SHORT COMMUNICATION

3β-ACETYLOLEANOLIC ACID FROM DREPANOCARPUS LUNATUS

T. DAHL* and T. B. H. McMurry*†

Faculty of Science, University of Lagos, Lagos, Nigeria

P. I. AMENECHI

Government Chemical Laboratories, Federal Ministry of Health, Lagos

and R. T. APLIN

Dyson Perrins Laboratory, University of Oxford, Oxford (Received 13 April 1966)

THERE are a number of leguminoseae which grow in the mangrove swamps around Lagos and in other coastal regions of West Africa. These species have to cope with brackish water that has a salt content which varies considerably from wet to dry season. We have examined one of the more common species, *Drepanocarpus lunatus*, in the hope that this might provide unusual extractives.

The wood‡ was debarked, broken up, air dried and extracted with light petroleum. From the concentrated extract we were able to isolate two substances. One of these was very rapidly identified with β -sitosterol, by comparison with an authentic specimen.

$$CO_2H$$
 CO_2H
 CO_2H
 CO_2H
 CO_2H
 CO_2H
 CO_2H

The other substance, obtained in 0·1 per cent yield, proved to be 3β -acetyloleanolic acid (I). It showed the presence of carbonyl and hydroxyl absorption in the i.r. region. The mass spectrum indicated a molecular weight of 498 (M⁺, 2%), and combined with elemental

- * Present address: University Chemical Laboratory, Trinity College, Dublin 2, Ireland.
- † To whom inquiries should be sent.
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- ¹ J. HUTCHINSON and J. M. DALZIEL, Flora of West Tropical Africa (2nd Ed.), Vol. 1, p. 519. Crown Agents for Overseas Governments and Administrations (1954–1958).

analysis suggested a molecular formula of $C_{32}H_{50}O_4$. The presence of an acetyl group was suggested by a peak at m/e 438 (9%) in the mass spectrum (mol. wt. – HOAc), and peaks at m/e 248 (100%), 233 (7%), 203 (61%) can be derived from the cations (II). (II – CH₃) and (II – HO₂C) respectively. A peak at m/e 133 (13%) corresponds to (III).

Comparison of melting points and a mixed melting point with an authentic specimen obtained from *Philadelphus ceronarius*³ confirmed its identity as the acetyl derivative of oleanolic acid. The methyl esters obtained from both specimens were identical, as were the acids obtained on hydrolysis. Acetyloleanolic acid has also been obtained from other species, Ligustrum japonicus, Arabia manschurica, Eugenia jambolana, Leucothoe grayana, and Pterocarpus angolensus.

EXPERIMENTAL

The main stems of *Drepanocarpus lunatus* were debarked, allowed to dry in the air for several days, and reduced to a coarse powder (2 kg). This was extracted in a Soxhlet apparatus with light petroleum (b.p. $60-80^{\circ}$) for 32 hr. The extract was concentrated to 25 ml, and on standing the resulting solution afforded 3β -acetyloleanolic acid (2·0 g) as colourless needles, m.p. 262-264 (lit. records m.p. $264-265^{\circ}$). undepressed with authentic material [α] $^{18}_{19}+74\cdot9^{\circ}$ (c. 0·83 in CHCl₃). (Found: C, 77·1; H, 10·0. Calc. for $C_{32}H_{50}O_4$: C, 77·06; H, $10\cdot11^{\circ}_{10}$). ν_{max} (Nujol) 3150. 1725, 1675, 1250, 1180, 1155 cm $^{-1}$. Thin-layer chromatography (light petroleum (b.p. 40-60) -ether (2:1) on silica gel G) showed only one spot with an identical R_f value to authentic material. Its methyl ester prepared by the action of diazomethane had m.p. $214-217^{\circ}$ (lit. m.p. 221-223). The oleanolic acid obtained by hydrolysis had m.p. $298-299^{\circ}$ (lit. m.p. $303-304^{\circ}$), methyl ester m.p. $198-199^{\circ}$ (lit. $96-198^{\circ}$).

Further concentration of the mother liquor and chromatography on alumina gave β -sitosterol (200 mg) as needles, m.p. 139, undepressed with an authentic sample.

- ² H. Budzikiewicz, J. M. Wilson and C. Djerassi, J. Am. Chem. Soc. 85, 3688 (1963).
- ³ R. Chan, Personal communication.
- ⁴ T. TAKEMOTO, N. YAHAGI and N. NISHIMOTO, J. Pharm. Soc Japan 75, 737 (1955); Chem. Abstr. 50, 3492 (1956).
- ⁵ N. K. Kochetkov, A. Ya. Khorlin, V. E. Vaskovski and V. E. Zhvirbi is, Zh. Obschei. Khim. 31, 658 (1961); Chem. Abstr. 55, 22367 (1961).
- ⁶ A. G. R. NAIR and S. SANKARA SUBRAMANIAN, J. Sci. Ind. Res. (India) 21B, 457 (1962): Chem. Abstr. 57, 17076 (1962).
- ⁷ Y. KONDO, T. SUGIYAMA, Y. TSUCHIDE and T. TAKEMOTO, Yukugaku Zasshi 83, 731 (1961); Chem. Abstr. 59, 14294 (1963).
- ⁸ F. E. KING, C. B. COTTERILL, D. H. GODSON, L. JURD and T. J. KING, J. Chem. Soc. 3693 (1953).
- ⁹ C. DJERASSI, G. H. THOMAS and H. MONSIMER, J. Am. Chem. Soc. 77, 3579 (1955).