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SYNTHESIS OF β-ELEMENE AND ELEMOL*

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Though the crystalline monocyclic sesquiterpene alcohol, elemol (I), has been the subject of investigation for a number of years its gross structure and absolute configuration were established comparatively recently(1,2). A number of compounds related to elemane have been synthesized(3,4,5). We now report the synthesis of β-elemene (II), a naturally occurring sesquiterpene(6) and elemol (I).

The total synthesis of the (+) acid (III) has been carried out(7). It can be prepared conveniently from (-)-α-santonin(4). The conversion of the (+) acid (III) to elemane-2,3-12-triol (IV) has already been reported(5). The tritosylate (V), prepared by the tosylation of triol (IV), on heating with sodium iodide in acetone furnished the iodocompound (VI) which underwent elimination reaction, on treatment with potassium-t-butoxide in dimethylsulphoxide(8) to furnish β-elemene (II) identified by comparison (IR spectrum and GLC behaviour) with an authentic sample.

Treatment of β-elemene with molar proportion of perbenzoic solution in chloroform at 0-10° for 20 hours followed by lithium aluminium hydride reduction of the epoxidation product furnished a mixture of starting material and the alcohols (VII) and (I). The mixture of alcohols, which was separated from β-elemene by column chromatography on alumina, showed two peaks with retention times of 12.7 and 16.5 minutes

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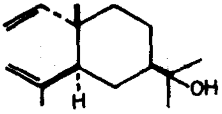
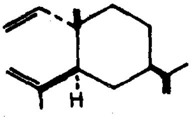
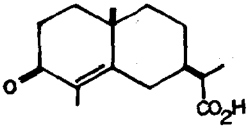


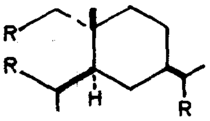
I



II



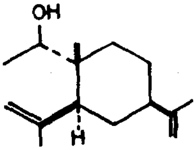
III



IV, R = $\mathrm{CH_2OH}$

V, R = $\mathrm{CH_2OTs}$

VI, R = $\mathrm{CH_2I}$



VII

on GLC analysis*. The component with retention time of 12.7 minutes is probably the secondary alcohol (VII) since the mixture of alcohols after Jones oxidation(9) followed by GLC analysis failed to exhibit this peak. The mixture of alcohols (VII) and (I) was oxidised with Jones reagent(9) and chromatographed on grade II alumina using initially pet. ether and subsequently ether as eluents. The ether eluted fraction which is composed predominantly of elemol (I) on the basis of its IR spectrum and GLC behaviour readily furnished elemol-3,5-dinitrobenzoate identified by direct comparison (m.p., mixed m.p., and IR spectrum) with an authentic sample.

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* GLC examination was carried out on a Griffin and George MK II VPC apparatus equipped with a polyester column (length of the column 6 ft, diameter, 6 mm), at 180° with a gas flow of hydrogen of 1.1 ml/sec.

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