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SHORT COMMUNICATION

CONSTITUENTS OF *ELEPHANTOPUS SCABER* (COMPOSITAE)

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Abstract—*Elephantopus scaber* has been found to contain epifriedelinol, lupeol, stigmasterol, potassium chloride, and a mixture of triacontan-1-ol and dotriacontan-1-ol.

Elephantopus scaber L., a wild shrub commonly found throughout the tropics, is used medicinally as a diuretic, tonic, anthelmintic and in the treatment of various stomach disorders.[1] Previous work on this plant by Ghanim and his co-workers[2] resulted in the isolation of only luteolin-7-glucoside. Related species that have been studied are *E. spicatus*[3] and *E. mollis*[4] which were screened for alkaloids and antibacterial substances respectively, and *E. elatus*, chemically examined by Kupchan and his colleagues[5] who isolated two novel tumour-inhibitory sesquiterpene dilactones, elephantin and elephantopin.

We have examined the *E. scaber* collected in Singapore. The whole plant material, dried and powdered, was extracted successively with light petroleum and ethanol. Alumina column chromatography of the neutral light petroleum extract yielded epifriedelinol, lupeol, stigmasterol, and a mixture of triacontan-1-ol and dotriacontan-1-ol. The ethanol extract furnished a large quantity of potassium chloride and a small amount of a phenolic compound, m.p. 208–210° (decomp.), which was insufficient for characterization. However, no luteolin-7-glucoside could be isolated from these extracts.

EXPERIMENTAL

M.ps were taken on a Hoover capillary apparatus. I.r. spectra were determined in Nujol using a Perkin-Elmer 137 instrument. Petroleum ether refers to the fraction of b.p. 56–70°.

Isolation of Epifriedelinol, Lupeol, a Mixture of Dotriacontan-1-ol and Triacontan-1-ol, Stigmasterol and Potassium Chloride

Ground whole plant (670 g) was exhaustively extracted (Soxhlet) successively with (*a*) petroleum ether for 72 hr and (*b*) 95% ethanol for 72 hr.

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(*a*) The petroleum ether extract was separated into an acidic fraction (0·58 g) and a neutral fraction (19·1 g). The acidic fraction failed to give any crystalline compound on chromatography on silica gel. Chromatography of the neutral sticky residue on alumina (350 g) yielded, on elution with benzene–$CHCl_3$ (10:1), an orange semi-solid which recrystallized thrice from benzene–$CHCl_3$ as colourless plates (0·12 g), m.p. 274–276°, $[\alpha]_{\mathrm{D}}^{25} + 25{\cdot}2°$ (*c* 1·14 in $CHCl_3$). (Found: C, 83·45; H, 12·44. Calc. for $C_{30}H_{52}O$: C, 84·05; H, 12·23 per cent.) The i.r. spectrum of the triterpene alcohol was identical with that of an authentic specimen of epifriedelinol. Literature[6] gives epifriedelinol m.p. 279–283°, $[\alpha]_{\mathrm{D}}^{13} + 24°$.

Elution of the column with benzene-$CHCl_3$ (10:3) furnished an orange solid (0·5 g) which recrystallized thrice from ether–petroleum ether as colourless needles, m.p. 206–212°, $[\alpha]_{\mathrm{D}}^{26} + 27{\cdot}7°$ (*c* 1·38 in $CHCl_3$). (Found: C, 84·17; H, 11·66. Calc. for $C_{30}H_{50}O$: C, 84·44; H, 11·81 per cent.) The compound gave a pink colour in the Liebermann–Burchard test and imparted a yellow colour to tetranitromethane. It formed an acetate, m.p. 205–211°, $[\alpha]_{\mathrm{D}}^{21} + 38{\cdot}4°$ (*c* 1·34 in $CHCl_3$) (Found: C, 82·40; H, 11·60. Calc. for $C_{32}H_{52}O_2$: C, 81·99; H, 11·18 per cent); a benzoate, m.p. 258–264° (Found: C, 83·20; H, 10·20. Calc. for $C_{37}H_{54}O_2$: C, 83·72; H, 10·25 per cent); and on oxidation with Jones reagent[7] yielded a ketone, m.p. 166–170° (Found: C, 84·82; H, 11·53. Calc. for $C_{30}H_{48}O$: C, 84·84; H, 11·39 per cent). The i.r. spectra of the triterpene alcohol and its benzoate were identical with those of authentic samples of lupeol and lupenyl benzoate, respectively. The mother liquors from the recrystallization of lupeol deposited a small amount of a colourless solid which on recrystallization twice from benzene–petroleum ether afforded a solid m.p. 89–90° (Found: C, 82·56; H, 14·31. Calc. for $C_{32}H_{66}O$: C, 82·32; H, 14·25 per cent). Concentration of the same mother liquor yielded a colourless residue which was twice recrystallized from the same solvents to give a solid, m.p. 87–88° (Found: C, 82·08; H, 13·63. Calc. for $C_{30}H_{62}O$: C, 82·11; H, 13·85 per cent). Both samples gave no coloration with tetranitromethane and their identical i.r. spectra showed a medium absorption at 3311 cm^{-1} (OH stretching) and a strong doublet at 730 and 720 cm^{-1} (methylene chain rocking). These substances are probably a mixture of dotriacontan-1-ol and triacontan-1-ol. Literature[8] gives dotriacontan-1-ol, m.p. 89·3–89·5°, and triacontan-1-ol, m.p. 86·3–86·5°.

Further elution of the column with $CHCl_3$–methanol (20:1) afforded an orange semi-solid which was recrystallized thrice from ether–petroleum ether to give colourless needles (0·12 g), m.p. 160–164°, $[\alpha]_{\mathrm{D}}^{25} - 45{\cdot}6°$ (*c* 2·85 in $CHCl_3$) (Found: C, 84·26; H, 11·65. Calc. for $C_{29}H_{48}O$: C, 84·40; H, 11·70 per cent). The compound gave a green-grown colour in the Liebermann–Burchard reaction and formed a monoacetate, m.p. 139–140°, $[\alpha]_{\mathrm{D}}^{25} - 47{\cdot}8°$ (*c* 0·92 in $CHCl_3$) (Found: C, 82·02, H, 11·40. Calc. for $C_{30}H_{50}O_2$: C, 81·88; H, 11·08 per cent). Literature[9] gives stigmasterol m.p. 168–169°, $[\alpha]_{\mathrm{D}} - 47{\cdot}3°$ (*c* 1·36 in $CHCl_3$); and the acetate, m.p. 139–140°, $[\alpha]_{\mathrm{D}} - 54°$ (*c* 1·78 in $CHCl_3$). The i.r. spectra of the sterol and its acetate were identical with those of authentic specimens of stigmasterol and its acetate, respectively.

(*b*) The ethanol extract on cooling deposited prisms (9·7 g) which was found to be KCl. The ethanol extract was concentrated and a portion of the dark sticky concentrate was worked up according to the method described by previous workers[2] but no luteolin-7-glucoside could be isolated. The other portion was triturated successively with 5% aq. Na_2CO_3 and 5% aq. NaOH. The acidified NaOH solution was extracted with ether and the dried extract on evaporation yielded a small amount of sticky greenish solid. This substance was successively recrystallized from methanol, acetone–benzene, and acetone–$CHCl_3$ to give a pale yellow solid, m.p. 208–210° (decomp.) (Found: C, 60·06; H, 4·75. $C_9H_8O_4$ required: C, 60·00; H, 4·48 per cent). This compound gave an intense dirty green coloration with $FeCl_3$ in alcohol. Its i.r. spectrums showed a strong absorption at 1653 cm^{-1} (chelated *o*-hydroxy chelated carbonyl group) and medium bands at 1610 and 1590 cm^{-1} (aromatic C═C stretching). The phenol formed a diacetate which crystallized from benzene–$CHCl_3$ in colourless needles, m.p. 202–203° (Found: C, 59·72; H, 5·00. $C_{13}H_{12}O_6$ required: C, 59·09; H, 4·58 per cent). The i.r. spectrum of acetate included strong bands at 1770 and 1205 cm^{-1} (vinyl acetate), 1689 cm^{-1} (non-chelated aromatic carbonyl).

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