

TLC gave the following approximate percentages: monoalcohols 71.7, hydrocarbons 4, fatty acids 17.3, diols 5.2 (tentatively), other components 1.8%.

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STEROLS AND TRITERPENES FROM *EUPATORIUM PERFOLIATUM*

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Key Word Index—*Eupatorium perfoliatum*; Compositae; sterols; triterpenols; dotriacontane sitosterol; stigmasterol; α -amyrin; 3β -hydroxy-ursa-20-ene.

Plant. *Eupatorium perfoliatum*. *Use.* Unknown.

Previous work. Isolation of terpenes,¹ flavonoids^{2,3} and on sister species.⁴

Present work. The dried, powdered leaf and twigs (1200 g) were extracted successively with petrol. (b.p. 30–60°) and EtOH. The ethanolic extract gave negative the tests for sesquiterpene lactones and alkaloids.⁵ The petrol. extract was chromatographed on silica gel. The fractions were eluted successively with C₆H₆–CHCl₃, acetone, EtOH and worked up as usual, all the common compounds were identified by IR, NMR, $[\alpha]$, m.m.p., co-TLC and if possible the properties of their acetates and ketones. The chromatographic column afforded: dotriacontane (2.76 g); α -amyrin (0.26 g); sitosterol (0.48 g); stigmasterol (0.30 g), and 3β -hydroxy-ursa-20-ene (6.2 g), tetranitromethane and L–B tests positive. m.p. 204–206°, soln in CHCl₃, $[\alpha]_{589}^{260} + 26.0^\circ$; $[\alpha]_{578} + 28.0^\circ$; $[\alpha]_{546} + 31.1^\circ$; $-\ [\alpha]_{436} + 57.4^\circ$ $[\alpha]_{365} + 97^\circ$; $[\alpha]_{316} + 149$ (Anal. Found: C, 83.84; H, 11.60; O, 4.58. Calcd. for C₃₀H₅₀O: M⁺ 426, C, 84.44, H, 11.81; O, 3.75%. UV: $\lambda_{\max}^{\text{EtOH}}$ 207 nm (ϵ 2300). IR: $\nu_{\max}^{\text{KBr}}$ 3300, 2890, 1625, 1450, 1370, 1360, 1225, 1160, 1125, 1025, 970, 880 cm⁻¹. NMR: typical triterpenol insaturated. MS: M⁺ 426 (100%), m/e (%) 411 (15), 393 (4), 313 (13), 250 (9), 242 (7), 231 (11), 217 (13), 216 (18), 215 (53), 205 (16), 204 (48), 203 (15), 202 (22), 201 (24), 200 (26), 189 (26), 188 (21), 187 (44), 174 (15), 162 (15), 160 (23), 135 (45), 121 (44), 109 (51), 108 (53), 95 (56), 93 (44), 83 (26), 81 (53), 71 (18), 69 (58), 67 (33), 57 (22), 55 (53), 43 (44), 3β -acetoxy-ursa-20-ene, needles, m.p. 202° (Anal. Found: C, 82.05, H, 11.04; O, 7.10. Calcd. for C₃₂H₅₂O₂: C, 81.99, H, 11.18; O, 6.83% soln CHCl₃ $[\alpha]_{589}^{250} + 39.8^\circ$ $[\alpha]_{578} + 41.8^\circ$ $[\alpha]_{546} + 47.8^\circ$; $[\alpha]_{436} + 83.8^\circ$; $[\alpha]_{365} + 136^\circ$; $[\alpha]_{316} + 216.8^\circ$. IR: $\nu_{\max}^{\text{KBr}}$ 1725, 1225 cm⁻¹.

* Part III in the series "The Chemistry of Mexican *Eupatorium* genus". For Part II see Ref. 4.

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NMR: (δ , H_2), 2.01 (s, 3H). *ursa-20-en-3-one*. Plates, m.p., 162–1963° (*Anal.* Found: C, 84.44; H, 10.96; O, 4.28. Cald. for $C_{30}H_{48}O$: C, 84.84; H, 11.39 O, 3.77%. Soln $CHCl_3$ $[\alpha]_{589}^{25} + 49^\circ$ $[\alpha]_{578}^{25} + 51.0^\circ$ $[\alpha]_{546}^{25} + 60.3^\circ$; $[\alpha]_{436}^{25} + 114.3^\circ$ $[\alpha]_{365}^{25} + 207.0^\circ$ $[\alpha]_{316}^{25} + 222.4^\circ$. $\nu_{\max}^{KBr}$ 1700 cm^{-1} . *3 β -hydroxy-ursane*. m.p. 204–206°. (*Anal.* Found, C, 84.26, H, 12.23, O, 4.02. Cald. for $C_{30}H_{52}O$: C, 84.04, H, 12.23; O, 3.73%) Soln $CHCl_3$ $[\alpha]_{589}^{25} - 12.9^\circ$; $[\alpha]_{578}^{25} - 13.4^\circ$; $[\alpha]_{546}^{25} - 13.8^\circ$; $[\alpha]_{436}^{25} - 22.8^\circ$; $[\alpha]_{363}^{25} - 31.1^\circ$.

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SESQUITERPENES FROM THE ROOTS OF *LIGULARIA HODGSONII*

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Key Word Index—*Ligularia hodgsonii*; Compositae; sesquiterpene; furanoeremophilane; bakkenolide A (fukinanolide); eremophilanolide.

Plant. *Ligularia hodgsonii* Hook. f. (Compositae). *Source.* Mt. Yakeishi-dake, Iwate prefecture, Japan. Voucher specimen is kept in the Herbarium of National Science Museum, Tokyo (TNS 201645). *Previous work.* On roots (furanoeremophilan-14 β ,6 α -olide).¹

Present work. Dried roots (1.7 kg) were extracted with hot C_6H_6 . The solvent was evaporated to give the residue (54 g) which was dissolved in warm light petrol. The soluble material was sublimed at 100° under reduced pressure (1 mmHg) and the material sublimed (11 g) was chromatographed on silica gel. Elution with light petrol. gave a colourless oil (48 mg), $C_{15}H_{22}O$ (M^+ at m/e 218), $[\alpha]_D - 12^\circ$ ($CHCl_3$). UV: $\lambda_{\max}^{MeOH}$ 221 nm (ϵ 7000). IR: (liquid film) 1649, 1568, 1094 cm^{-1} ; PMR ($CDCl_3$) δ 0.95 (3H, s; tert-Me), δ 0.99 (3H, d, J 7 Hz; sec-Me), δ 1.99 (3H, d, J 1 Hz; $-\underline{CH=C=Me}$), δ 7.13 ppm (1H, m; $-\underline{O-CH=C=Me}$), identical ($[\alpha]_D$, IR, PMR, MS) with furanoeremophilane.² Elution with light petrol.– Et_2O (100:3) afforded a crystalline compound, which was recrystallized from light petrol. to give 690 mg of bakkenolide A³ (fukinolide⁴), m.p. 80.5–81.5°, $C_{15}H_{22}O_2$ (M^+ at m/e 234), $[\alpha]_D + 20^\circ$ (MeOH). IR: (Nujol) 1780, 1665, 900 cm^{-1} . PMR: (C_6D_6) δ 0.68 (3H, d, J 6

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