

CHAPTER V

Section A : Investigation on the neutral part of Aleurites montana

Isolation of friedelin and β -sitosterol from the neutral part of trunk bark and stem of Aleurites montana.

Section B :

Extraction : Dried and powdered trunk bark and stem of Aleurites montana, was extracted with benzene in a soxhlet apparatus. The residue obtained by distilling off the benzene was taken in ether. The ether extract was washed with aqueous sodium hydroxide solution and then with water till it was neutral. The ether solution was dried over sodium sulphate (anhydrous) and the ether evaporated when a gummy residue was obtained.

Table VIII

Chromatography of the neutral gummy residue

The above gummy neutral part was chromatographed over deactivated alumina and the following fractions were collected.

| Fraction No. | Eluent | Eluate | Melting point of the residue |
|--------------|--------------------------------|----------------|------------------------------|
| 1 | Petroleum ether | Solid with oil | 244-48° |
| 2 | Petroleum ether: benzene (4:1) | Nil | |
| 3 | Petroleum ether: benzene (3:2) | Solid | 128-32° |

Section C

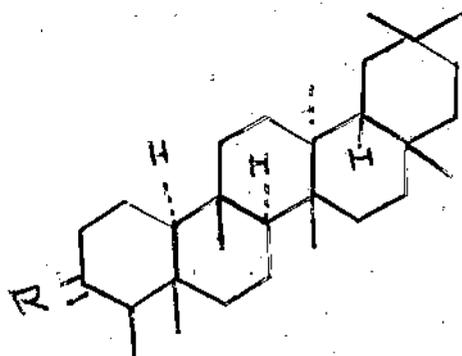
Examination of fraction No. 1 and isolation of friedelin 68

Fraction 1 (table VIII) on rechromatography over alumina followed by several crystallisation from chloroform and methanol mixture furnished fine needle shaped crystals, m.p. 256-58°, (α)_D - 36°. Elemental analysis and mass spectrum showed the molecular formula of the compound to be C₃₀H₅₂O (M^+ 426). It developed no coloration with tetranitromethane indicating that it was a saturated compound. It gave a violet coloration in Liebermann Burchardt reaction and a positive Zimmermann color test indicating that the compound is a triterpene ketone, the keto group being at the customary C-3 position⁵¹.

The compound 68 gave a reddish yellow 2,4-dinitrophenyl hydrazone derivative, C₃₆H₅₄N₂O₄, m.p. 286-88°, (α)_D + 52° and an oxime, C₃₀H₅₁NO m.p. 294-6°, showing that the oxygen atom was present as a carbonyl group. The infrared spectrum of the compound 68 showed a peak at 1705 cm⁻¹ indicating that the carbonyl group was present as a six membered ring ketone. The compound showed UV absorption, λ_{max} 285 m μ , (ϵ =70.3). The above chemical and physical data closely corresponded to that of friedelin and the compound 68 was found to be identical with an authentic sample of friedelin by m.m.p. determination and IR comparison. Lithium aluminium hydride reduction of compound 68 gave epi-friedelanol 69, C₃₀H₅₂O m.p. 274-6°, (α)_D + 9° and friedelanol 70, C₃₀H₅₂O, m.p. 297-9°, (α)_D + 15.6°, which were purified by chromatography followed by crystallisation.

Examination of fraction 3 : Isolation and identification of β -sitosterol, 71

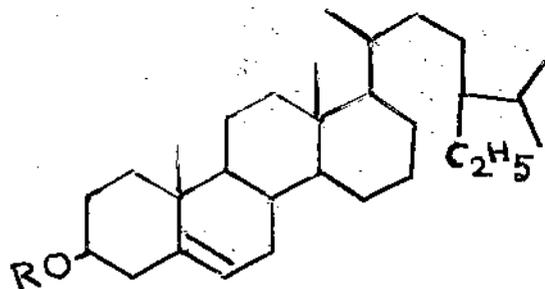
The crystalline solid obtained from the above chromatogram in the petroleum ether:benzene eluates (4:1) melted at $128-32^{\circ}$. The above solid after several crystallisations from methanol-chloroform mixture afforded a colorless solid m.p. $136-7^{\circ}$, $(\alpha)_D - 36.6^{\circ}$. Elemental analysis corresponded with the molecular formula $C_{29}H_{50}O$. On treatment with acetic anhydride and pyridine it furnished a crystalline material which after several crystallisations from a mixture of chloroform and methanol afforded the pure acetate, $C_{31}H_{52}O_2$, m.p. $127-9^{\circ}$, $(\alpha)_D - 40^{\circ}$. The acetate was identified as β -sitosterol acetate by direct comparison with an authentic specimen of β -sitosterol acetate 72. Hence the parent alcohol was identified as β -sitosterol 71.



68, R = O

69, R = R = H, (OH, α)

70, R = H, (OH, β)



71, R = H

72, R = CH_3CO-