CHAPTER-III

Reinvestigation on the Neutral Part of the Benzene Extract of the Fern Oleandra nerifolia: Isolation of a New Triterpene 29-Ethoxyhopane, $C_{32}H_{56}O$, along with Filicene, Nerifoliol and β-Sitosterol.

Section A: Extraction:

Dried and powdered rhizomes of the fern Oleandra nerifolia (syn. Oleandra pistillaris) was extracted with benzene in a soxhlet apparatus for 20 hours. The gummy solid residue obtained after the evaporation of benzene was taken up in ether. The ether solution was washed with 10% aqueous sodium hydroxide solution and then with water till neutral and dried over anhydrous sodium Sulphate. Removal of ether gave a gummy residue which was chromatographed as diseussed in Section B.

Section B: Chromatography of the neutral part.

Table-1

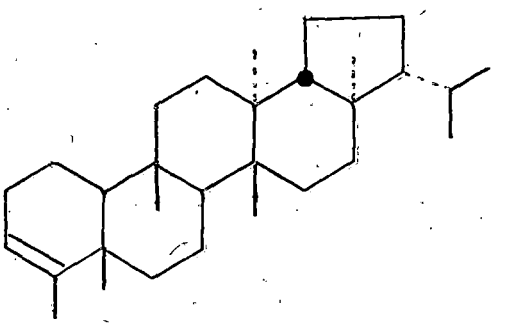
Fraction No.	Eluent	Eluate	Melting point of the residue in °C
1.	Petrol	Solid with oil	-
2.	Petrol: Benzene (3:2)	Solid (0.5 gm)	236-40°
3.	Petrol: Benzene (2:3)	Solid (1.0 gm)	130-34°

Further elution with more polar solvents did not yield any solid material.

Section C: Examination of Fractions 1-3:

Fraction No. 1: Isolation of a New Triterpene, 29-Ethoxyhopane, $C_{32}H_{56}O$ and Filicene:

The fraction No. 1 (Table-1) on careful rechromatography over activated alumina afforded a waxy solid. The waxy solid on crystallisation from a mixture of chloroform and acetone furnished a solid $C_{30}H_{50}$, m.p. 226-28°, $(\alpha)_D 50°$, which was found to be identical (mmp, IR and TLC) with an authentic specimen of filicene (22).



(22)

The mother liquor from the crystallisation of filicene was found by TLC on 12% silver nitrate impregnated silica gel plate to be a mixture of three components. Fractional crystallisation of the residual solid from the mother liquor from a mixture

of chloroform and methanol (3:1) furnished a solid, m.p. 179-80°, $(\alpha)_D 27.16^\circ$. This compound was found to be a novel triterpene, $C_{32}H_{56}O$, namely, 29-ethoxyhopane. The chemistry and structure elucidation of this compound has been described in Chapter-IV.

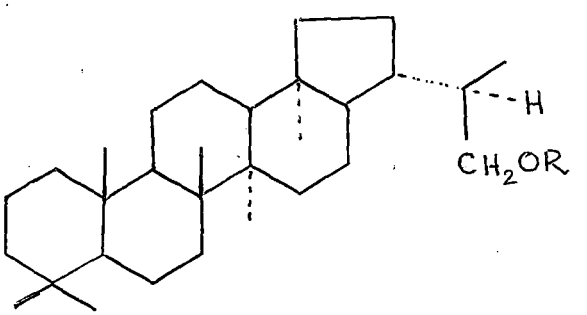
The residue from the final mother liquor after the separation of the above novel triterpene was rechromatographed over alumina impregnated with 20% silver nitrate. Elution of the column with petrol first gave a solid, which on crystallisation from a mixture of chloroform and methanol afforded a solid, m.p. 182-83°. The elemental analysis of this solid corresponded to the molecular formula $C_{30}H_{50}O$. Further elution of the column with petrol afforded another solid, which on crystallisation from a mixture of chloroform and methanol gave another solid, m.p. 163-64°. Elemental analysis suggested the molecular formula as $C_{30}H_{50}$. The structure elucidations of these two compounds were not possible because of their very poor yield. Further work is in progress to isolate them in quantity to enable us to investigate their structures.

Fraction No. 2: Isolation and Identification of Nerifoliol:

Rechromatography of the fraction No. 2 (Table-1) over a column of active alumina and elution with a mixture of petrol and benzene (2:3) gave an alcohol, $C_{30}H_{52}O$, m.p. 242-44°, $(\alpha)_D 35^\circ$, (M^+428), IR ν_{max}^{nujol} 3320 cm^{-1}.

On acetylation, the alcohol furnished an acetate, $C_{32}H_{54}O_2$, m.p. 195-96°, $(\alpha)_D^{20°}$, IR ν_{max}^{nujol} 1730, 1225 cm^{-1}.

The physical and chemical data of the alcohol and its acetate showed that they were identical with nerifoliol (hopan-29-ol) (17a), isolated by Pandey and Mitra[6] from the same plant Oleandra nerifolia, and its acetate (17b) respectively.



(17a) R = H

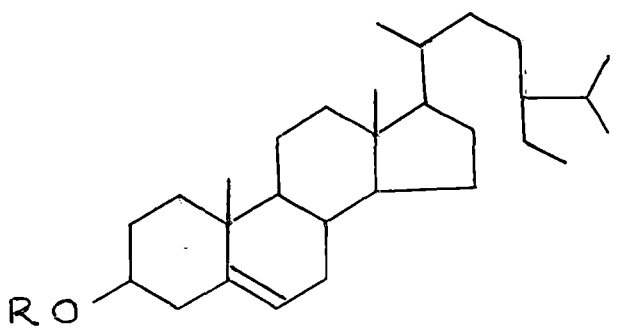
(17b) R = $COCH_3$

Fraction No. 3: Isolation and Identification of β-Sitosterol:

Fraction No. 3 (Table-1) on rechromatography over a column of active alumina and elution with a mixture of petrol and benzene (1:4) gave a solid, which on crystallisation from a mixture of chloroform and methanol furnished fine needle shaped crystals of an alcohol, $C_{29}H_{50}O$, m.p. 136-37°, $(\alpha)_D$-32°.

On acetylation it gave an acetate, $C_{31}H_{52}O_2$, m.p. 127-29°, $(\alpha)_D$-40°.

The alcohol and its acetate were identified as β-sitosterol (23a) and β-sitosteryl acetate (23b) respectively by direct comparison (m.m.p., IR and Co-TLC) with their respective authentic specimens.



(23a) R = H

(23b) R = $COCH_3$.