Isolation of Lupeol group of Triterpenes from *Dillenia indica* Linn. and *Diospyros perigrina*

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IN connection with our work on the reactions of triterpenes we needed large quantity of betulinic acid. The isolation of betulinic acid from the bark of *Dillenia indica*[1] and *Diospyros perigrina*[2] was reported. Since the other parts of *D. indica* were not examined earlier, we examined the fruit and heart-wood with a view that these parts of the plant also would yield betulinic acid.

The ether extracts of the powdered heart-wood and fruit of *D. indica* were fractionated into acidic (0.05%) and neutral (0.02%) fractions with 5% NaOH. The acidic fractions from both the parts were found to be identical (m.p. 298°–300°; $(\alpha)_D^{28}+5°$: crystalise from methanol as colourless needles) and identified as betulinic acid by comparison of the compound and its methyl ester (CH_2N_2 : m.p. 220°–222°) with betuilinic acid and methyl betulinate (TLC and IR). The neutral fraction from the wood was purified by chromatography over alumina and identified as β-sitosterol (m.p. 136°–37°; $(\alpha)_D^{28}-36°$) by comparison with authentic sample. The neutral fraction from the fruit (m.p. 250°–52°; $(\alpha)_D^{28}+19°$: crystalise from methanol as colourless needles) was identified as betulin by comparison of the compound and its diacetate (Ac_2O-pyridine : m.p. 220°) with betulin and betulin diacetate (TLC and IR).

The acidic fraction of the ether extract of the bark of *Diospyros perigrina* furnished the reported betulinic acid[2] in an yield of 3% and nothing was reported about the neutral fraction. In the present investigation the neutral fraction (0.03%) showed 2 spots on TLC. So it was absorbed over alumina column and eluted with benzene and benzene-chloroform (5 : 1). The benzene eluate furnished lupeol (m.p. 210°–12°; $(\alpha)_D^{28}+24°$: crystalise from methanol : compared with authentic sample). The benzene chloroform eluate gave betulin.

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Studies on an Acid-Induced Cyclization Product of Ostruthin

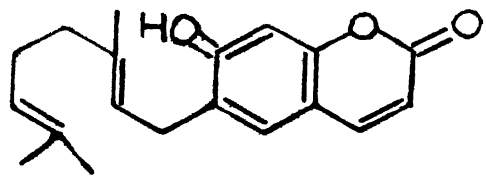
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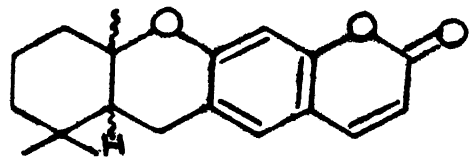
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THE isolation of ostruthin (I) in a very high yield from the root and stem bark of *Atalantia missionis* Oliver was reported by us earlier[1]. The present paper deals with the structure of an acid-induced cyclization product of ostruthin.

Ostruthin (I, 350 mg) was dissolved in glacial acetic acid and then two drops of conc. H_2SO_4 was added. It was then heated on a steam bath for 15 min. On working up the product followed by purification by column chromatography over silica gel column a colourless crystalline compound, $C_{19}H_{22}O_3$, m.p. 175°–78° (M^+ 298°), was obtained which was found to be homogeneous in TLC. The same product was also obtained on treatment of ostruthin with Ac_2O/H_2SO_4. Unlike Ostruthin it did not give any positive ferric chloride colour test. Its IR spectrum in Nujol mull showed a band at 1720 cm^{-1} for the coumarinic lactone group but did not show any band for a free hydroxyl group and this indicated that the above product is most probably an acid-induced cyclization product (II).



(I)



(II)

The possible genesis of some of the important fragment ions in the mass spectrum of (II) is shown below.

The NMR spectrum of (II) (60 MHz, $CDCl_3$) showed sharp singlets at 0.92δ and 1.05δ for the six protons of two tertiary methyl groups ($\equiv C\text{-}CH_3$). The sharp singlet at 1.26δ (3H) is due to the tertiary methyl group attached to the carbon linked to the oxygen atom of the pyran ring. The signals at

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** The = OH in Fig. (I) to be treated as — OH.