Tetrahedron. Vol. 28, pp. 325 to 331. Pergamon Press 1972. Printed in Great Britain

THE STRUCTURE OF ISOINCENSOLE-OXIDE

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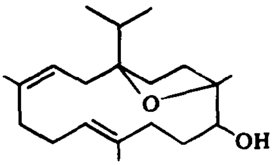
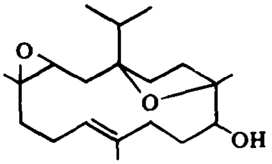
(*Received in the UK* 4 *August* 1971; *Accepted for publication* 13 *September* 1971)

Abstract—A new macrocyclic diterpene, Isoincensole-oxide (I), has been isolated from Frankincense in very small amount. Isoincensole-oxide (I) can be obtained synthetically from Incensole; chemical and physico chemical data support the structure 12-isopropyl-1,5,9-trimethyl-1,12-oxido-5,6-epoxy cyclotetradec-9en-2ol.

INCENSOLE[1] and incensole-oxide[2] are two macrocyclic diterpenes, recently isolated from Frankincense, the resin produced by *Boswellia Carteri.*



incensole (†)



incensole-oxide (†)

From the same source, we succeeded in isolating a third substance, always found along with incensole-oxide in the chromatographic fractions of the neutral part of the resin. This substance, isomeric with incensole-oxide and therefore named isoincensole-oxide (I), shows chromatographic behaviour very similar to that of incensole-oxide‡ and it can be separated from its isomer by chromatography (silica gel) of their benzoyl derivatives.

Isoincensole-oxide (I), obtained by hydrolysis of its oily benzoate, crystallizes from petrol and has m.p. 84–85°: $[\alpha]_D = -13\cdot9°$ ($CHCl_3$). It has a molecular formula $C_{20}H_{34}O_3$, according to the composition and mol. wt. (322) deduced from its mass spectrum.

Spectroscopic properties are very similar to those of incensole-oxide. The IR spectrum shows an OH stretch band (3600 cm^{-1}) and two bands at 1060, 1035 cm^{-1}: since these two bands are present in the ketone (III) (see later) both are due to ethereal oxygen. On the other hand, incensole[1] and incensole-oxide[2] show only one band in this region; although the multiplicity of the "1100 cm^{-1} band" has been observed,[3] at first sight the presence of a second ethereal bridge could be suspected.

As in the case of incensole-oxide, the NMR spectrum of I shows the signals of the isopropyl group and the presence of three Me groups, two of which are bonded to carbon atoms carrying oxygen, while the third is bonded to an unsaturated carbon

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* In alphabetic order.

† Stereochemistry undetermined.

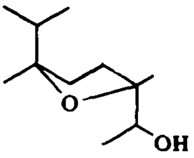
‡ Isoincensole-oxide (I) and incensole-oxide are distinguishable by TLC on $AgNO_3$/silica plates (experimental).

(experimental). The signal at 3·50 δ must be due to the hydrogen of the secondary alcoholic group (since it is shifted downfield (to 5·00 δ) in the corresponding benzoate) and an olefinic proton appears at 6·05 δ.

The main difference in the NMR spectra of two isomeric substances is the position of a low field signal due to one proton: this signal is easily attributable to an epoxide proton in the case of incensole-oxide, whereas in the spectrum of I this signal falls out (4·23 δ)* of the range in which epoxide protons usually lie.[5]

It is possible to confirm the presence of one double bond by catalytic reduction of isoincensole-oxide (I), since I absorbs a molecule of hydrogen, giving a compound $C_{20}H_{36}O_3$ (II), probably an epimeric mixture (unsharp m.p.).

Chromic oxidation of I gives a ketone (III), $C_{20}H_{32}O_3$, m.p. 158–159°, which shows (IR) a carbonyl absorbance at 1716 cm^{-1}: compared with its parent alcohol, it is possible to observe in the NMR spectrum the downfield shift of a signal (from 1·13 to 1·40 δ) due to a Me group bonded to a carbon atom carrying oxygen. These data, by analogy with incensole- and incensole-oxide,[1,2] indicate the presence of the partial structure M.



M

Several attempts to obtain chemical evidence on the nature of the third oxygen atom, which must be ethereal or epoxidic (no absorbance in the OH region in the IR spectrum of ketone III) were unsuccessful. I is recovered unchanged after 20 hr at reflux in THF with LAH: no hydrogenolysis could be observed by catalytic reduction at high pressure and temperature. In the same way, an attempt at reduction with Li/$EtNH_2$—a reagent proved to be in some instances more efficient than LAH for reductive cleavage of epoxides[6] and active also on allyl ethers[7]—was unsuccessful.

A careful examination of the reaction mixture of incensole with *p*-nitroperbenzoic acid allowed us to isolate, through a rather complex procedure, in addition to incensole-oxide,[2] two compounds, one of which was identical in all respects (NMR, IR, m.m.p.) with isoincensole-oxide (I), and the other, (IV) a new isomer $C_{20}H_{34}O_3$, having m.p. 148–150°.

As expected, compound IV shows an NMR spectrum very similar to that of I: however, in this instance (as in the case of incensole-oxide) a signal at 3·40 δ can be easily attributed to an epoxide proton.

In principle, since compounds I and IV are formed by epoxidation of incensole, which contains two double bonds, they can have two of the following three structures (A, B, C).

Moreover, in considering the spectroscopic properties, discussed before, and the lack of the characteristic reactions of the oxirane ring in compound I, two additional

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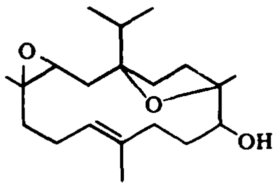
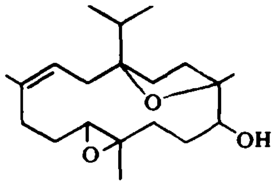
* Allylic protons, bonded to a carbon carrying an ethereal oxygen, fall in this range: the calculated value, obtained with the Shoolery's rules is 4·38.[4]

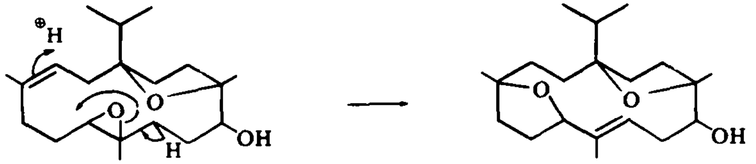


A (differing from incensole-oxide by the stereochemistry of epoxide)

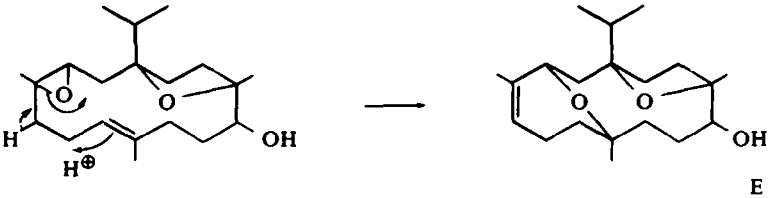


B different stereo-
C chemistry of epoxide

structures (D and E), arising from an intramolecular reaction of the epoxide with the double bond* (catalyzed by acids) could be considered.



D



E

In order to choose between these five hypotetical structures attributable to compounds I and IV, the mass spectral data of the three isomers I, IV and incensole-oxide) have been very indicative.

I and IV showed almost superimposable fragmentation patterns. Very peculiarly the parent peak is also the base peak of the spectrum.† The mass spectrum of incensole-oxide is entirely different: in this instance most of the ion current is held by ions having lower mass (base peak is 143). Hence it is very likely that compound I and IV are stereoisomeric‡ whereas incensole-oxide is a positional isomer; this corresponds to the structures B and C for I and IV reported above.

Evidence on the structure of isoicensole-oxide (I) was obtained in the following way. The benzoyl derivative of I was converted into the corresponding diol (V), $C_{27}H_{40}O_6$, m.p. 170–173°, which shows the expected spectroscopic properties (experimental).

The diol (V), when oxidized with $Pb(CH_3COO)_4$ yields an aldehyde (appropriate signals in the NMR and IR spectra), $C_{27}H_{38}O_6$ (VI), which distills with decomposition.

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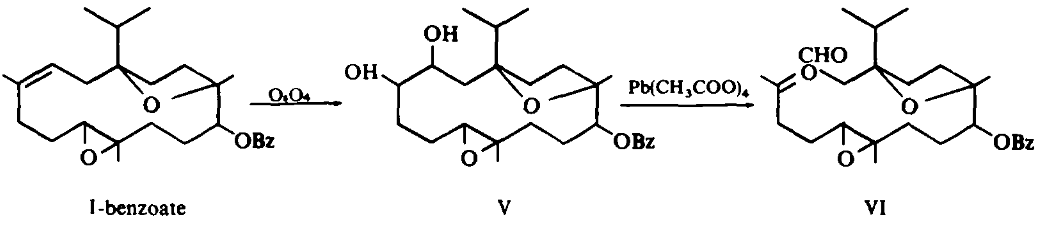
* The acid-catalyzed reaction of epoxy olefins yields alcohols or ethers, *via* an intramolecular attack of the double bond on the incipient carbonium ion.[8] Actually, there are no examples in which the oxygen acts as a nucleophile.

† It is well known that alcohols give very low molecular peaks.[9]

‡ In view of the recent developments in this field,[10] this statement should be accepted with some care.[11]

The epoxide proton appears in the NMR spectrum at $\delta = 3{\cdot}76$ (in benzene); moreover, an AB part of an ABX system centred at 2·52 δ is easily recognizable. These signals are due to the α protons of the aldehyde group: as demonstrated by NMR double resonance.



Irradiating at 2·52 ppm, the multiplet at 10·03 δ becomes a sharp singlet: the signals centred at 2·52 δ become a pure AB pattern by irradiating at 10·03 δ.

The sequence $-\overset{|}{\underset{|}{C}}-CH_2-CHO$ (no hydrogens in β position to the aldehyde group) is coherent only if I possesses the structure B—C.

The unusual chemical behaviour of these diterpenes deserves a further comment. The inertness of the oxirane ring towards the nucleophilic reagents can be explained, if we assume that "the back side" of the epoxide is completely shielded by the residual part of the molecule: a full knowledge on configurations and conformations of these molecules should clarify such unusual properties.

EXPERIMENTAL

IR spectra were recorded with a Perkin-Elmer 257. NMR spectra were recorded with a Varian A-60, unless otherwise stated. Chemical shifts are quoted in δ-values downfield from TMS as internal standard.

M.ps and rotary powers were determined as described before, similarly TLC and preparative chromatography, as well as column chromatography were carried out using the same materials and equipment.[1] Silver nitrate/silica plates were prepared by using an $AgNO_3$ solution (12% w/v) instead of water.

Mass spectra were determined on an AEI MS 12 instrument with a direct inlet system and source temperature of 130°.

Isoincensole-oxide (I)

Chromatography of the neutral fraction of Frankincense. 20 g of neutral fraction[1] were chromatographed over alumina (600 g; Brokmann activity II–III), using C_6H_6 as eluent.

Fractions 1–10 (300 ml each) contained 8·50 g of a mixture each of incensole: fractions 11–17 (200 ml each) contained crystalline incensole-oxide contaminated by an oily material and fractions 18–21 (200 ml each) gave an oil. Crystallization from petroleum-ether 40–70° of the residue of fractions 11–17 gave pure incensole-oxide (0·25 g): combination of the mother-liquor and fractions 18–21 gave an oily residue (0·98 g). This residue on TLC was complex mixture containing two substances (incensole-oxide and isoincensole-oxide (I)) distinguishable only on $AgNO_3$/silica plates (C_6H_6–ether 8:2 as eluent).

Purification of the incensole-oxide/isoincensole-oxide (I) *mixture.* 5·80 g of the oily residue obtained as described before were chromatographed on silica (350 g), using C_6H_6–ether as eluent and increasing the amount of ether from 1% to 3%. The first set of fractions (30 × 200 ml), containing impurities, were discarded. The residue of the subsequent 15 fractions (5% ether as eluent) dissolved in petroleum-ether 40–70°, and left overnight at 0° gave a new crop of incensole-oxide. The mother-liquor, evaporated *in vacuo* gave 1·65 g of a mixture of incensole-oxide and isoincensole-oxide (I) (each of I) which shows only one spot on TLC (two spots on $AgNO_3$/silica TLC).

Incensole-oxide and isoincensole-oxide benzoates. To the mixture reach of isoincensole-oxide (I) (1·05 g), dissolved in dry pyridine (7 ml), BzCl (0·7 ml), was added and the solution left at room temp. for 24 hr.

After the usual work-up, the crude product (1·25 g) showed two spots on TLC (hexane-ether 8:2). The mixture was chromatographed on silica (75 g), using 3% ether in hexane as eluent. After a pre-run of six fractions (50 ml each), the subsequent set of 20 fractions contained 0·74 g of benzoate of I.

Using 15% ether, four fractions containing incensole-oxide benzoate (0·16 g) were eluted. The isoincensole-oxide (I) benzoate, purified by short-path distillation, is a colourless viscous oil, b.p. 190–195°/0·6 mm. NMR (CCl_4): 0·88 (3H, d, $J = 6{\cdot}5$ c/s): 1·02 (3H, d, $J = 6{\cdot}5$ c/s) (i-propyl): 1·02 (3H) $(\mathrm{CH_3}{-}\underset{\diagdown O \diagup}{\overset{|}{\mathrm{C}}{-}\overset{|}{\mathrm{C}}}{-})$: 1·27 (3H) $(\mathrm{CH_3}{-}\underset{|}{\overset{|}{\mathrm{C}}}{-}\mathrm{O}{-})$: 1·64 (3H) $(\mathrm{CH_3}{-}\overset{|}{\mathrm{C}}{=}\overset{|}{\mathrm{C}}{-})$: 4·17 (1H, broad signal) $(\underset{\diagdown O \diagup}{{-}\mathrm{CH}{-}\overset{|}{\mathrm{C}}}{-})$: 5·00 (1H, broad signal) $({-}\overset{|}{\mathrm{C}}\mathrm{H}{-}\mathrm{OBz})$: 5·84 (1H, broad signal) $({-}\mathrm{CH}{=}\overset{|}{\mathrm{C}}{-})$: 7·45 (m): 8·00 (m) (—Ph). (Found: C, 75·96: H, 8·64. $C_{27}H_{38}O_4$ requires C, 76·02: H, 8·98%).

Isoincensole-oxide (I). Isoincensole-oxide (I) benzoate (0·74 g) was dissolved in a methanolic soln (5 ml) of KOH (7% w/v) and allowed to stand at room temp. for 24 hr. The mixture, worked up in the usual way, gave a residue (0·5 g) of crude I, which crystallized from petroleum-ether 40–70° as prisms. M.p. 84–85°, $[\alpha]_D = -13{\cdot}9°$ (C=O, 760). $\nu_{max}^{(CCl_4)}$ (cm^{-1}): 3620 (—OH): 1120, 1060, 1030 (C—O). NMR ($CDCl_3$): 0·87 (3H, d, $J = 7$ c/s): 1·02 (3H, d, $J = 7$ c/s) (i-propyl): 1·07 (3H) $(\mathrm{CH_3}{-}\underset{\diagdown O \diagup}{\overset{|}{\mathrm{C}}{-}\overset{|}{\mathrm{C}}}{-})$: 1·13 (3H) $(\mathrm{CH_3}{-}\underset{|}{\overset{|}{\mathrm{C}}}{-}\mathrm{O})$: 1·60 (3H) $(\mathrm{CH_3}{-}\overset{|}{\mathrm{C}}{=}\overset{|}{\mathrm{C}}{-})$: 3·50 (1H, broad signal) $({-}\overset{|}{\mathrm{C}}\underline{\mathrm{H}}{-}\mathrm{OH})$: 4·23 (1H, broad signal) $(\underset{\diagdown O \diagup}{{-}\mathrm{CH}{-}\overset{|}{\mathrm{C}}}{-})$: 6·05 (1H, broad signal) $({-}\mathrm{CH}{=}\overset{|}{\mathrm{C}}{-})$. (Found: C, 74·65: H, 11·39. $C_{20}H_{34}O_3$ requires C, 74·49: H, 10·63%).

Dihydro-isoincensole-oxide (II)

Isoincensole-oxide (I) (0·25 g), dissolved in EtOH 95° (25 ml) was hydrogenated at room temp. and atm. press in the presence of Pd/C (10%, 0·10 g), previously saturated with H_2. The hydrogenation stopped when one mole of H_2 had been absorbed. After filtration and solvent removal, the dihydro-isoincensole-oxide (II) was crystallized from petroleum-ether 40–70°, m.p. 143–144° (sintering from 130°). $\nu_{max}^{(CCl_4)}$ (cm^{-1}): 3620 (—OH): 1035, 1050 (C—O): NMR: no signals in the olefinic protons region. (Found: C, 74·26: H, 11·39. $C_{20}H_{36}O_3$ requires C, 74·02: H, 11·18%).

Insoincensole-oxide ketone (III)

Isoincensole-oxide (I), (0·10 g), dissolved in pyrydine (5 ml), was added to a CrO_3-pyridine complex, prepared from pyridine (5 ml), CrO_3 (0·50 g) and water (0·1 ml). The soln was allowed to stand for 50 hr at 25°. After the usual work up, the residue was purified on prep. TLC (C_6H_6-ether 7:3 as eluent). The ketone (III) crystallized from MeOH, m.p. 158–159°. $\nu_{max}^{(CCl_4)}$ (cm^{-1}): 1716 (C=O); 1070, 1034 (C—O): NMR ($CDCl_3$): 0·86 (3H, d, $J = 7$ c/s): 0·97 (3H, d, $J = 7$ c/s) (i-propyl): 1·08 (3H) $(\mathrm{CH_3}{-}\underset{\diagdown O \diagup}{\overset{|}{\mathrm{C}}{-}\overset{|}{\mathrm{C}}})$: 1·40 (3H) $(\mathrm{CH_3}{-}\underset{|}{\overset{|}{\mathrm{C}}}{-}\mathrm{O})$ · 1·61 (3H) $(\mathrm{CH_3}{-}\overset{|}{\mathrm{C}}{=}\overset{|}{\mathrm{C}}{-})$: 4·32 (1H, broad signal) $(\underset{\diagdown O \diagup}{{-}\mathrm{CH}{-}\overset{|}{\mathrm{C}}}{-})$: 6·10 (1H, broad signal) $(\overset{|}{\mathrm{C}}\mathrm{H}{=}\overset{|}{\mathrm{C}}{-})$. (Found: C, 74·86: H, 9·96. $C_{20}H_{32}O_3$ requires C, 74·96: H, 10·06%).

Isoincensole-oxide (I) *and isomer* (IV) *by oxidation of incensole*

Reaction of incensole with p-nitroperbenzoic acid. To incensole (8·50 g) dissolved in $CHCl_3$ (200 ml): commercial *p*-nitroperbenzoic acid (5 g) was added with stirring at room temp. over 2 hr. The obtained suspension was shaken with sat. $NaHCO_3$aq and the organic phase washed with water. After solvent removal, crystalline incensole-oxide was filtered off by adding petroleum-ether 40–70°. The mother-liquor residue (6·5 g) was a complex mixture on TLC: incensole (the starting material), incensole-oxide, isoincensole-oxide (I) and isomer (IV) were detectable. Other more polar substances were present. This mixture was fractionated on silica (200 g), using hexane–EtOAc 8:2 as eluent. Pure incensole (0·68 g) was collected in the first four fractions (150 ml each). Fractions 5–13 contained 2 g of a residue which was a mixture of incensole-oxide, isoincensole-oxide (I) and isomer (IV) (having very similar R_f values on TLC). Subsequent fractions (containing more polar compounds) were discarded. By addition of petrol 40–70° to the residue of fractions 5–13, incensole-oxide (0·40 g) was collected: the residue of mother-liquor weighed 1·6 g.

Synthetic isoincensole-oxide (I). The residue of mother-liquor (1·60 g), dissolved in pyridine (11 ml) was treated with BzCl (1·1 ml) and left at room temp. for 24 hr. The mixture, worked up by the usual procedure was analyzed by TLC (hexane–ether 8:2) and showed the presence of: isoincensole-oxide (I) benzoate, incensole-oxide benzoate and (IV) benzoate. By chromatography on silica column (ratio 1:60,3% ether in hexane as eluent) pure isoincensole-oxide (I) benzoate (0·70 g) was collected in several fractions (40 ml each). Subsequent elution gave a mixture (0·49 g) of Incensole-oxide benzoate and IV benzoate. Isoincensole-oxide (I) benzoate, hydrolized with a 7% methanolic solution (w/v), gave almost pure I which recrystallized from petrol 40–70° and showed the same m.p. (undepressed m.m.p.) and spectroscopic properties as the natural I.

Isomer (IV). The incensole-oxide benzoate/isomer IV benzoate mixture (0·49 g) was submitted to hydrolysis by dissolving in a 7% (w/v) methanolic soln of KOH and standing at room temp. for 48 hr. The usual work-up gave a mixture (0·30 g) of incensole-oxide and isomer (IV). Chromatography on silica column (ratio 1:60, C_6H_6–ether 95:5 as eluent) yielded 5 fractions (100 ml each) containing the crude isomer (IV) (0·15 g) which, recrystallized from light petroleum 80–100°, m.p. 148–150: $\nu_{max}^{(CCl_4)}$ (cm^{-1}): 3620 (—OH): 1080, 1060, 1035 (C—O): NMR ($CDCl_3$): 0·87 (3H, d, J = 7 c/s): 1·00 (3H, d, J = 7 c/s) (i-propyl): 1·05 (3H) $(\mathrm{CH_3{-}\underset{\diagdown\,O\,\diagup}{\overset{|}{C}{-}\overset{|}{C}}{-}})$: 1·12 (3H) $(\mathrm{CH_3{-}\overset{|}{\underset{|}{C}}{-}O})$: 1·57 (3H) $(\mathrm{CH_3{-}\overset{|}{C}{=}\overset{|}{C}{-}})$: 3·40 (1H, broad signal) $(\mathrm{{-}CH{-}\overset{|}{C}{-}})$ with $\underset{\diagdown\,O\,\diagup}{}$: 3·78 (1H, broad signal) $(\mathrm{{-}\overset{|}{C}\underline{H}{-}OH})$: 5·32 (1H, broad signal) $(\mathrm{{-}CH{=}\overset{|}{C}{-}})$.

(Found: C, 74·31: H, 10·27. $C_{20}H_{34}O_3$ requires C, 74·49: H, 10·63%).

Isoincensole-oxide benzoate diole (V)

To a pyridine (4 ml) MeOH (3 drops) soln of I benzoate (0·42 g), OsO_4 (0·25 g) was added. The mixture was stored for one night at room temp. After dilution with water (5 ml) and dioxane (30 ml) H_2S was bubbled for three hr. The suspension obtained was centrifuged: the recovered soln decolourized with charcoal and concentrated *in vacuo*. The residue was diluted with water and ether extracted. The ethereal phase was shaken with HCl 2N, washed with water and dried. A residue (0·4 g) was obtained after solvent removal. TLC analysis (C_6H_6–EtOAc 1:1) showed two products by prep. TLC on silica (C_6H_6–EtOAc 7:3), the product $<R_f$ (diol V) (0·23 g) was isolated. V crystallized from hexane–C_6H_6, m.p. 170–175°; $\nu_{max}^{(CCl_4)}$ (cm^{-1}): 3500 (—OH): 1720 (C═O): 1372, 1350, 1318 (CH$_3$—C—CH$_3$): NMR ($CDCl_3$): 0·92 (3H, d, J = 7 c/s): 0·95 (3H, d, J = 7 c/s) (i-propyl): 1·09 (3H) $(\mathrm{CH_3{-}\underset{\diagdown\,O\,\diagup}{\overset{|}{C}{-}\overset{|}{C}}{-}})$: 1·11 (3H) $(\mathrm{CH_3{-}\overset{|}{\underset{|}{C}}{-}O})$: 1·30 (3H) $(\mathrm{CH_3{-}\overset{|}{\underset{|}{C}}{-}O})$: 4·14 (1H, broad signal) $(\mathrm{CH{-}\overset{|}{\underset{|}{C}}{-}O})$: 4·64 (1H, broad signal) $(\mathrm{{-}\overset{|}{C}H{-}OB_z})$. (Found: C, 70·31: H, 8·68. $C_{27}H_{40}O_6$ requires C, 70·40: H, 8·75%).

Isoincensole-oxide benzoate diol (*V*) *oxidation with* Pb $(CH_3COO)_4$

Pb(OAc)$_4$ (0·28 g) (containing 10% AcOH) was added to diol V (0·20 g), dissolved in C_6H_6 (7 ml) and MeOH (8 drops).

This soln was left for 6 hr at room temp.: diluted with ether, shaked with HCl 2N and sat. NaHCO$_3$aq, washed with water and dried. Solvent removal yielded an oily residue (0·2 g) which proved on TLC (C_6H_6–ether 8:2) to be a mixture, the most abundant showing a characteristic pink-yellow colour. Separation on prep. TLC of this substance gave pure aldehyde VI as an oil (0·08 g) which decomposed on distillation. $\nu_{max}^{(CCl_4)}$ (cm^{-1}): 1720 (C═O): 1710 (C═O): 1370, 1315 (CH$_3$—C—CH$_3$): 1180, 1110, 1030 (C—O); NMR

(CCl_4): 0·88 (3H, d, $J = 7$ c/s): 0·89 (3H, d, $J = 7$ c/s) (i-propyl): 1·07 (3H) ($\mathrm{CH_3{-}}\underset{\diagdown\mathrm{O}\diagup}{\mathrm{\overset{|}{C}{-}\overset{|}{C}}}\mathrm{{-}}$): 1·25 (3H) ($\mathrm{CH_3{-}\underset{|}{\overset{|}{C}}{-}O}$); 2·00 (3H) ($\mathrm{CH_3{-}\overset{|}{C}{=}O}$); 3·36 (2H) ($\mathrm{{-}\underset{|}{\overset{|}{C}}{-}\underline{CH_2}{-}CHO}$); 3·98 (1H, broad signal) ($\mathrm{{-}}\underset{\diagdown\mathrm{O}\diagup}{\mathrm{CH{-}\overset{|}{C}}}\mathrm{{-}}$): 5·02 (1H, broad signal) ($\mathrm{{-}\overset{|}{C}H{-}OB_z}$). At 100 MHz in benzene: 0·75 (3H, d, $J = 7$ c/s): 0·84 (3H, d, $J = 7$ c/s) (i-propyl): 0·97 (3H) ($\mathrm{CH_3{-}}\underset{\diagdown\mathrm{O}\diagup}{\mathrm{\overset{|}{C}{-}\overset{|}{C}}}\mathrm{{-}}$): 1·14 (3H) ($\mathrm{CH_3{-}\underset{|}{\overset{|}{C}}{-}O}$): 1·66 (3H) ($\mathrm{CH_3{-}\overset{|}{C}{=}O}$); 2·52 (2H) ($\mathrm{{-}\underset{|}{\overset{|}{C}}{-}\underline{CH_2}{-}CHO}$); 3·77 (1H, broad signal) ($\mathrm{{-}}\underset{\diagdown\mathrm{O}\diagup}{\mathrm{CH{-}\overset{|}{C}}}\mathrm{{-}}$); 5·20 (1H) ($\mathrm{{-}\overset{|}{C}H{-}OB_z}$); 7·18: 8·10 ($\mathrm{{-}C_6H_5}$); 10·03 (1H, (—CHO). (Found: C, 70·78; H, 8·21. $C_{27}H_{38}O_6$ requires C, 70·71; H, 8·35%).

Acknowledgments—We are grateful to Dr. C. Iavarone (this Institute) for 60 MHz spectra and to Prof. C. G. Casinovi (Instituto Superiore di Sanità-Roma) for 100 MHz spectrum. We thank Italian CNR for financial support.

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