

sterol,⁹ rutin,³ isoquercitrin³ and quercetin;³ *C. roxburghii*: lupeol,⁷ glucocapparin¹¹ and thiodamine;¹² *C. tapia*: β -amyrin, lupeol and betulinic acid.⁸

Plant part examined. Trunk bark. *Isolation and identification.* The powdered bark was defatted with light petroleum. The petroleum fraction was concentrated *in vacuo* and a white precipitate was obtained in a 0.7% yield. After recrystallization (acetone and EtOH), the compound was identified as lupeol by the following physical methods. It gave a positive Lieberman–Burchard test for triterpenes; m.p. 212–214°; $[\alpha]_D^{25} + 26.2^\circ$ (conc. 0.67 in CHCl_3); a violet chromogenic reaction with 70% H_2SO_4 after spraying TLC chromatograms. TLC on silica gel G gave the following R_f values: benzene (0.25), CH_2Cl_2 (0.40); EtOAc–MeOH (4:1) (0.90). The UV spectrum (MeOH): λ_{max} 200 nm (log 3.81). IR spectrum (KBr) at ν_{max} 3320(s), 1640(w), 1445(m), 1380(m), 1180(w), 1105(w), 1040(w), 1010(w), 980(w), and 940(w) cm^{-1} . The purity of the compound was evaluated by GLC (SE-30 Scot, 50 \times 0.02), $T = 230^\circ$; 18 min (solvent CHCl_3).

An NMR spectrum in CDCl_3 , using TMS as internal standard, showed signals at 4.64 δ (d, 2 protons), 3.15 δ (s, 1 proton), 1.30–1.78 δ ($-\text{CH}_2-$ and methine protons, 26), 0.74–1.05 δ (overlapping 7 C– CH_3 protons), the MS showed a molecular ion at m/e 426, which was also the base peak. Other major ions were M– CH_3 (23%) m/e 218 (78%), m/e 207 (75%) and m/e 189 (80%).

Finally, the compound was acetylated (Ac_2O and pyridine), and the acetate had m.p. 214–215°. The IR lacked bands at 3340 and 1104 cm^{-1} , which were replaced by bands at 1740 and 1245 cm^{-1} , indicative of the ester function.

¹¹ A. KJAER and H. THOMSEN, *Acta Chem. Scand.* **16**, 783 (1962).

¹² R. N. CHAKRAVARTI, *Indian Med. Gaz.* **86**, 152 (1951).

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COMPOSITAE

7,24(28)-STIGMASTADIEN-3 β -OL FROM *VERNONIA AMYGDALINA*

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Key Word Index—*Vernonia amygdalina*; Compositae; 7,24(28)-stigmastadien-3 β -ol.

Vernonia amygdalina (H) (Ibo name—Onugbu) is a valuable shrub wide spread in Nigeria. It is more commonly known as ‘bitter leaf’ because the leaves and the stem have a bitter taste when chewed, though there is a report of a ‘sweet’ variety.¹ The leaves of ‘bitter leaf’ are eaten after macerating with water to remove the bitter components and are believed to help digestion. It is not certain if other species like *Vernonia anthelmintica* and *Vernonia colorata* which are found elsewhere are eaten, but chemical investigations have been carried out.^{2a,b,3}

¹ B. OKIGBO, Agricultural Dept., University of Nigeria, Nsukka, personal communication.

² (a) D. J. FROST and J. P. WARD, *Tetrahedron Letters* 3779 (1968); (b) W. SUCROW, *Tetrahedron Letters* 2443 (1968).

³ TOUBIANA and RAOUL, *Compt. Rend.* **268C**, 82 (1969).

Unlike *V. anthelmintica* and possibly *V. colorata*, *V. amygdalina* does not produce seeds and in Nigeria, it is propagated by cuttings. Work on *V. anthelmintica* seeds^{2a} revealed that 7,24(28)-stigmastadien-3 β -ol was present along with other compounds; while *V. colorata*³ showed the presence of a sesquiterpene ester-hydroxy vernolide.

Light petroleum (b.p. 60–80°) extraction of the pulverized dry stem of *V. amygdalina*⁴ gave a brown oil. This oil was chromatographed on silica gel (Merck: Kieselgel, 0.2–0.5 mm, 30–70 mesh ASTM). Light petroleum and benzene fractions eluted oily materials that did not crystallize; 20% CHCl₃–benzene eluted a solid which was recrystallized from MeOH to give light green crystals (leaflets) m.p. 148–151° (C₂₉H₄₈O MW (MS) of 412). IR showed it to be a steroidal alcohol. It gave an acetate (Ac₂O–HOAc using *p*-toluene sulphonic acid as catalyst), m.p. and m.m.p., with an authentic sample^{2a} of acetate of 7,24(28)-stigmastadien-3 β -ol, 150–152°. The compounds were identical by TLC.

EXPERIMENTAL

Extraction of stem. The dried powdered stem of *V. amygdalina* (1.5 kg), extracted with light petroleum, gave an oil (4.5 g).

Isolation of 7,24(28)-stigmastadien-3 β -ol. The oil (4.5 g) was dissolved in light petroleum and chromatographed on silica gel (135 g). The column was eluted progressively and 20% CHCl₃–benzene eluted solids which crystallized from MeOH or benzene. Repeated crystallization yielded 7,24(28)-stigmastadien-3 β -ol as light green crystals (leaflets) (97 mg) m.p. 148–151° (Calc. C, 84.40; H, 11.72. Found: C, 84.31; H, 11.67%) with M⁺ as 412, C₂₉H₄₈O has M⁺ as 412.67; ν_{\max} 3300 cm⁻¹ (broad) in Nujol. (Acetate m.p. 150–152°.)

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⁴ Samples of *V. amygdalina* stem supplied by Mrs. V. N. ARENE and Mrs. C. EKUNDAYO of College of Education, University of Lagos.

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EUPHORBIACEAE

CONSTITUENTS OF *EUPHORBIA TINCTORIA*

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Key Word Index—*Euphorbia tinctoria*; Euphorbiaceae; nonacosane; octacosanol; γ -euphorbol.

Plant. *Euphorbia tinctoria* L. *Source.* Central parts of Iran plateau. *Use.* Plant is used as purgative. *Previous work.* None.

Roots, stem, leaves and flowers were extracted with light petroleum (40–60°). The residue was dissolved in petroleum and chromatographed on Kieselgel S. *Nonacosane*. C₂₉H₆₀ (Found, C, 84.90; H, 14.60. Req'd: C, 85.20; H, 14.80% m.p., m.m.p., IR and NMR). Earlier petroleum fractions and crystallization (MeOH–petroleum). *Unidentified ketone*. (m.p. 77°. IR 1720 cm⁻¹). From petroleum–benzene fractions (80–20) crystallized with MeOH. γ -*Euphorbol*. (m.p. 71–96°, IR 3400 cm⁻¹. Acetate, benzoate m.p., m.m.p. and IR 1740 cm⁻¹ and NMR). From benzene fractions. Crystallized from MeOH. *Octacosanol*. C₂₈H₅₈O (Found: C, 81.56; H, 14.08. Req'd: C, 81.67; H, 14.23%, IR 3418 cm⁻¹, m.p., m.m.p. Acetate, benzoate, m.p., m.m.p., IR 1740 cm⁻¹). From benzene–CHCl₃ (95–5) fractions: crystallized from MeOH–acetone (80–20).

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