CHAPTER -V

Reinvestigation of the neutral part of *Sapium sebiferum* Roxb.

Isolation of Moretenone:

The neutral portion B (160 g) of the benzene extract of *Sapium sebiferum* Roxb. [for extraction see page 60] was chromatographed over alumina (500 g) deactivated with 20 ml of 10% aqueous acetic acid. The chromatogram was developed with petrol and eluted with the following solvents:

Chromatography of 160 g of gummy material

Table - IX

Eluent	Fraction 100 ml each	Residue on evaporation	M.P. °C
Petrol	1-5	Oil	
	6-16	Solid	180-90°
Petrol:benzene (9:1)	17-22	Solid	200-215°
Petrol:benzene (@:1)	23-25	Oil	
Petrol:benzene	26-28	Solid	85-6°
(3:2)	29-35	Solid	200-20°

Eluent	Fraction 100 ml each	Residue on evaporation	M.P. °C
Petrol:benzene (1:1)	36-40	Solid	200-20°
Benzene	41-46	Nil	
Benzene:ether	47-56	Solid	210-15°

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

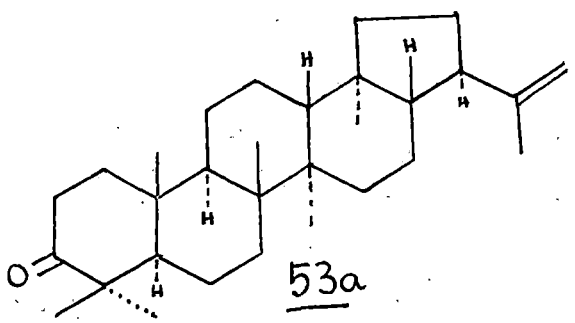
TLC examination of fractions (17-22) (Table -IX) showed that they were identical compounds and thus they were combined (2.5 g) m.p. 180-90° and was chromatographed over active alumina (120 g). The solid dissolved in benzene (5 ml) was placed on a column of alumina. The chromatogram was developed in petrol and was eluted with the following solvents (Table-X).

Chromatography of the above material (2.5 g)

Table - X

Eluent	Fractions 50 ml each	Residue on evaporation	M.P. °C
Petrol	1-3	Trace oil, soluble in petrol	---
Petrol:benzene (4:1)	4-7	Trace low melting solid soluble in petrol	50-60°
Petrol:benzene (3:2)	8-15	Crystalline solid	185-95°

The fractions 8 - 15 (Table - X) were combined (2.2 g) and crystallised from a mixture of chloroform and methanol to afford pure sample of moretenone 53a m.p. 198-99°, $[\alpha]_D$ 50°. It showed no depression in melting point when mixed with authentic sample of moretenone, IR of which are superimpossible.



53a

Found: C, 84.44%; H, 11.33%
Mol. wt. 407 (Rast)

Calculated for $C_{30}H_{48}O$ C, 84.84%; H, 11.39%
Mol. wt. 424

UV (95% ethanol): λ_{max} 287 nm (ϵ, 71.5)

IR (KBr disc): ν_{max}^{nujol} 1705, 1640, 875 cm^{-1}

Mass spectra: m/e 189, 205, 381, 424.

^1H NMR : Signals at 0.70, 0.94, 0.95, 1.02 (6-H), 1.08 (6-methyl groups); 1.68 (-C = C -CH_3); 2.33, 2.43 (-CO-CH_2 -protons); 4.68 (C = CH_2) ppm.

Colour reaction tests:

a) Tetranitromethane developed a yellow colour

b) Leibermann-Burchard reaction: This compound developed a violet colouration with a mixture of acetic anhydride and conc. sulphuric acid.

c) Zimmermann colour test was positive.

Fractions (17-22), (26-28), (29-35), & (36-40) in Table-IX were earlier identified as, 3-epimoretenol, moretenol, a low melting paraffin alcohol and β-sitosterol respectively by Pradhan[46b] et al.

Fractions (47-56) were found to be diol and whose identification is under progress.