Some non-alkaloidal constituents of the bark of 

clelea grandifolia engl.*

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As no further work is intended, the result of a preliminary examination of the bark of clelea grandifolia Eng., Rutaceae, is reported. T. grandifolia is stated (Colonial Products Research Council, London, List of Indigenous Plants of the Colonial Empire of Possible Scientific and Technical Interest, 1945) to be used medicinally in Africa. Related genera, e.g., Toddalia, Smithia, and Casimiroa have been shown to contain alkaloids. The present examination showed the presence of c. 1·2 per cent. alkaloids in T. grandifolia. The material, however, was amorphous in character and was not further examined. From the non-basic fractions were isolated the alcohol, tetracosan-1-ol, the triterpenoid, lupeol, a phytosterol which is probably β-sitosterol, and a substance apparently not previously described, C_{32}H_{64}O or C_{36}H_{78}O. There was also present an unidentified green syrup with a blue fluorescence.

Experimental

All melting points are corrected. Microanalyses were carried out in the C.S.I.R.O. Micro-analytical Laboratory under the direction of Dr. K. W. Zimmerman.

(i) Milled bark (1·9 kg) of T. grandifolia was mixed with lime (0·7 kg) and extracted exhaustively in a Soxhlet with methylene chloride (A), then with methanol (B). Both extracts were separately evaporated and the residues distributed between aqueous acetic acid and ether.

(ii) From the aqueous acid solutions were recovered after basification 22 g crude base from A and 1·5 g from B. The products were amorphous and neither the bases nor their salts could be obtained crystalline before or after chromatographing on alumina.

(iii) The non-basic ether-soluble fraction from A dissolved in methanol deposited a large quantity of gum, which was discarded. The methanol was removed, and the residue, dissolved in light petroleum, chromatographed on alumina. The column was eluted successively with light petroleum, benzene, and methylene chloride.

(iv) The first light petroleum fractions gave after purification tetracosan-1-ol, m. p. 76·5–77 °C (cf. Shah, Phalnikar, and Bhide 1947) (Found : C, 81·1; H, 14·2%). Calc. for C_{32}H_{64}O : C, 81·8; H, 14·2%). The substance gave no depression in m. p. when mixed with an authentic sample prepared by LiAlH_4 reduction of tetracosanoic acid. The X-ray diagrams of the two specimens were identical.

(v) The later light petroleum eluates yielded after crystallization from light petroleum a substance, m. p. 170·5–171 °C (Found : C, 85·1; H, 11·5; O, 4·1%; active "H", nil %). Calc. for C_{32}H_{64}O : C, 84·8; H, 11·3; O, 3·9%; for C_{36}H_{78}O : C, 84·9; H, 11·3; O, 3·8%). The substance gives a pink colour changing to pale blue in the Liebermann-Burchhardt test. A

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mixture with stigmasterol (C_{29}H_{48}O, m.p. 170–171 °C, cf. Fieser and Fieser 1949) melted at 140 °C.*

(vi) The first benzene eluates gave lupeol, m.p. 214–216 °C (Found: C, 84·1; H, 12·1; active "H", 0·23%). Calc. for C_{29}H_{48}O: C, 84·4; H, 11·8; active "H", 0·23%). Acetate, m.p. 215·5–216·5 °C (Found: C, 82·0; H, 11·2; O, 7·0%. Calc. for C_{31}H_{50}O_2: C, 82·0; H, 11·2; O, 6·8%). Benzoate, m.p. 269–270 °C (Found: C, 83·8; H, 10·3; O, 6·0%). Calc. for C_{31}H_{50}O_2: C, 83·7; H, 10·3; O, 6·0%). The m.p.’s of the lupeol and its derivatives were not depressed when mixed with authentic specimens.

(vii) The later benzene eluates contained a sterol, probably β-sitosterol, m.p. 133–134 °C (Found: C, 81·9; H, 12·0; active “H”, 0·23%. Calc. for C_{29}H_{48}O·H_2O: C, 82·2; H, 12·1; active “H”, 0·24%). This substance melts at 134–136 °C when mixed with a sitosterol of m.p. 136–137 °C isolated from Atalaya hemiglauca (F. Muell.) Benth. (unpublished data). Both give the same colour reaction in the Liebermann-Burckhardt test.

(viii) The methylene chloride eluates yielded a green syrup with a blue fluorescence which would not crystallize.

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References


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