

## CHAPTER II

### Reinvestigation on the benzene extract of the bark of *Sapium baccatum* Roxb.

The isolation of Taraxerone, 1-Hexacosanol, Taraxerol,  $\beta$ -sitosterol, Baccatin, 3,3'-di-O-methyl ellagic acid and 3-acetoxy aleuritic acid from the benzene extract of *Sapium baccatum* Roxb have been reported earlier<sup>3,4,5,6,7,8,9</sup>.

#### Section A : Extraction:

Dried and powdered trunk bark and stem of *Sapium baccatum* Roxb was extracted with benzene for about twenty hours in a Soxhlet apparatus. On cooling the benzene extract to room temperature, a yellow solid separated out. This was separated by filtration and was identified as 3,3'-di-O-methyl ellagic acid<sup>7</sup>. The filtrate was concentrated when a brown gummy residue was left behind. The residue was taken up in ether and the ether solution was washed with 10% aqueous sodium hydroxide solution to separate the acidic constituents and then with water to make the ether solution neutral. The ether solution was dried over anhydrous sodium sulphate and ether was distilled off. The gummy residue (neutral part) thus obtained was dissolved in

minimum amount of benzene and was chromatographed over deactivated alumina and the following fractions were separated.

Section B: Chromatography of the neutral part

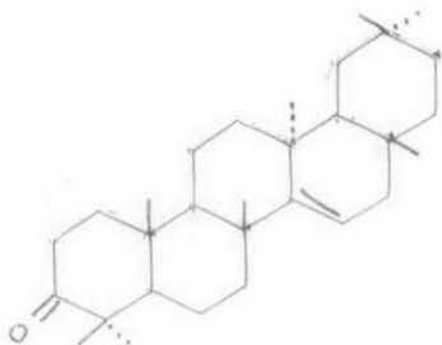
Table

Fraction No.	Eluent	Eluate	Melting point of the residue
1.	Petroleum ether	Solid with trace oil	220°-225°
2.	Petroleum ether: Benzene (4:1)	Solid	65-68°
3.	Petroleum ether: Benzene (3:2)	Solid	265°-270°
4.	Petroleum ether: Benzene (2:3)	Solid	130°-132°
5.	Benzene	Oil	-
6.	Benzene:Ether (4:1)	Solid	210°-220°

Section C: Examination of the fractions 1-6:

Fraction No. 1:

It has already been reported<sup>5</sup> that this fraction contains Taraxerone possessing structure



m.p. 233-240°

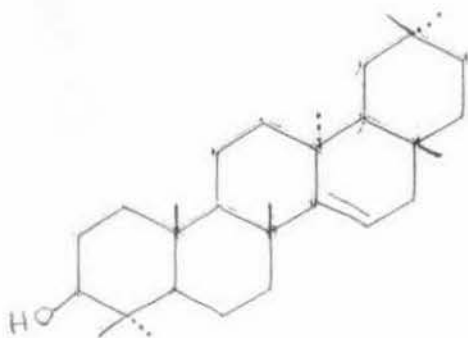
( $\alpha$ )<sub>D</sub> 10.5°

Fraction No. 2:

This fraction contains 1-hexacosanol m.p. 73-80°.

Fraction No. 3:

This fraction has been shown<sup>5</sup> to contain taraxerol.

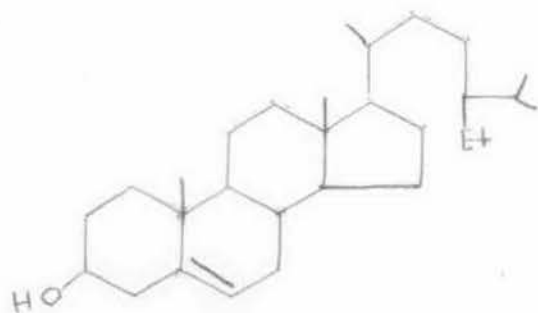


m.p. 273-280°

( $\alpha$ )<sub>D</sub> 73.7°

Fraction No. 4:

It has been reported<sup>3</sup> that this fraction contains  $\beta$ -sitosterol.



m.p. 136-137°

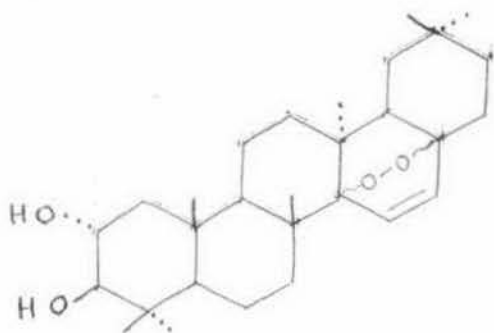
( $\alpha$ )<sub>D</sub> -32°

Fraction No. 5:

This fraction on rechromatography could not produce any solid material.

Fraction No. 6:

This fraction has been shown<sup>9</sup> to contain a nor-triterpene, Baccatin possessing the structure.

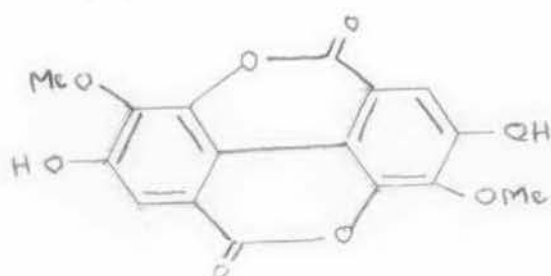


m.p. 223-229°

( $\alpha$ )<sub>D</sub> -9.09°

Section D:

The yellow insoluble solid which separated from the benzene extract on cooling has been characterised<sup>7</sup> as 3,3'-di-O-methyl ellagic acid possessing the structure



Section B:

Examination of the acid fraction of Sapium baccatum

Roxb:

The alkali soluble portion of the ether extract of Sapium baccatum Roxb was acidified with 10% aqueous hydrochloric acid and the precipitated solid was extracted with ether. The ether solution of the acid was washed with water to make it free from mineral acid, dried with anhydrous sodium sulphate. The ether solution was then concentrated when a gummy residue

was obtained. This was chromatographed in a column of B.D.H. silicagel. The chromatogram was developed with petroleum ether and was eluted with the following solvents:

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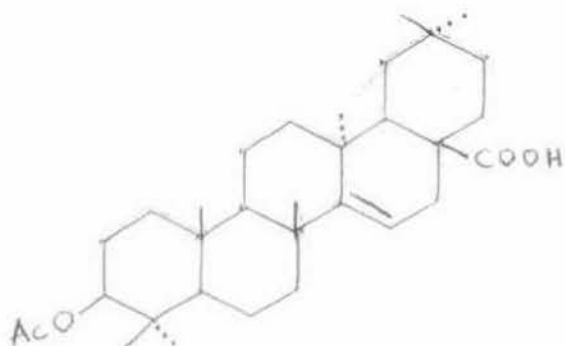
Eluent	Fractions 100 ml each	Residue on evaporation
Petroleum ether	1-6	Trace oil
Petroleum ether: Benzene (4:1)	7-10	Trace oil
Petroleum ether: Benzene (3:2)	11-19	Oily solid
Petroleum ether: Benzene (2:3)	20-26	Trace oil
Petroleum ether: Benzene (1:4)	27-36	Solid

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

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Fractions 27-36 were combined and the mass was crystallized from a mixture of chloroform and methanol when crystals of 3-acetoxy aleuritic acid m.p. 278-281<sup>o</sup> were obtained.



The structure of the compound<sup>9</sup> was further confirmed by esterification of the compound and comparing the product with authentic acetyl methyl ascorbate (mp and I.R. comparison).