: Model Thermo Nicolet FT-IR 200 (USA) was used for recording absorption in the infrared region.

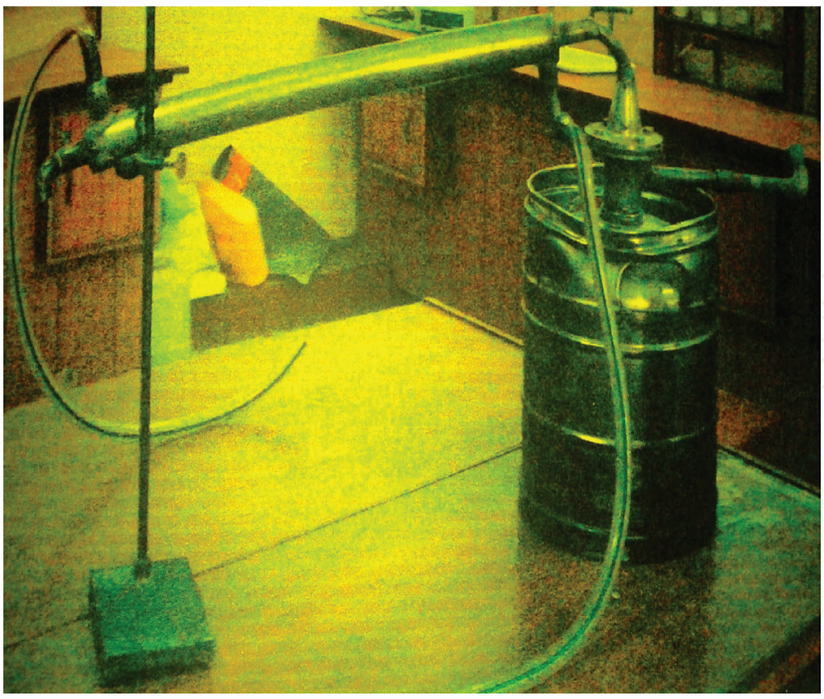


Figure 2. Stainless steel equipment for hydrodistillation of lemon grass.

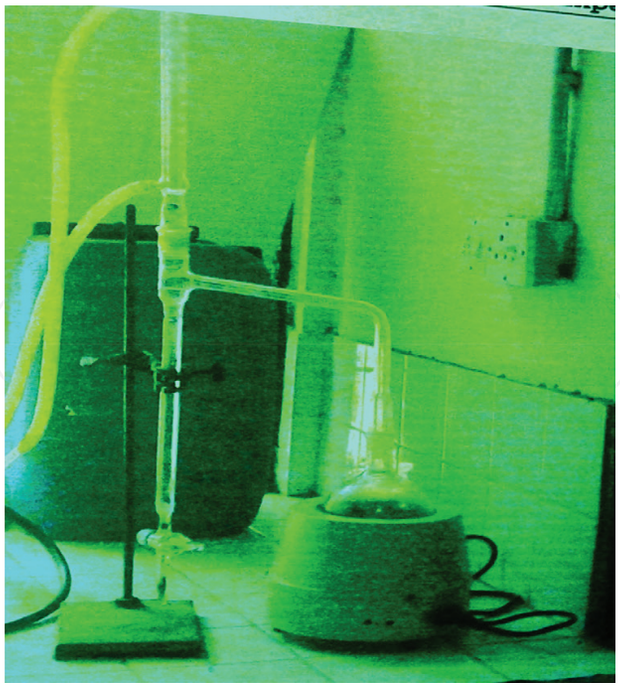


Figure 3. Dean-Stark apparatus.

Gas chromatograms: Gas chromatography studies were carried out on "Shmadzu" Model GC 14A, packed column SE-30, stationary phase PEG (polyethylene glycol), temperature condition 80–210°C, 5°C increment/min sampling recording temperature 80–210°C/10 min.

- GC of citral ethylene glycol acetal was carried out on packed column SE-30, column condition 185°C° for 2 min to 210°C for 5 min, detector flame ionization detector (FID) 270°C, injector 240°C.

2.3. Citral acetals

2.3.1. Distillation of citral

The purpose of experiment is to synthesize citral acetals, citral was redistilled under vacuum and collected the fraction between 110 and 117°C. Then IR and GC of this citral were recorded and graphs were plotted.

2.3.2. Preparation of citral propylene glycol acetal

In a round bottomed distillation flask (500 ml), a solution of citral (16 g), propylene glycol (22 g) in toluene (60 ml), and 5–7 crystals of para-toluene sulfonic acid as a catalyst is added. Then fit the flask with Dean-Stark apparatus and heated the flask at 110°C with continuous stirring. An azeotropic mixture of water and toluene was distilled off, sodium bicarbonate used to neutralize the reaction residue, benzene is used for extraction of above residue and it is dried over $MgSO_4$ then benzene distilled off and the residual product was redistilled under

vacuum and product fraction at 121–125°C is citral propylene glycol having fruity odor. GC and IR of the fractions were recorded.

2.3.3. Citral ethylene glycol acetal

To a citral solution (8 g) was added ethylene glycol (11 g) and benzene (10 ml) with few crystals of para-toluene sulfonic acid in a 250 ml round bottomed flask and stirred by heading. Water was expelled as azeotrop blend with benzene. The remaining item was extracted with hexane and cleaned with sodium bicarbonate arrangement, washed with water and hexane layer was passed over magnesium carbonate. The hexane concentrate was refined off. The left in the cup was refined under vacuum, utilized and gathered three parts at various divisions. These fractions gave fruity odor. GC and IR are recorded.

2.3.4. IR absorption spectroscopy of compounds

A very small amount of the compounds lemon grass essential oil, distilled citral, citral propylene glycol acetal and citral ethylene glycol acetal), was placed separately between two high purity plates of sodium chloride with the help of hypodermic syringe. IR spectra were then taken for these liquids. Before sampling the plates were washed with anhydrous ether then the compounds are smeared between two plates and spectra were recorded.

2.3.5. GC of compounds

A very small amount of compounds (citral and citral acetals) was injected in to a column of PEG-coated on celite support. Nitrogen was used as the carrier gas the flame ionization detector was fitted with it, the temperature was kept at 80–210°C. The peaks obtained were then identified and results were noted.

3. Results

Essential oil from lemon grass was extracted by hydrodistillation. This oil, also known as citral, is tested for its chemical composition and functional groups by gas chromatography-mass spectrometry (GC-MS) and IR spectroscopy and obtained the results.

Citral acetals by using citral was synthesized and tested by GC and IR spectroscopy, results are obtained and graphs are plotted.

3.1. Interpretation of IR spectra of lemon grass

The IR spectra of lemon grass oil having strong characteristic peaks at 3476 show the presence of OH and peaks at 2967, 2917, 2856, and 2759 cm^{-1} show the C–H stretching, a peak at 1686 shows the unsaturated conjugated C=O group present in citral, and peaks at 1650 1613, and 1445 show the C=C stretching, (see **Table 1** and **Figure 4**).

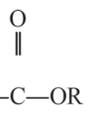
Sr.#	Peaks	Intensity	Assignment
1	3476	Broad	–OH
2	2967	Sharp & Str	–C–H
3	2917	Sharp & Str	C=O
4	2856	Sharp	CH_3,CH_2,C–H
5	1710	Str	C=O
6	1686	Sharp & Str	α,β unsaturation
7	1650	Sharp	–HC=CH–
8	1613	Sharp	–HC=CH–
9	1445	Sharp	C–H
10	1376	Sharp & Str	O–H
11	1194	Sharp & Str	
12	1153	Str	–HC=CH–
13	1120		
14	1043	Str	

Table 1. IR absorption spectra of lemon grass oil.

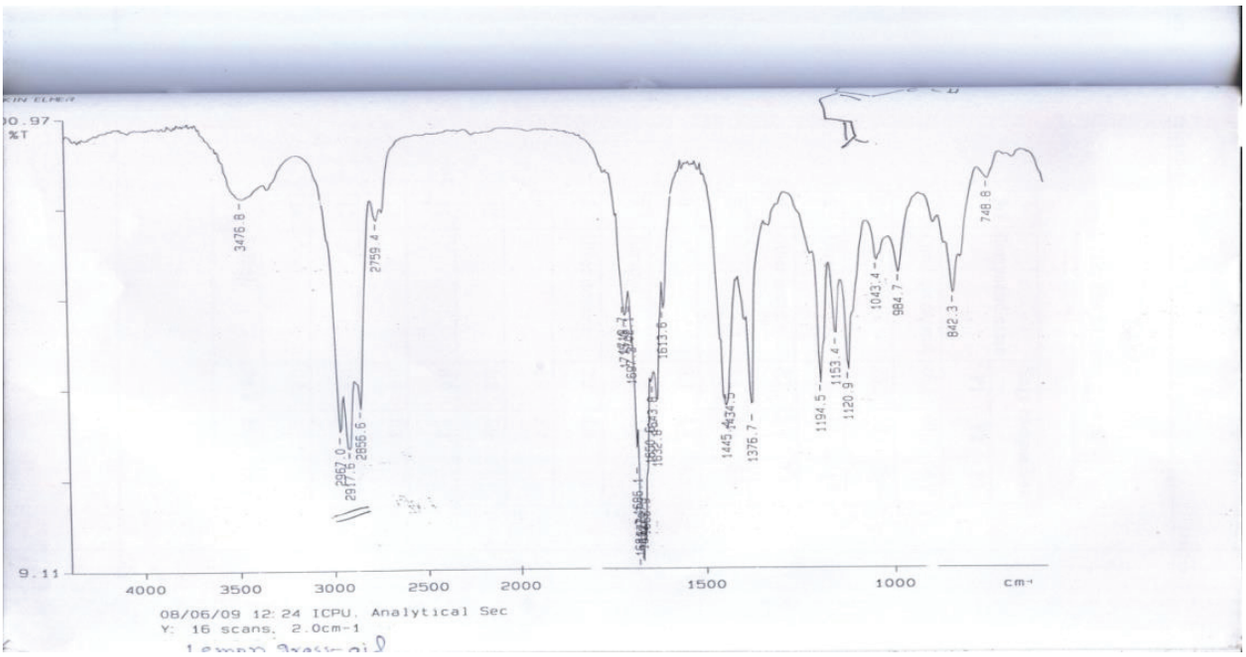


Figure 4. IR absorption spectra of lemon grass oil.

3.2. Interpretation of GC data of lemon grass oil

Different fractions of lemon grass oil spectra on GC showed peaks of six major components. It contained 36, 40 and 34% citral A and 25, 20 and28% citral B, and 7, 5 and 8% in three fractions of steam distilled essential oils, respectively. Fractions 1, 2, and 3 also showed peaks of myrcene along with geranial and other isomers. It is reported that essential oil is usually contained citral A (47%), citral B (33%), myrcene (10%), and geraniol (2%) by GC analysis, see **Table 2** and **Figure 5**, obtained by gas chromatography and gas chromatograph of lemon grass.

Sr#	Compound name	Percentage composition		
		F1	F2	F3
1	Citral (cis and trans)	36.5	40	34.6
2		25.2	10	16.4
3	Myrcene	7.1	10	11.6
4	Other isomers	3.7	5.4	7.4
5		3.7	4.8	5.4
6		3.4	4.4	4.5
7	Geraniol	2.6	3.8	3.0
8	Other isomers	2.2	3.8	
9		1.9	2.9	
10		1.1	1.9	
11		0.9	1.3	
12		0.9	1.3	
13		0.8		
14		0.8		
	Total	86.2	89.6	82.9

Table 2. GC data for lemon grass oil.

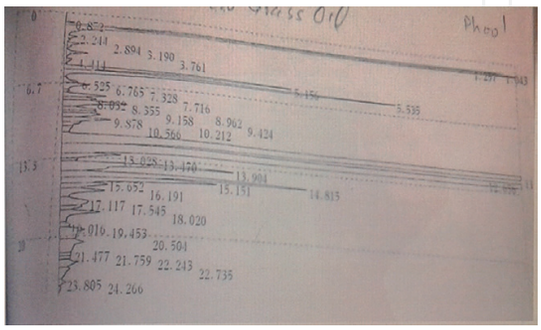


Figure 5. GC data for lemon grass oil.

3.3. Interpretation of IR spectra of citral-propylene glycol acetal

The IR spectra indicate peaks at 3019, 2925, and 2869, which is associated with the C–H stretching. The medium top peaks at 1667, 1514, 1460, and 1380 show the presence of the C=C stretching due to unimmersion in citral part in this compound. The extending vibrations at 1056, 1106, are because of ether linkage in the given compound (see **Table 3** and **Figure 6**).

3.4. Translation of GC information of citral propylene glycol acetal

The response blend of citral propylene glycol acetal later extraction with benzene, redistilled under vacuum at 64–88°C. Noteworthy, parts are appeared in **Table 4**. The redistilled compound is thymol rose. As the citral contained cis and trans geometrical isomers, it gave numerous items by response to propylene glycol, because of taking after reason.

The citral propylene glycol acetal anticipated that it would give four more isomers because of development of two hilter kilter focuses at C2 and C4 of 1,3-dioxo-4 methyl-citral acetal (see **Table 4** and **Figure 7**).

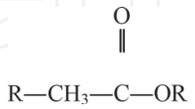
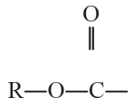
Sr.#	Peaks			Intensity	Assignment
	F1	F2	F3		
1	3019	3019	3019	Sharp & Str v. close	$-CH_3, CH_2$, C–H
2	2925	2925	2925	Sharp & Str	C=O
3	2871	2871	2869	Sharp	CH_3, CH_2, C–H
4			1894	Wk & sharp	Isomers
5			1744	Wk & sharp	
6			1667	Wk & sharp	–CH=CH–
7	1608	1608		Med	Unsaturation
8	1514	1514	1514	Sharp & med	C=C
9	1460	1460	1460	Sharp & Str	O–H
10	1380	1380	13880	Sharp & Str	
11	1108	1106	1106	Str	
12	1055	1055	1056		

Table 3. IR absorption data of citral propylene glycol acetal.

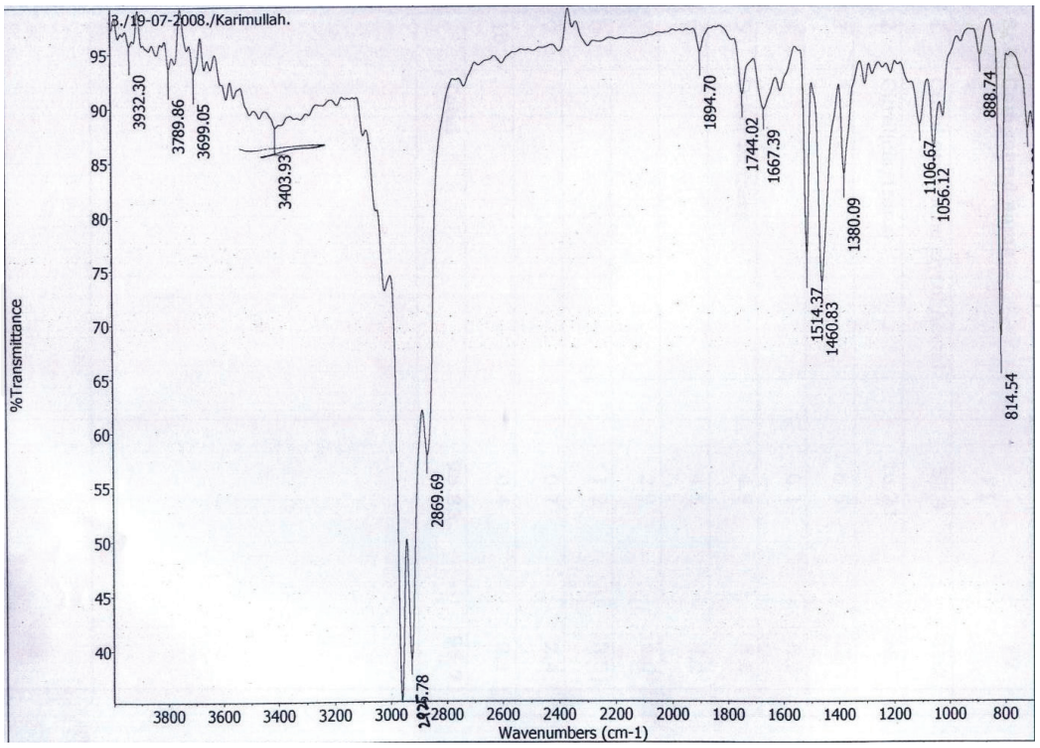


Figure 6. IR absorption spectra of citral propylene glycol acetal.

Sr.#	Compound name	% Age composition F1	F2
1	Citral propylene glycol acetal	55.4	31.2
2	Citral (cis/trans)	16.23	18.5
3		6.5	10.3
4		6.1	6.2
5	Other isomers	4.9	5.8
6		4.5	4.2
7		2.9	4.0
8		1.2	3.5
9		0.7	2.4
10		0.4	2.1
	Total	98.83	95.7

Table 4. GC data of citral propylene glycol acetal.

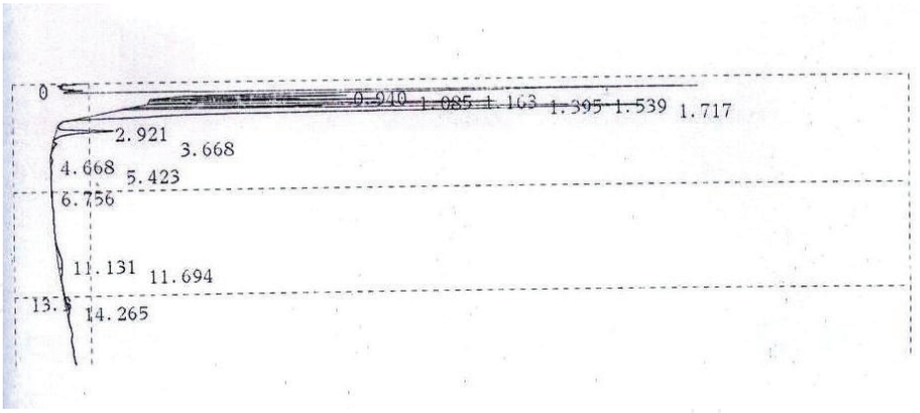


Figure 7. GC data for citral propylene glycol acetal.

3.5. Interpretation of infrared absorption spectra of citral ethylene glycol acetal

The IR spectra of citral ethylene glycol acetal have trademark top at 2924, which demonstrates the C–H extending. Top at 2867 demonstrates the nearness of CH_3, CH_2, and CH in the given compound. Crest at 1665 shows unimmersion in compound. Top at 1055 demonstrates the nearness of C–O and at 814 shows C=C. Other crest at 1513, 1460, and 1379 demonstrates the nearness of different isomers in the compound (see **Table 5** and **Figure 8**).

Sr.#	Peaks	Intensity	Assignment
1	3747	Wk	
2	3649	Med	C–H
3	2924	Sharp & Str	C–H
4	2867	Sharp & Str	CH_3,CH_2,CH
5	1665	Med	C=C
6	1513	Sharp & Str	C=O
7	1460	Sharp	C–H
8	1379	Med	OH
9	1055	Med	
10	814	Med	C=C

Table 5. IR absorption data of citral ethylene glycol acetal.

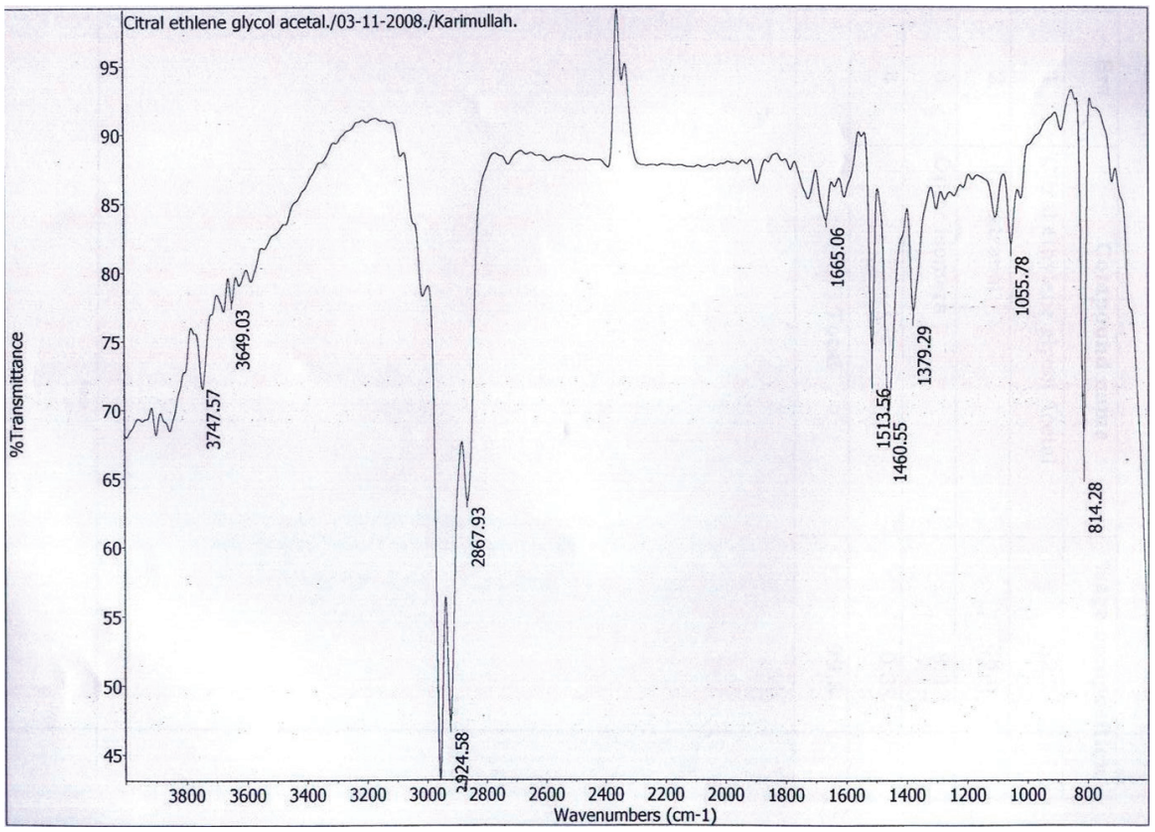


Figure 8. IR absorption spectra of citral ethylene glycol acetal.

3.6. Elucidation of GC information of citral ethylene glycol acetal

Citral ethylene glycol acetal was subjected to GC examination and rate arrangement of major components was resolved by GC, indicating two pinnacles of citral ethylene glycol acetal which are two isomers: cis and trans. This is defended as the beginning citral has two geometrical isomers. Different isomers are additionally present in little sum (see **Table 6** and **Figure 9**).

Sr#	Compound name	% Age composition
1	Citral ethylene glycol acetal	52.7
2	(Cis & trans)	38.5
3	Other isomers	8.3
4		0.3
Total		99.8

Table 6. GC data for citral ethylene glycol acetal.

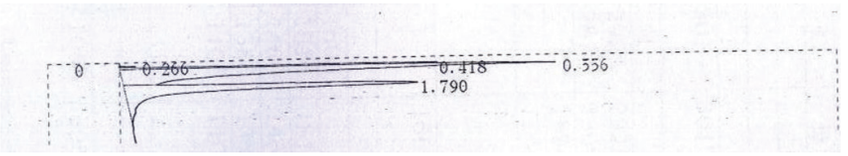


Figure 9. GC data for citral ethylene glycol acetal.

4. Conclusion

Citral is a vital transitional synthetic for the blend of flavors and vitamin A and vitamin E.

Citral is acquired from fragmentary refining of lemon grass too add up to blend from myrcene and 2-methy-4-hydroxy-yet 1-ene on business scale. The citral acetals are additionally imperative halfway for the union of flavor and in addition to their utilization in aroma and beauty care products.

The R&D development of the syntheses of various acetals and ketals is being carried out recently and their biochemical aspects are also under investigations.

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