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 ($CDCl_3$) δ 1·62 (18H, *s*; olefinic Me), δ 1·70 (6H, *s*; olefinic Me), δ 2·03 (20H, broad signal; methylene protons), δ 5·15 (6H, broad signal; olefinic protons); MS M^+ at *m/e* 410 ($C_{30}H_{50}$); GLC R_t 7·2 min (H-523, 1·5 m, 260°, N_2, 1·4 kg/cm^2), 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. 262·5–263°, $[\alpha]_D^{25}$ − 25° (*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-3β-ol* (*epi*-friedelinol), m.p. 285–287°, $[\alpha]_D^{25}$ + 24° (*c* 0·7, $CHCl_3$), IR (Nujol) ~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 *α-spinasterol* (5α-stigmasta-7, 22-dien-3β-ol)(**1**)(0·5 g), m.p. 153–155°, $[\alpha]_D$ − 4° (*c* 3·0, $CHCl_3$), IR (Nujol) ~3450, ~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 δ 5·1. The positions of the C-18 and C-19 Me singlets (δ 0·56 and 0·81) agree well with the calculated values (δ 0·58 and 0·78)[7] for Δ^7-steroid. Acetylation of **1** with Ac_2O and pyridine gave an acetate, m.p. 176–178°, $[\alpha]_D$ − 6·5° (*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 α-spinasteryl acetate.

[7] 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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Key Word Index—*Baccharis coridifolia*; Compositae; wax; fatty acids; fatty alcohols; hydrocarbons.

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.* Gencral characteristics were described.[1]

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

TABLE 1. MAIN COMPONENTS OF *Baccharis* wax

Carbon No.	Sat. fatty acids (%)	Unsat. fatty acids (%)	Hydrocarbons (%)	Monoalcohols (%)
12	—	Traces	—	—
13	Traces	0·12	—	—
14	7·65	Traces	—	—
15	Traces	—	—	—
16	25·28	3·60 (16:1)	—	—
17	Traces	13·91	—	—
18	5·97	34·0 (18:1) 5·0 (18:2) 0·92 (18:3)	—	0·76
19–22	—	—	—	Traces
23	—	—	—	0·13
24	2·95	—	—	0·17
25	—	—	0·65	0·49
26	—	—	1·10	0·42
27	—	—	6·00	1·00
28	—	—	4·03	9·34
29	—	—	30·53	3·12
30	—	—	5·08	54·74
31	—	—	33·20	3·51
32	—	—	1·13	26·11
33	—	—	15·02	—
34	—	—	—	—
35	—	—	3·27	—

Present work. The wax was isolated as a white solid, m.p. 82°, in 0·5% yield from whole plant by extraction with petrol. It was saponified by known procedures.[2] The saponifiable components (19·1%) were separated by TLC (silica gel G using petrol–Et_2O–HOAC, 90:10:1) as (i) methyl esters of saturated and unsaturated fatty acids, by previous methylation with an ethereal solution of CH_2N_2, and (ii) methyl esters of hydroxylated fatty acids (IR 3370 cm^{-1}, OH) and probably lactones. GLC of fraction (ii) showed eleven peaks that were not identified. Fraction (i) was examined by GLC (isothermically at 180°, glass column 3·60 m × 2 mm, packed with 3% EGS on chromosorb G-DMGS 100–200 mesh) directly and after reduction of the double bonds of the unsaturated fatty acids, to ascertain their carbon atom number. The principal unsaturated components were determined by the use of appropriate standards (Table 1). From the unsaponifiable components (80·8%), the hydrocarbons were extracted with petrol; by GLC at 250° in a glass column 1·80 m × 2 mm, packed with 10% DC 200 on gas-chrom Q 80–100 mesh) the mixture showed a high proportion of C_{29}, C_{31} and C_{33} hydrocarbons, as is common in higher plants.[3] Alcohols were then extracted afterwards with Et_2O, and the monoalcohols were isolated free from diols by preparative TLC (using petrol.–Et_2O–HOAC 7:3:1); then they were acetylated and examined by GLC (240° on a glass column 1·80 m × 2 mm, packed with 1·5% SE30 on Chromosorb W-DMCS 80–100 mesh). An Et_2O-insoluble fraction was considered to be composed mainly by diols, but was not further investigated. Preparative

[2] RAZAFINDRAZAK, J. and METZGER, J. (1963) *Bull. Soc. Chim. Fr.* 1630.
[3] EGLINTON, C. and HAMILTON, R. J. (1963) *Chemical Plant Taxonomy* (SWAIN, T., ed), Academic Press, New York.

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 C_6H_6–$CHCl_3$, acetone, EtOH and worked up as usual, all the common compounds were identified by IR, NMR, [α], 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 $CHCl_3$, $[\alpha]_{589}^{26°}$ + 26·0°; $[\alpha]_{578}$ + 28·0°; $[\alpha]_{546}$ + 31·1°; − $[\alpha]_{436}$ + 57·4° $[\alpha]_{365}$ + 97°; $[\alpha]_{316}$ + 149 (*Anal.* Found: C, 83·84; H, 11·60; O, 4·58. Calcd. for $C_{30}H_{50}O$: M^+ 426, C, 84·44, H, 11·81; O, 3·75%. UV: λ_{max}^{EtOH} 207 nm (ε 2300). IR: ν_{max}^{KBr}, 3300, 2890, 1625, 1450, 1370, 1360, 1225, 1160, 1125, 1025, 970, 880 cm^{-1}. NMR: tipical 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 $C_{32}H_{52}O_2$: C, 81·99, H, 11·18; O, 6·83% soln $CHCl_3$ $[\alpha]_{589}^{25°}$ + 39·8° $[\alpha]_{578}$ + 41·8 $[\alpha]_{546}$ + 47·8; $[\alpha]_{436}$ + 83·8°; $[\alpha]_{365}$ + 136°; $[\alpha]_{316}$ + 216·8°. IR: ν_{max}^{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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