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ANGIOSPERMAE DICOTYLEDONAE

CELASTRACEAE

HYDROCARBONS AND TERPENOIDS FROM THE BARK OF *LOPHOPETALUM RIGIDUM*

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Key Word Index—*Lophopetalum rigidum*; Celastraceae; friedelin; lupeol; betulin; sitosterol; alkanes.

Plant. Lophopetalum rigidum Ridley, indigenous to Sarawak. *Studies on sister species. L. toxicum*,¹ *L. wightianum*.²

Pulverized dry bark (950 g) was extracted first with carefully purified 60–80 petrol.—extract *A*, then with MeOH—extract *B*.

Extract A. On evaporation gave a yellow–green gum (16.5 g) which separated into five fractions by chromatography on SiO₂ eluting with petrol.–CHCl₃ and CHCl₃–MeOH mixtures. (1) (10% CHCl₃–petrol.)—white crystalline solid m.p. 82–83° from EtOAc. *R_f* 0.79 (SiO₂, 5% CHCl₃–C₆H₆). IR shows saturated ester; MS *m/e* ~ 900 (*M*⁺). Mother-liquor on evaporation, afforded a sweet smelling brown oil GLC–MS analysis of which shows the presence of the *n*-alkanes C₁₄ to C₂₀ (confirmed by direct comparison with a synthetic mixture). (2) (30% CHCl₃–petrol.)—friedelin (0.18%) m.p., m.m.p. 256–257° from C₆H₆ (lit.,³ 261–264°) [*α*]_D²⁰ –19° (C₆H₆). (Found: C, 84.4; H, 11.8. Calc. for C₃₀H₅₀O: C, 84.4; H, 11.8%). (3) (80% CHCl₃–petrol.)—lupeol (0.54%) m.p., m.m.p. 206–208° from MeOH (lit.,¹ 212–213°). [*α*]_D²⁰ +25.2° (CHCl₃). (Found: C, 84.3; H, 11.7. Calc. for C₃₀H₅₀O: C, 84.4; H, 11.8%). Acetate m.p. 214° from HOAc–Ac₂O (lit.,¹ 211–212°). (Found: C, 82.0; H, 11.2. Calc. for C₃₂H₅₂O₂: C, 82.0; H., 11.2%). (4) (100% CHCl₃)—sitosterol (0.003%) m.p., m.m.p. 135–137° from aq. MeOH (lit.,⁴ 137–138°) [*α*]_D²⁰ –38.3° (CHCl₃). (Found: C, 84.0; H, 12.3. Calc. for C₂₉H₅₀O: C, 84.0; H, 12.15%). (5) (5–60% MeOH–CHCl₃)—betulin (0.013%) m.p., m.m.p. 250–253° from aq. MeOH (lit.,¹ 252°) [*α*]_D²⁰ +20.3° (CHCl₃). (Found: C, 81.3; H, 11.4. Calc. for C₃₀H₅₀O₂: C, 81.4; H, 11.4%). Diacetate m.p. 210–211° from HOAc–Ac₂O (lit.,¹ 220°). (Found: C, 77.3; H, 10.3. Calc. for C₃₄H₅₄O₄: C, 77.5; H, 10.3%).

Extract B. On evaporation gave a red sticky solid (35 g) which, despite a very rigorous examination, resisted all attempts at purification. No evidenced of the polyol dulcitol was detected. This last observation is unusual since dulcitol is very common from members of the Celastraceae and is abundant in the sister species *L. toxicum*.¹

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³ M. SAINSBURY *Phytochem.* **9**, 2209 (1970).

⁴ L. J. SWIFT, *J. Am. Chem. Soc.* **74**, 1099 (1952).