

with ammonia and extracted with  $\text{CHCl}_3$ . The  $\text{CHCl}_3$  solution was evaporated to obtain a crude alkaloid. Recrystallization from petroleum gave colorless needles, m.p. 197–198°,  $[\alpha]_D^{25} -12^\circ$  (EtOH),  $\text{C}_{19}\text{H}_{27}\text{O}_6\text{N}^*$  which was estimated as senkirkine<sup>4</sup> from TLC, IR, NMR and mass spectral data and identical with the authentic sample by mixed m.p. and IR spectrum. The fresh roots (37 kg) and the dried leaves (3.65 kg) yielded 73 mg (0.0003%) and 130 mg (0.004%) of senkirkine, respectively.

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\* Molecular formula was measured by high resolution mass spectrometer and the analytical value was in good agreement with the theoretical value.

<sup>4</sup> L. H. BRIGGS, R. C. CAMBIE, B. J. CANDY, G. M. O'DONOVAN, R. H. RUSSELL and R. N. [SEELYE, *J. Chem. Soc.* 2492 (1965).

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## ERICACEAE

### LUPEOL AND $\beta$ -SITOSTEROL IN *ARBUTUS MENZIESII*\*

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THE MOST abundant triterpenoid of *Arbutus menziesii*, betulinic acid, has already been reported.<sup>1</sup> Two other bark constituents have now been examined.

Benzene fractions from the silica gel chromatography reported in our previous communication<sup>1</sup> were rechromatographed over neutral alumina (activity 3), affording an intimate mixture of two substances. The two pure components were separated by acetylation of the mixture and a single chromatography (silica gel) of the resulting acetates. The first of the two pure components was a colorless solid, m.p. 212–213°,  $[\alpha]_D^{25} + 33.4^\circ$  (c. 0.981,  $\text{CHCl}_3$ ). The mass spectrum of this component revealed a molecular ion peak at  $m/e$  426 (426.3860);  $\text{C}_{30}\text{H}_{50}\text{O}$  requires formula mass 426.3862. The cracking pattern, having intense

\* Taken from the Honours B.Sc. Thesis of Thomas N. McCaig, University of Victoria (1969).

<sup>1</sup> FRANK P. ROBINSON, JR. and HENRI MARTEL, *Phytochem.* 9, 907 (1970).

peaks at  $m/e$  218, 207 and 189 was similar to that of lupane triterpenoids,<sup>2</sup> suggestive of lupeol. Lupeol was further characterized through preparation of its acetate, m.p. 215–216° (lit.<sup>3</sup> m.p. 217–217.5°), benzoate, m.p. 258–259° (lit.<sup>3</sup> m.p. 264–266°), its hydrogenation product, lupanol, m.p. 201° (lit.<sup>4</sup> m.p. 201–202°), and its oxidation product, lupen-3-one, m.p. 167° (lit.<sup>5</sup> m.p. 172–174°). The mass spectrum of the hydrogenation product had a molecular ion peak at  $m/e$  428 (428.4017);  $C_{30}H_{52}O$  requires formula mass 428.4018. The mass spectral fragmentation pattern of the oxidation product of lupeol was virtually identical to the published pattern for lupen-3-one.<sup>2</sup> Therefore, the first component isolated from the benzene fractions was lupeol.

The second component, m.p. 133–134°, had a mass spectrum characteristic of a 3 $\beta$ -hydroxy steroid<sup>6</sup> [ $M-H_2O$ ,  $M-C_{10}H_{21}$  (side chain)]. This component was compared (IR, NMR, MS) and found identical with an authentic sample of  $\beta$ -sitosterol.  $\beta$ -Sitosterol was further characterized through preparation of its acetate, m.p. 131° (lit.<sup>7</sup> m.p. 125–6°).

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<sup>3</sup> THOMAS FITZGERALD, J. L. BEAL and J. B. LAPIDUS, *J. Pharm. Sci.* **52**, 712 (1963).

<sup>4</sup> T. R. AMES, T. G. HALSALL and E. R. H. JONES, *J. Chem. Soc.* 450 (1951).

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<sup>6</sup> R. RYHAGE and E. STENHAGEN, *J. Lipid Res.* **1**, 361 (1960).

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## EUPHORBIACEAE

### CONSTITUENTS OF *EXCOECARIA AGALLOCHA*

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*Plant.* *Excoecaria agallocha* L. *Uses.* Used in Sarawak as an ingredient of dart-poison and as a fish poison.<sup>1</sup> *Previous work.* None.

*Wood latex.* Chromatographed using  $SiO_2$ .  $\beta$ -Amyrin. M.p., mixed m.p., superimposable IR spectra.  $\beta$ -Amyrenone. Mixed m.p., superimposable IR spectra with authentic specimen prepared from  $\beta$ -amyrin. 3-Epi- $\beta$ -amyrin.  $C_{30}H_{50}O$ , m.p. 228°, acetate, m.p. 120°. Oxidized to  $\beta$ -amyrenone, mixed m.p., superimposable IR spectra. *Cycloartenol.*

\* On leave of absence from Kojin Co. Ltd.

<sup>1</sup> F. G. BROUNE, *Forest Tree of Sarawak and Brunei and their Products*, p. 180, Government Printing Office, Kuching, Sarawak, (1955).