

Previous studies of *A. tripartita* ssp. *tripartita* collected in Wyoming<sup>2</sup> resulted in the isolation of desacetoxymatricarin and ridentin. These studies collectively show the presence of closely related but not necessarily identical sesquiterpene lactones in samples collected from different locations.

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## TRITERPENES AND STEROL FROM THE ROOTS OF *ASTER SCABER*

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**Key Word Index**—*Aster scaber*; Compositae; friedelin; friedelan-3 $\beta$ -ol (*epi*-friedelinol); squalene;  $\alpha$ -spinasterol.

*Plant.* *Aster scaber* Thumb. (Compositae). *Source.* Hachioji, Tokyo, Japan. Voucher specimen is kept in the Herbarium of National Science Museum, Tokyo (TNS 314700). *Previous work.* On sister species, *A. tartaricus* (on flowers,<sup>1</sup> on roots<sup>2</sup>).

*Present work.* The residue obtained from the C<sub>6</sub>H<sub>6</sub> extract of *A. scaber* roots was extracted with light petrol.-C<sub>6</sub>H<sub>6</sub> (1:1). The solvents were evaporated to give a residue, which on repeated chromatographic separation followed by subsequent purification procedures afforded squalene,<sup>3</sup> friedelin,<sup>4</sup> friedelan-3 $\beta$ -ol (*epi*-friedelinol)<sup>5</sup> and  $\alpha$ -spinasterol.<sup>6</sup>

### EXPERIMENTAL

Dried roots (11 kg) of *A. scaber* were extracted with hot C<sub>6</sub>H<sub>6</sub> and the solvent was evaporated to give a residue (117 g). This was extracted with light petrol.-C<sub>6</sub>H<sub>6</sub> (1:1) and the residue (102 g) obtained after removal of the

<sup>1</sup> KARRER, P. and JUCKER, E. (1943) *Helv. Chim. Acta* **26**, 626.

<sup>2</sup> TAKAHASHI, M., KAMISAKO, W., ISHIMASA, S. and MIYAMURA, K. (1959) *J. Pharm. Soc. Japan*, **79**, 1281; MORIYAMA, Y., TANAHASHI, Y., TAKAHASHI, T. and OURISSON, G. (1968) *Bull. Soc. Chim. Fr.* 2890; and references therein.

<sup>3</sup> BATES, R. B. AND GALE, D. M. (1960) *J. Am. Chem. Soc.* **82**, 5749; and references therein.

<sup>4</sup> BOITEAU, P., PASICH, B. and RATSIMAMANGA, A. R. (1964) *Les Triterpénoides en Physiologie végétale et animale*, pp. 174, Gauthier-Villars, Paris.

<sup>5</sup> Ref. 4, p. 173.

<sup>6</sup> FIESER, L. F. and FIESER, M. (1959) *Steroids*, p. 352, Reinhold, New York; CLARK-LEWIS, J. W. and DAINIS, I. (1967) *Australian J. Chem.* **20**, 1961.

solvents was separated into 40 fractions by chromatography over alumina (2.5 kg) and successive elution with  $C_6H_6$  (fractions 1–17),  $C_6H_6-Et_2O$  (10:1) (fractions 18–22),  $C_6H_6-Et_2O$  (1:20) (fractions 23–26),  $Et_2O$  (fractions 27–28) and  $MeOH$  (fractions 29–40). The eluted fractions (each 700 ml) were collected and examined by TLC (silica gel).

Fractions 1–2 were combined and the solvent was distilled off. The residue (16 g) on further chromatography over silica gel (200 g; eluent: light petrol.) gave a residue (7 g) which was subjected to distillation under reduced pressure (0.1 mmHg; at 65°). The distillate was discarded and the residue was dissolved in  $Et_2O-EtOH$  (1:1). The soluble part was chromatographed on silica gel (200 g; eluent: light petrol.) to afford *squalene* (150 mg), a colourless oil, IR (liquid film) 2950, 2920, 2850, 1665, 1440, 1380 and 830  $cm^{-1}$ ; PMR ( $CDCl_3$ )  $\delta$  1.62 (18H, s; olefinic Me),  $\delta$  1.70 (6H, s; olefinic Me),  $\delta$  2.03 (20H, broad signal; methylene protons),  $\delta$  5.15 (6H, broad signal; olefinic protons); MS  $M^+$  at  $m/e$  410 ( $C_{30}H_{50}$ ); GLC  $R_i$  7.2 min (H-523, 1.5 m, 260°,  $N_2$ , 1.4 kg/cm<sup>2</sup>), identical (IR, PMR, MS, TLC and GLC) with an authentic sample.

Fractions 15–17 gave a solid (0.8 g), which was crystallized from ethyl acetate to give *friedelin* (0.5 g) m.p. 262–263°,  $[\alpha]_D^{25} - 25^\circ$  (c 1.0,  $CHCl_3$ ), IR (Nujol) 1706  $cm^{-1}$ , MS  $M^+$  at  $m/e$  426 ( $C_{30}H_{50}O$ ), identical (m.p., m.m.p.,  $[\alpha]_D$ , IR, PMR, MS and TLC) with an authentic specimen.

Fraction 28 gave a residue (2.5 g), which was chromatographed on silica gel (200 g; eluent:  $C_6H_6$ , each fraction: 150 ml). The fractions 6–8 gave a residue (0.3 g), which was crystallized from  $C_6H_6-Me_2CO$  (1:1) to afford *friedelan-3 $\beta$ -ol* (*epi-friedelinol*), m.p. 285–287°,  $[\alpha]_D^{25} + 24^\circ$  (c 0.7,  $CHCl_3$ ), IR (Nujol)  $\sim$  3450  $cm^{-1}$ , MS  $M^+$  at  $m/e$  428 ( $C_{30}H_{52}O$ ), identical with a genuine sample in all respects (m.p., m.m.p.,  $[\alpha]_D$ , IR, PMR, MS and TLC). The fractions 10–12 gave a residue (0.3 g) containing aliphatic alcohols. No further examination of this residue was effected. The fractions 16–22 were combined to give a residue (0.8 g), which was purified by chromatography over silica gel (30 g; eluent:  $C_6H_6$ ) followed by crystallization from  $C_6H_6-MeOH$  (1:1) to afford  $\alpha$ -*spinasterol* (5 $\alpha$ -stigmasta-7, 22-dien-3 $\beta$ -ol)(1) (0.5 g), m.p. 153–155°,  $[\alpha]_D - 4^\circ$  (c 3.0,  $CHCl_3$ ), IR (Nujol)  $\sim$  3450,  $\sim$  1660, 1035 and 975  $cm^{-1}$ ; MS  $M^+$  at  $m/e$  412 ( $C_{29}H_{48}O$ ). The positions of the side chain Me signals in the PMR spectrum (in  $CDCl_3$ ) of **1** were identical with those of the same signals in a PMR spectrum of authentic stigmasterol. In the spectrum of **1**, a broad signal due to three olefinic protons appears at around  $\delta$  5.1. The positions of the C-18 and C-19 Me singlets ( $\delta$  0.56 and 0.81) agree well with the calculated values ( $\delta$  0.58 and 0.78)<sup>7</sup> for  $\Delta^7$ -steroid. Acetylation of **1** with  $Ac_2O$  and pyridine gave an acetate, m.p. 176–178°,  $[\alpha]_D - 6.5^\circ$  (c 2.3,  $CHCl_3$ ), whose IR spectrum (in Nujol: 1735, 1660, 1250, 1165, 1100, 1035, 975, 900, 850, 835 and 800  $cm^{-1}$ ) was identical with that of authentic  $\alpha$ -spinasteryl acetate.

<sup>7</sup> BHACCA, N. S. and WILLIAMS, D. H. (1964) *Applications of NMR Spectroscopy in Organic Chemistry*, p. 21. Holden-Day, San Francisco.

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## WAX OF *BACCHARIS CORIDIFOLIA*

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*Plant.* *B. coridifolia* D.C. (mío-mío, romerillo), branched bush 40–80 cm in height, toxic to cattle, common in Argentina, Uruguay, south of Brazil and Paraguay. *Source.* Province of Entre Ríos, Argentina. *Previous work.* General characteristics were described.<sup>1</sup>

<sup>1</sup> ARATA, P. N. (1877), *Anales Soc. Cientif. Arg.* **4**, 34; BRANDL, J. and SCHAEFTEL, G. (1915) *Arch. Pharm.* **253**, 195; ARREGUINE, V. (1918) Thesis. Fac. Cs. Exactas. Físicas y Naturales. Univ. Buenos Aires.