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# 11TH INTERNATIONAL CONGRESS OF ESSENTIAL OILS, FRAGRANCES AND FLAVOURS

Vol. 2

# 11TH INTERNATIONAL CONGRESS OF ESSENTIAL OILS, FRAGRANCES AND FLAVOURS

New Delhi, India, 12-16 November, 1989



*Proceedings: Volume 2*

## KEYNOTE PRESENTATIONS

*Editors*

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**11th International Congress of  
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New Delhi, India, 12-16 November, 1989**

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## Foreword

The Eleventh International Congress of Essential Oils, Fragrances and Flavours is being held in India the first time this is a unique opportunity, the like of which may not come again in many years.

The activities of the Congress will cover almost all the aspects of essential oils, fragrances and flavours including chemistry, agriculture, botany, biotechnology, neurobiology, chemical technology, toxicology, safety, legislation, trade, industry, etc. In fact, most of the things which are likely to be of interest to us will be accommodated.

A large number of distinguished persons of diverse specialization and interest from different parts of the world have assembled for the occasion. Over 120 lectures including 81 technical lectures dealing with different facets of the growth and the development of the subjects will be delivered. In addition, many papers have also been accepted for poster presentation. In fact, we have received more papers than we can comfortably accommodate. Poster presentation, therefore, became an inescapable necessity.

Eminent scientists, industrialists, representatives of the business houses, known for their contributions, both from India and the overseas, were especially identified and invited for participation. The response has been excellent and gone beyond the point of overflowing.

Most of the lectures are being printed as part of the proceedings. In this volume, six of the 'Keynote Lectures' to be delivered by eminent scientists are published. These 'Keynote Lectures' have a little extra significance. Therefore, the papers have been printed without any alteration and noticeable editing.

India's contributions in the areas of essential oils, fragrances and flavours have been quite significant. Some of the Indian scientists have acquired international reputation. It is, therefore, in the fitness of things that the 11th Congress is being held in India. We express our grateful thanks to all the participants for gracing the occasion.

**PROF. S.C. BHATTACHARYYA**  
Chairman,  
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# Molecular Engineering for Sandalwood Aroma

Sukh Dev\*

Multi-Chem Research Centre, Nandesari, Vadodara, India

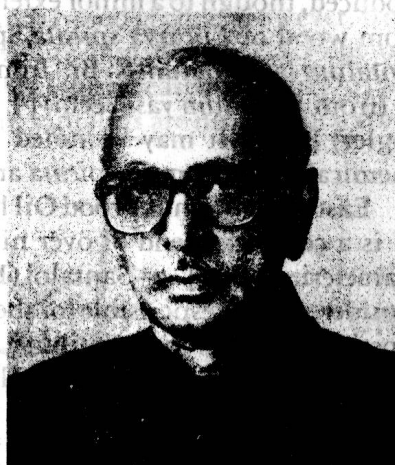
**Dr. Sukh Dev**, (b. 1924, Chakwal, undivided India) is one of the most distinguished scientists of India. He graduated from DAV College, Lahore (M.Sc. 1945) and later at the Indian Institute of Science, Bangalore (Ph.D. 1950, D.Sc. 1960).

Served as Research Associate (1948-53) and Lecturer (1953-1959) at the Indian Institute of Science, Bangalore and for one year at the University of Illinois, Urbana, USA. He then took over as The Head of the Division of Organic Chemistry, National Chemical Laboratory, Pune (1960). From there he moved (1974) to Baroda as the Director of MALTI-CHEM Research Centre, from where he retired recently (1988). Currently he is INSA S.N. Bose Research Professor at the Indian Institute of Technology, New Delhi. He had been associated with several universities overseas as visiting professor.

In Honorary Editorial Advisory capacity he has been associated with Tetrahedron and Tetrahedron Letters, Indian Journal of Chemistry and several other very prestigious publications. He is the recipient of many awards and honours such as Sudborough Medal of IISc, Bangalore (1949), Guha Research Medal (1958), Shanti Swarup Bhatnagar award of the CSIR (1965), Acharya P.C. Ray Memorial Medal (1970), Dr. K.G. Naik Gold Medal, (1977), Vishwakarma Medal, Indian National Science Academy (1979), Ernest Guenther Award, American Chemical Society, USA (1979), Distinguished Alumni Award, IISc, Bangalore (1980), VASVIK Award, Vividhaxi Audyogik Samshodhan Vikas Kendra, Bombay (1980), FICCI Award, Federation of Indian Chambers of Commerce & Industry (1980), Prof. T.R. Seshadri Seventieth Birthday Commemoration Medal, Indian National Science Academy (1981), Meghnad Saha Medal, Indian National Science Academy (1987), Satyendra Nath Bose Research Professorship, Indian National Science Academy (1988).

He is a Fellow of the Indian National Science Academy (1968) and Indian Academy of Science (1974), and has been President of the Indian Chemical Society (1978-79).

He has been the Member of several important National and International Committees; was the Council Member, Indian National Science Academy, New Delhi (1986-88). He has been closely associated with the activities of the IUPAC and is currently the President, Organic Chemistry Division of IUPAC (1987-89). He has been invited to give lectures in many Universities and Institutions in India, Europe, the U.S.A., U.S.S.R., Japan, Australia, and Canada, and has participated in many international conferences as Chairman and Plenary Speaker. His research interest is extensive and wide and he has made outstanding contributions on essential oils, fragrances and flavours, including isolation, structure determination, synthesis of a large number of compounds. He is author of 50 patents and over 260 papers which have appeared in leading national and international journals. He is author of several noteworthy books on Terpenoids.



## ABSTRACT

3, 6, 6-Trimethylbicyclo [3.1.0] hexane-3-carboxaldehyde (abeo-caranaldehyde), a rearrangement product of carene epoxide, has been exploited as a building block for tailoring molecules expected to have sandalwood aroma. From these investigations 1'- (3, 6, 6-trimethylbicyclo [3.1.0] hexan-3-yl) -2'-methylpent-1'- (E)-en-3'-ol emerged as a good sandalwood aroma compound. Effect of geometry of the olefinic linkage, cleavage of cyclopropane ring, elongation of side-chain on sandalwood aroma has been studied. Structural requirements for sandalwood aroma are discussed.

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## INTRODUCTION

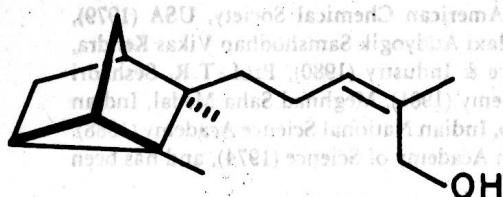
### East Indian Sandalwood Oil

Sandalwood, the fragrant heartwood of *Santalum album* Linn., has been recognized as a precious perfumery material in India since antiquity. Sandalwood (*Chandan* in Sanskrit) is mentioned in *Charaka Samhita*, the ancient (~ 900 B.C.) medical treatise of India, and in the still more ancient *Vedas*. Water distillation of sandalwood oil has been practised as a craft in India since ancient times.

Sandalwood oil is valued in perfumery for its sweet, warm, precious-woody fragrance of great tenacity. It is used as base in the traditional *attars* of India. Current world production is estimated to be ~ 100 tonnes [1]. Indian production is of the order of 60 tonnes/annum, while the balance is manufactured mostly in Indonesia. Sandalwood oil is produced by steam distillation of heartwood and roots; average yield amounts to 4-6% [2].

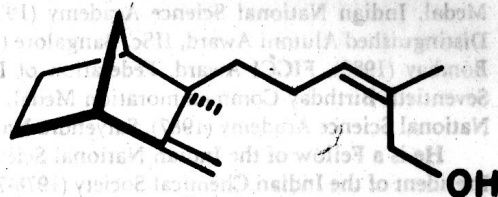
Sandalwood oil obtained from *Santalum album* is known in commerce as the East Indian Sandalwood Oil. Although commercially and fragrance-wise, this is the most important oil, several other products labelled as sandalwood oils, with diverse aroma profiles, have been or are being produced, though to a minor extent. Of these, mention may be made of Australian sandalwood oils (from wood of *Eucarya spicata* Sprag et. Summ. syn. *Santalum spicatum* A. DC.; from wood of *Santalum lanceolatum* R. Br.; from wood of *Santalum preissianum* Miq.) [3, 4], Fijian sandalwood oil (from *Santalum yasi* Seem) [4, 5], and African sandalwood oil (from wood of *Osyris tenuifolia* Eugler) [3, 4]. It may be noted that all these trees/shrubs belong to the family Santalaceae. Chemically oils from *E. spicata* and *S. yasi* are similar to East Indian Sandalwood Oil.

East Indian Sandalwood Oil has been thoroughly examined for its constituents [4, 6, 7, 8]. The oil is a complex blend of over ninety chemical entities, of which at least 50 have been suitably characterized, [6, 7].  $\alpha$ -Santalol (1) and  $\beta$ -santalol (2) account for up to 90% of the oil. Santalols are considered to be responsible for the warm, soft, woody character of the oil, while the minor and trace constituents possibly contribute towards the spicy, precious woody nuances. Minor and trace compounds, identified so far, belong to the class of hydrocarbons, alcohols, aldehydes, ketones, acids, phenols and heterocycles.



(+)-  $\alpha$ -Santalol

1



(-)-  $\beta$ -Santalol

2

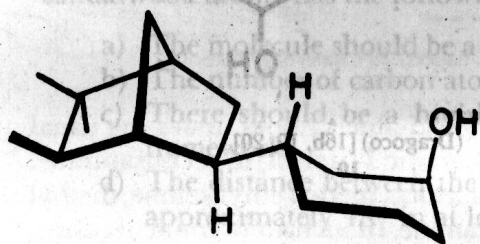
### Synthetic Compounds with Sandalwood Aroma

As already stated, East Indian Sandalwood Oil is highly prized in perfumery. The sandalwood tree, a small to mediumsized evergreen semi-parasitic plant, requires special climatic and soil conditions. In India, it is found in the comparatively dry regions of peninsular India, from where most of the sandalwood is obtained. Outside India, it is found in certain parts of Malayan Archipelago. Plantations of this tree have not been very successful. It is a slow-growing tree, reaching full maturity at an age of sixty to eighty years. As the tree grows, essential oil develops in the heartwood and roots. In a fully grown tree the heartwood has a girth of 40-60 cm, and the total yield of heartwood from such a tree may vary from 50 to 200 kg [2, 3]. Thus, the natural supply of sandalwood oil is rather restricted and the oil is quite expensive. During 1974, due to shortages, the price of sandalwood oil rose sharply from some US\$ 45/kg to 250/kg. Current price is around US\$ 180/kg. In view of this, sandalwood oil aroma has been a good target for cheaper synthetic compounds, which could be used as replacements or as extenders. Of course, it was realized that no

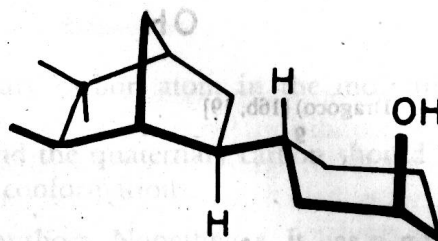
single chemical entity will ever duplicate the sandalwood oil aroma, as it is a complex blend of many compounds. All what is wanted is to get at that precious "sandalwood note".

Even before the 1974 price crunch, two synthetic compounds with sandalwood odour have been on the market. The first one to be introduced was Sandela (Givaudan, ~1960), a complex mixture of isomeric terpenylcyclohexanols. This material was first developed in Germany, and came to the notice of other countries from the technical data collected by the U.S. Army Intelligence [9]. Since then, a number of companies have undertaken its production under different trade names. It was only in 1964 that the exact structure (3) of the compound responsible for the sandalwood note in the product, could be established [10]. Later [11], it was shown that another isomer (4) has also strong sandalwood odour. These compounds are present in different preparations to a small extent (~5-8%) only. Since 1970, a lot of work has been carried out to modify the process such that the desired compounds (3, 4) are formed in larger amounts [12]. These products containing varying amounts of active 3-*trans*-isocamphylcyclohexanols (3, 4) are used in large quantities (>200 tonnes) in perfumery compositions. The second sandalwood aroma compound, Osyrol (5), was introduced by Bush Boake Allen in the early seventies [13].

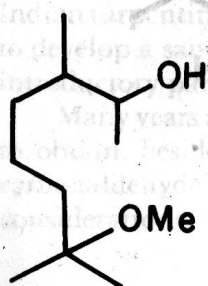
Both the above products appear to have resulted from chance discoveries and not from any specific research effort aimed at synthesizing compounds with sandalwood aroma. However, from around 1970 several groups appear to have launched efforts in this direction. Santalols are obvious targets. Though several synthesis of santalols have been achieved [14] and at least one effort [15] was directed towards a strategy aimed at commercial implementation, santalols are not manufactured



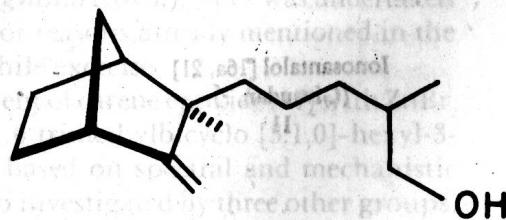
Threo-isomer  
3



Erythro-isomer  
4



Osyrol  
5



Dihydro-β-santalol  
6

commercially by any synthetic method. However, during these investigations interesting aspects of structure and sandalwood aroma, came to light:

- \* Racemic santalols have odours similar to these of naturally occurring optical isomers.
- \*\* Dihydro-β-santalol (6) has a strong sandal odour; thus side-chain olefinic linkage appears unimportant.
- \*\*\* Geometry of side-chain double bond does not have any profound effect on the odour.

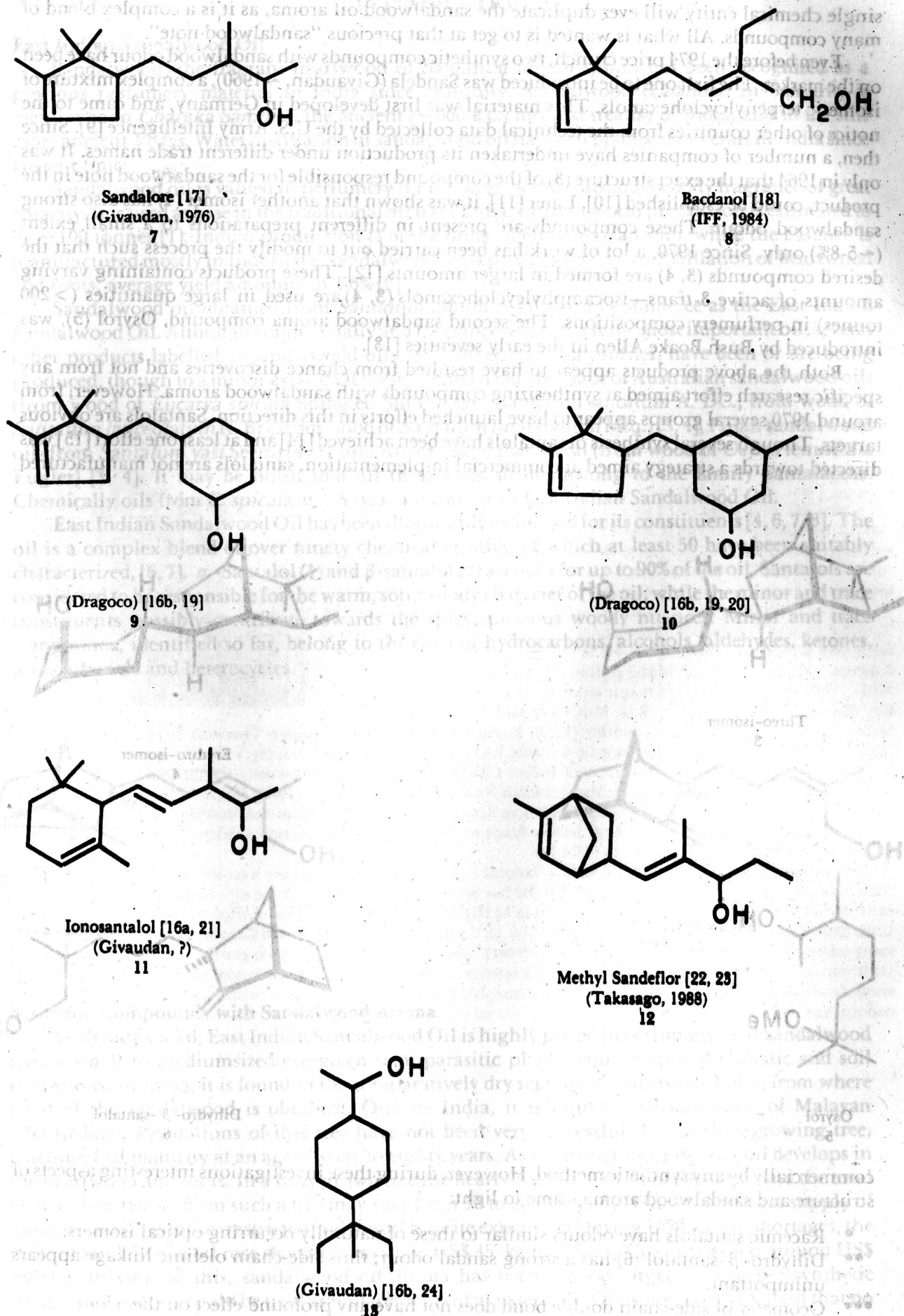
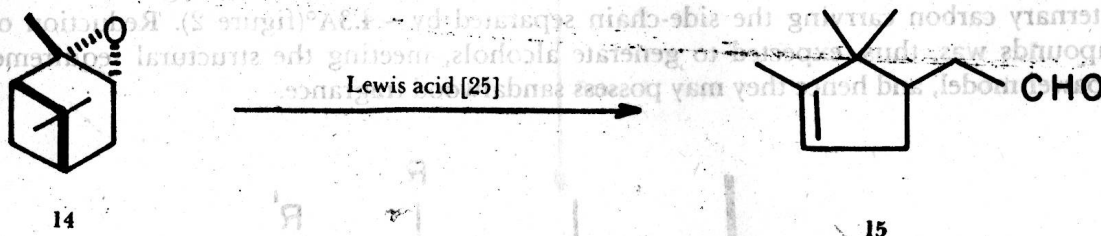


Figure 1. Some new sandalwood aroma synthetics.



Following on some working hypothesis, several workers in different companies have been engaged in synthesizing totally new chemical entities likely to have sandalwood odour [16]. From these investigations, several new sandalwood aroma compounds have emerged. Figure 1 shows some of these. Those, which appear to have been commercialized, are indicated by their trade name, as well as by the name of the company and the apparent year of introduction. It may be noted that  $\alpha$ -compholenic aldehyde (15), readily obtainable by Lewis acid catalysed rearrangement of  $\alpha$ -pinene epoxide (14), has served as a valuable building block for the elaboration of sandalwood aroma compounds. Cyclohexanols 9 and 10 were specifically tailored [16b] after the structures of terpenyl cyclohexanols (3, 4).



#### 5(4 $\rightarrow$ 3) - *abeo*-Caranaldehyde-derived Sandalwood Aroma Compounds

A working model, specially elaborated by Naipawer [16a], of structural requirements for sandalwood aroma has the following main elements:

- The molecule should be a monohydric alcohol.
- The number of carbon atoms should be 12-16.
- There should be a highly substituted or quaternary carbon atom in the molecular framework.
- The distance between the hydroxyl oxygen atom and the quaternary carbon should be approximately  $4A^\circ$  in at least, one of the reasonable conformations.

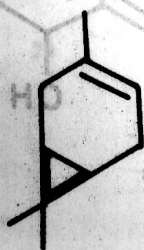
Several exceptions to this model were recognized by the authors. Nonetheless, it has a good predictive value. It was stated by these authors that compounds having a sandalwood odour conform to these features, though not all compounds meeting these requirements have sandalwood odour.

This hypothesis appeared quite appropriate for our projected work, described here.

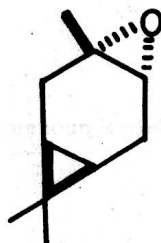
#### 5(4 $\rightarrow$ 3) - *abeo*-Caranaldehyde

In our effort to extend the sophisticated utilization [26] of car-3-ene (16), the chief component of Indian turpentine oil (from *Pinus roxburghii* Sarg. Syn. *P. longifolia* Roxb.), work was undertaken to develop a sandalwood aroma compound from car-3-ene. For reasons already mentioned in the introductory part of this paper, this appeared to be a worthwhile exercise.

Many years ago [27, 25c] we had examined the rearrangement of carene epoxide (17) with  $ZnBr_2$  to obtain, besides other products, some 9-19% yield of 3, 6, 6-trimethylbicyclo [3.1.0]-hexyl-3-carboxaldehyde (18). The stereochemistry depicted in 18 is based on spectral and mechanistic considerations. At around the same time, the reaction was also investigated by three other groups,



16



17

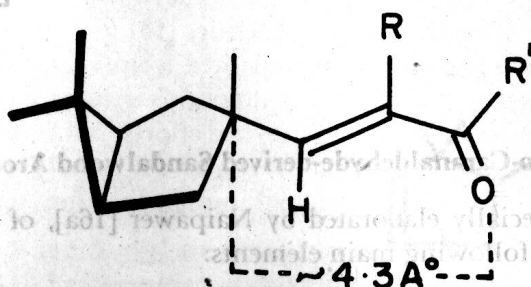


18

[28-30], with similar results. In a later study [25c], we exposed epoxide 17 to active silica gel, when the aldehyde 18 was produced in  $\sim 32\%$  yield. This product (18) which is 5(4 $\rightarrow$ 3)-abeo-caranaldehyde (abeo-caranaldehyde) for short, appeared appropriate as the starting building block for the purpose on hand. Of course, it was necessary to improve the conversion of 17 to 18. This was possible to some extent, when conditions were established for the vapour-phase isomerization of 17 over Zeolites to furnish 18 in  $>60\%$  yield [31].

**1'-(3, 6, 6-Trimethylbicyclo [3.1.0] hex-3-yl)-2'-methylpent-1'-(E)-en-3'-ol, a New Sandalwood Aroma Compound**

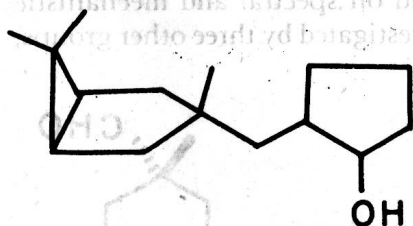
A study of molecular models (Dreiding) showed that products resulting from aldol condensation (followed by dehydration) of aldehyde 18 with different ketones, have the oxygen function and the quaternary carbon carrying the side-chain separated by  $\sim 4.3\text{\AA}$  (figure 2). Reduction of such compounds was, thus, expected to generate alcohols, meeting the structural requirements of Naipawer model, and hence they may possess sandalwood fragrance.



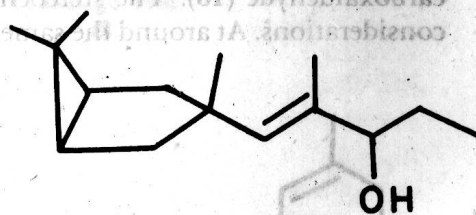
**Figure 2.** Approximate distance between oxygen function and concerned quaternary carbon.

Accordingly aldehyde 18 was condensed with a number of suitable ketones under the usual basic conditions (aq. methanolic NaOH or KOH Soln; 40% methanolic Triton-B) to get unsaturated ketones (19; figure 3) in yield of 60-90%. The ketones used were: acetone, methyl ethyl ketone, diethyl ketone, methyl isopropyl ketone, cyclopentanone and cyclohexanone. In such aldol condensations, the resulting unsaturated ketones invariably have the olefinic bond *trans*-configured (carbonyl function *trans* to the bulky group at the  $\beta$ -carbon atom) [32]. That, indeed, our products has *trans*-stereochemistry (19), was born out from their PMR spectral data.

These compounds were next subjected to reduction both with NaBH<sub>4</sub> and catalytic hydrogenation (hydrogen and Raney Ni), in separate reactions, to furnish unsaturated (21) and saturated alcohols (20) respectively (figure 3). It may be noted that the unsaturated alcohols will be single (but racemic) chemical entities, whereas the saturated alcohols are expected to be mixtures of diastereoisomers for compounds obtained *via* reaction with diethyl ketone, cyclopentanone or cyclohexanone. All the 12 compounds, thus obtained, were gotten evaluated by professional perfumers, in three different companies. Only two compounds (22, 23) emerged as possessing sandalwood fragrance. Of these, 1'-(3, 6, 6-trimethylbicyclo [3.1.0] hex-3-yl)-2'-methylpent-1'(E)-en-3'-ol (23) was adjudged best, having floral sandalwood fragrance of good dryout and retentivity.



22



23

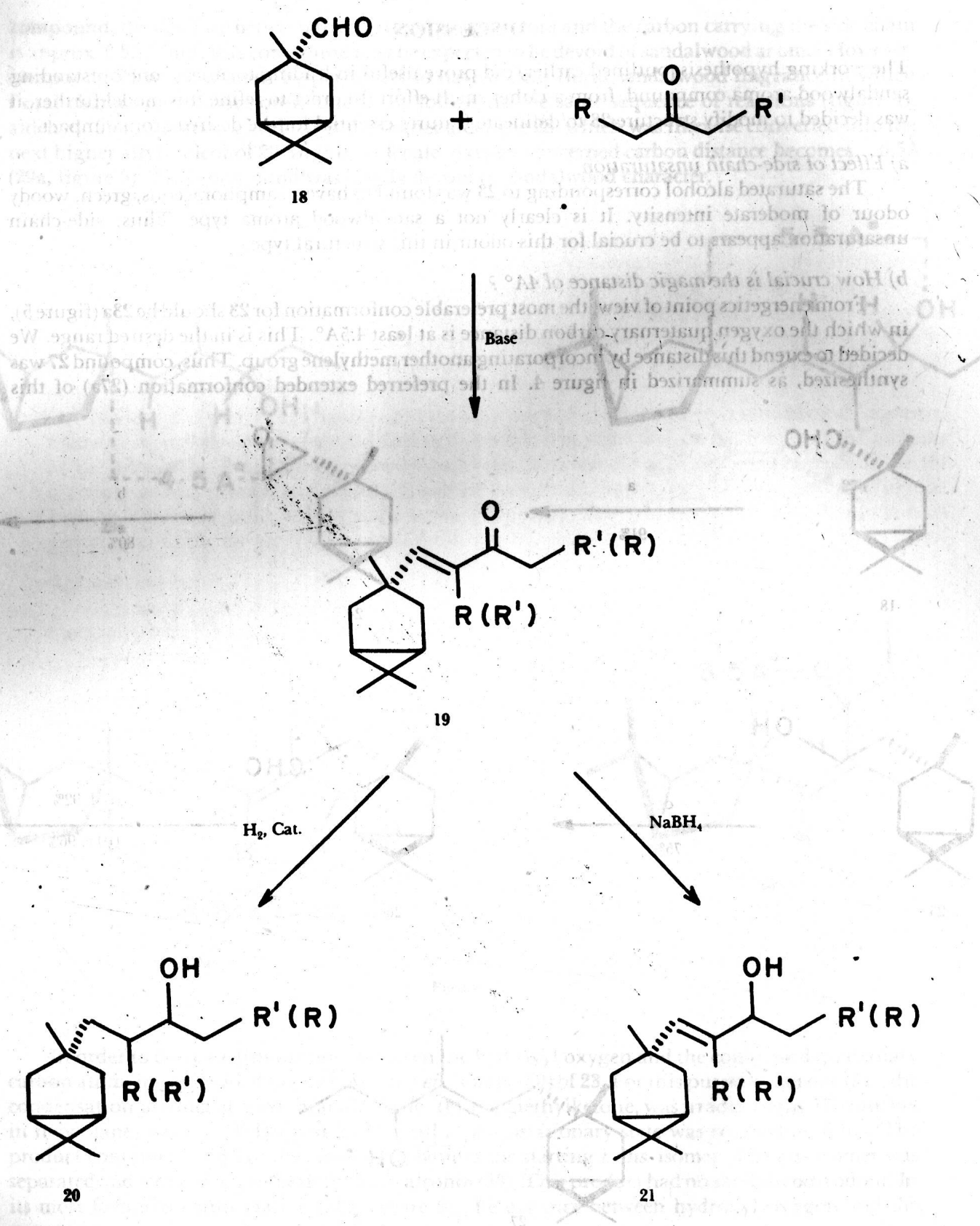


Figure 3. Synthesis of compounds likely to have sandalwood aroma.



## DISCUSSION

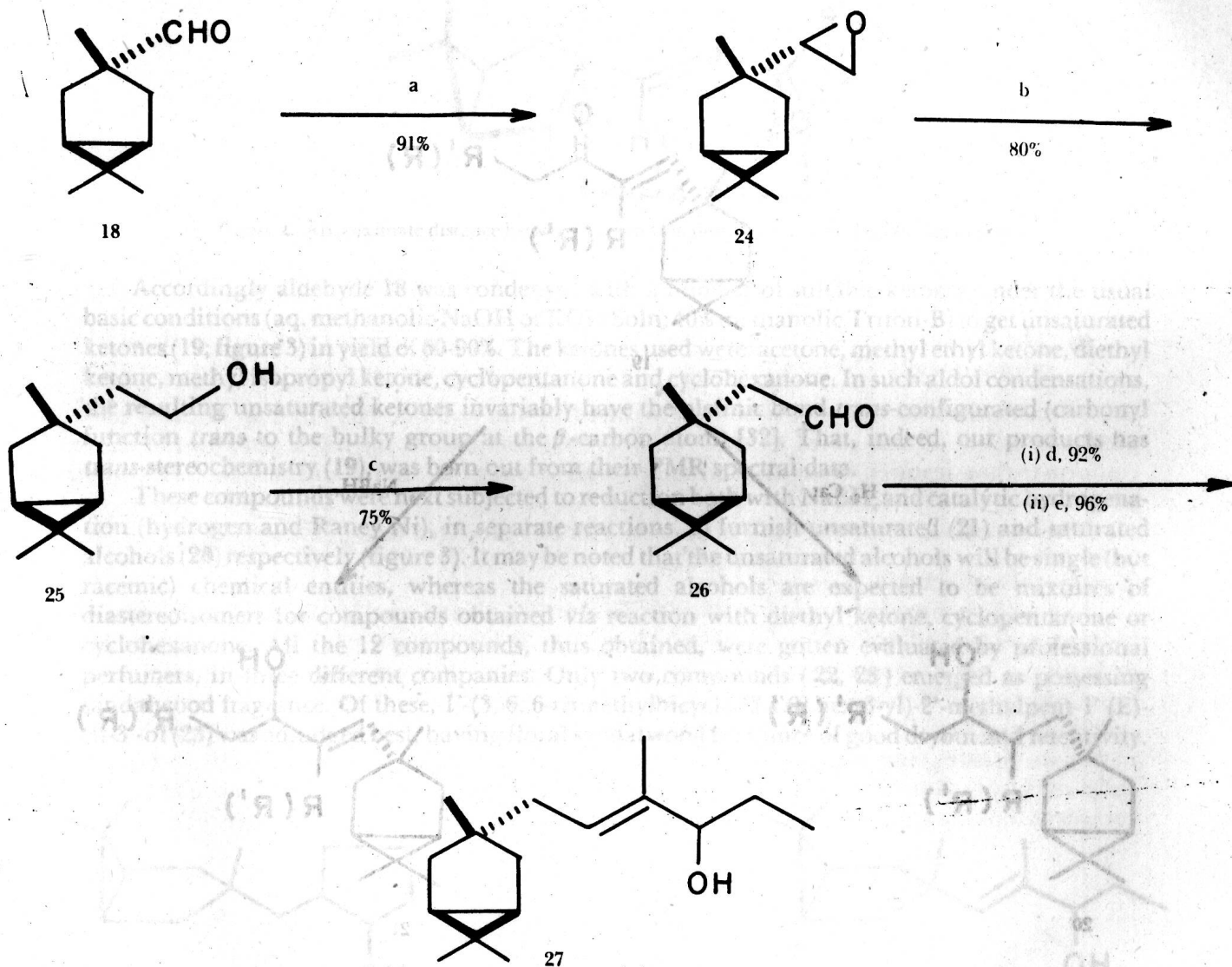
The working hypothesis, outlined earlier, did prove useful in leading to at least one outstanding sandalwood aroma compound, from a rather small effort. In order to refine this model further, it was decided to modify structure **23** to delineate features essential for the desired aroma impact.

a) *Effect of side-chain unsaturation*

The saturated alcohol corresponding to **23** was found to have a camphoraceous, green, woody odour of moderate intensity. It is clearly not a sandalwood aroma type. Thus, side-chain unsaturation appears to be crucial for this odour in this structural type.

b) *How crucial is the magic distance of  $4A^\circ$ ?*

From energetics point of view, the most preferable conformation for **23** should be **23a** (figure 5), in which the oxygen quaternary carbon distance is at least  $4.5A^\circ$ . This is in the desired range. We decided to extend this distance by incorporating another methylene group. Thus, compound **27** was synthesized, as summarized in figure 4. In the preferred extended conformation (**27a**) of this



- Reagents: (a)  $\text{Me}_2\text{S}$ ,  $\text{Me}_2\text{SO}_4$ /NaOH [Ref. 33]  
 (b)  $\text{H}_2$ , Ni (Raney)  
 (c) Pyridinium chlorochromate  
 (d) Pentanone-3/Triton-B  
 (e) LAH

Figure 4. Synthesis of 1'-(3,6,6-trimethyl-trimethyl-bicyclo (3.1.0) hex-3-yl)-3'-methylhex-2' (E)-en-4'-ol.

compound, the distance between the hydroxyl oxygen atom and the carbon carrying the side chain is approx.  $5.5\text{\AA}$ . Thus, this compound may be expected to be devoid of sandalwood aroma. However, compound **24** was evaluated by perfumers to have a clear floral sandalwood fragrance in which floral character was more pronounced. By following the same sequence of reactions (figure 4), aldehyde **26** was homologated to get the higher aldehyde **28**, which was likewise converted into the next higher allylic alcohol **29**. In this molecule, oxygen-concerned carbon distance becomes  $6.5\text{\AA}$  (**29a**, figure 5). This compound was totally devoid of sandalwood character.

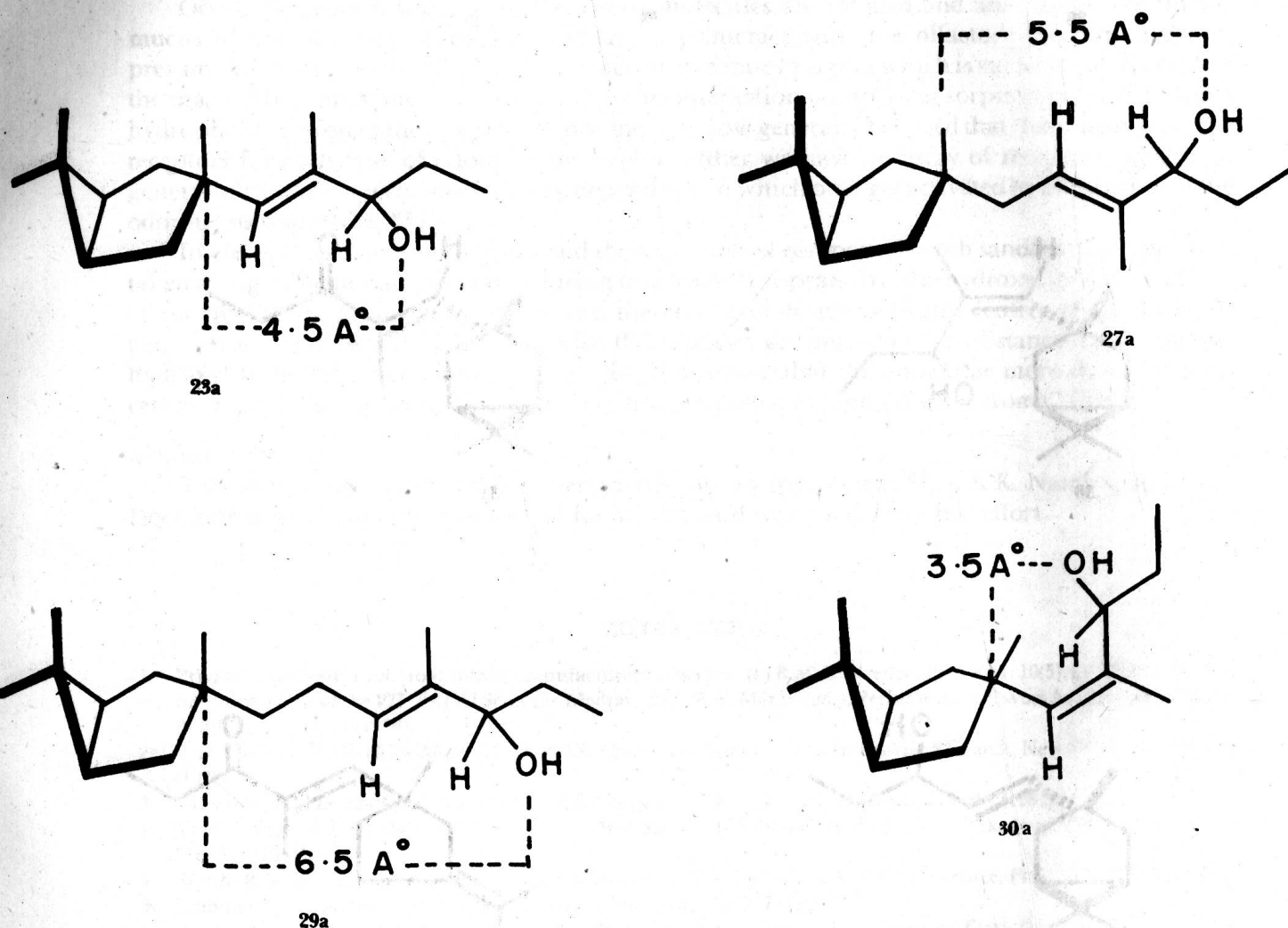


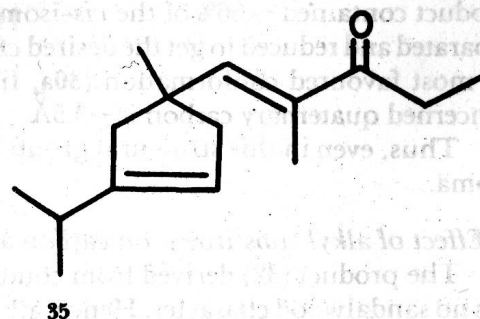
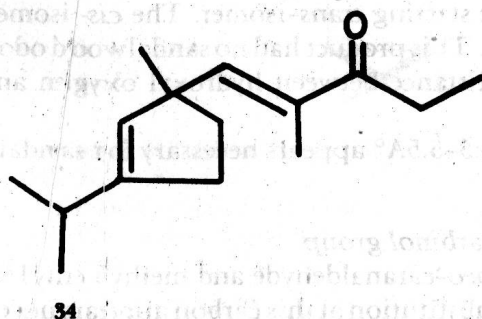
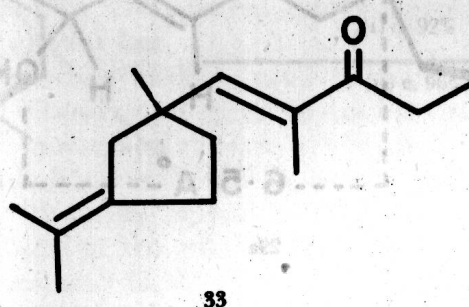
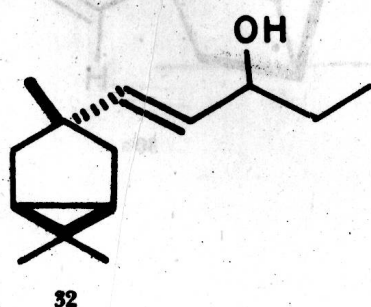
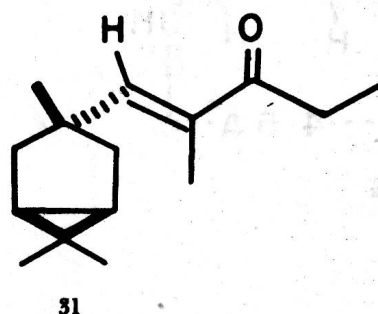
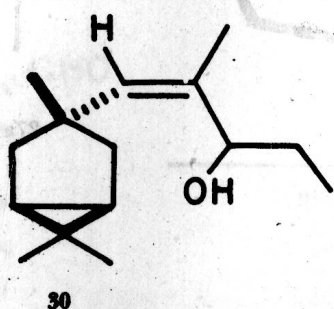
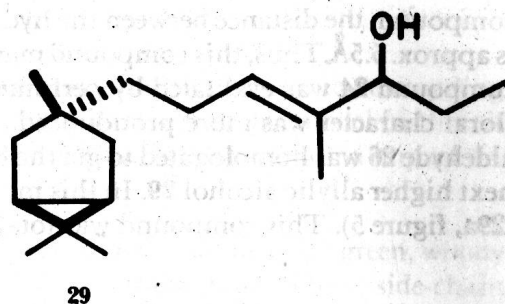
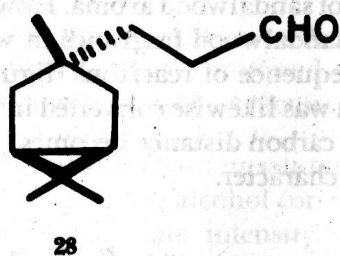
Figure 5.

In order to decrease this distance between the hydroxyl oxygen and the concerned quaternary carbon atom, it was decided to synthesize the *cis* isomer (**30**) of **23**. For this purpose, ketone (**31**), the condensation product of *abeo*-caranaldehyde (**18**) and diethylketone, was irradiated (as 1% solution in *n*-heptane) with a UV lamp at  $25 \pm 5^\circ$  till a photostationary state was reached ( $\sim 6$  hr). The product contained  $\sim 66\%$  of the *cis*-isomer, besides the starting *trans*-isomer. The *cis*-isomer was separated and reduced to get the desired *cis*-alcohol (**30**). This product had no sandalwood odour. In its most favoured conformation (**30a**, figure 5), the distance between hydroxyl oxygen and the concerned quaternary carbon is  $\sim 3.5\text{\AA}$ .

Thus, even in this structural group a distance of  $4.5\text{--}5.5\text{\AA}$  appears necessary for sandalwood aroma.

#### c) Effect of alkyl substituent on carbon atom *a* to the carbinol group

The product (**32**) derived from condensation of *abeo*-caranaldehyde and methyl ethyl ketone has no sandalwood character. Hence, alkyl (methyl?) substitution at this carbon appears necessary.





#### d) Effect of cyclopropane ring

In another series of reactions, the cyclopropane ring in **31** was cleaved ( $\text{HClO}_4$ , aq) and the products (**33-35**) isolated by fractional distillation and preparative GLC. Incidentally, this ketonic mixture has a clean vativer odour. Each of these ketones was separately reduced ( $\text{NaBH}_4$ ) to get the corresponding unsaturated alcohols, which were evaluated by the perfumer. None of these compounds exhibited sandalwood fragrance. Thus in this structure, cyclopropane ring appears essential for sandalwood aroma.

Odour perception begins when odorous molecules are inhaled and are "dissolved" in the mucus of the olfactory epithelium. There they interact with the olfactory receptor neurons, presumably at sites on the cilia. This interaction generates a signal which is suitably interpreted by the brain. There are some data to show that this interaction occurs by adsorption of odorants on a hydrophobic region of the receptor membrane. It is now generally believed that there are no specific receptors for each type of odour modality, but rather we have an array of receptors capable of generating a whole spectrum of odours, depending on which ones get activated by adsorption of the odorous substance [34, 35].

In view of the above mechanism and the structures of compounds with sandalwood fragrance, taken along with the data generated during this work, it appears that the hydroxyl group functions as the anchoring group on receptor and the rest of molecule activates centres responsible for generating "sandalwood fragrance". This then stresses the importance of distance separating the hydroxyl from the other interacting centres. It appears, that there must be more than one such centre responsible for generating signals to be interpreted as "sandalwood aroma" [36].

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