

## TRITERPENOID AND OTHER CONSTITUENTS OF *XANTHORHIZA SIMPLICISSIMA*

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**Key Word Index**—*Xanthorhiza simplicissima* Ranunculaceae, ferulic acid, fatty acids, ursolic acid, *n*-alkanes, sitosterol, campesterol, stigmasterol, cholesterol, palmitate.

**Plant** *Xanthorhiza simplicissima* Marsh. **Source** S. B. Penick and Co., New York. Lot No. 814-BKM-1. **Uses** Has been used as a medicinal in the United States.<sup>1</sup> **Previous work** On rhizomes and roots.<sup>2,3</sup>

**Present work** The dried, ground rhizomes and roots (10 kg) were extracted by percolation with EtOH. After removal of the solvent *in vacuo* at 40°, the residue (1.2 kg) was partitioned between 2% aq. citric acid and Et<sub>2</sub>O to give a basic and non-basic fraction. The non-basic fraction was fractionated by standard methods into neutral (55 g), acidic (40 g) and phenolic (9 g) fractions.

**Phenolic fraction** Chromatography over silicic acid and elution with CHCl<sub>3</sub> gave a fraction which, when re-chromatographed over silicic acid and eluted with CHCl<sub>3</sub>-MeOH (4:1) afforded *ferulic acid* (60 mg), m.p. 165.5–166.5° (light petrol),  $\lambda_{\text{max}}^{\text{MeOH}}$  216 nm (log  $\epsilon$  4.22), 234 (4.17), 295 (4.23), 320 (4.35),  $\nu_{\text{max}}^{\text{KBr}}$  3460, 1670, 1600, 1510 and 1270 cm<sup>-1</sup>. MS  $M^+$   $m/e$  194 (100%), 179 (24), 177 (10), 150 (14). Direct comparison (m.p., m.m.p., UV, IR and MS) with an authentic sample confirmed the identity.

**Acidic fraction** Chromatography over silicic acid and elution with light petrol-C<sub>6</sub>H<sub>6</sub> (4:1) gave a fatty acid fraction which crystallized from MeOH (200 mg), m.p. 70–71°,  $\nu_{\text{max}}^{\text{KBr}}$  2940, 2860, 1700, 1460, 1300 and 720 cm<sup>-1</sup>. GLC of the methyl esters on a 160 cm column of 0.8% OV-17 on Gas Chrom Q (80–100 mesh) showed the mixture to be composed of C<sub>22</sub> (24%), C<sub>23</sub> (15), C<sub>24</sub> (46), C<sub>25</sub> (8) and C<sub>26</sub> (6) saturated straight fatty acids. The identity was confirmed by GC-MS.

Elution with C<sub>6</sub>H<sub>6</sub>-CHCl<sub>3</sub> (1:3) gave *ursolic acid* (150 mg), m.p. 242–243° (light petrol-MeOH) (lit.<sup>4</sup> 224–244° [ether], 260–280° [EtOH], 280–281° [Et<sub>2</sub>O-light petrol]),  $\nu_{\text{max}}^{\text{KBr}}$  3450, 2950, 2890, 1685, 1450, 1030 and 990 cm<sup>-1</sup>,  $[\alpha]_D^{29} + 70^\circ$  (c 0.4 CHCl<sub>3</sub>). MS  $M^+$  456 (3%), 248 (100). Treatment with acetic anhydride-pyridine gave *O-acetylursolic acid* m.p. 268–270° (light petrol),  $\nu_{\text{max}}^{\text{KBr}}$  1735, 1690 and 1234 cm<sup>-1</sup>,  $[\alpha]_D^{29} + 53.7^\circ$  (c 0.7 CHCl<sub>3</sub>). Direct comparison (m.p., m.m.p., IR, MS) with an authentic sample of the compound and derivative confirmed the identity.

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<sup>1</sup> COOK, E. F. and LAWALL, C. H. (1926) *Remington's Practice of Pharmacy*, 7th Edn, p. 1165. Lippincott, Philadelphia, Pa.

<sup>2</sup> HUSSEIN, F. T., BEAL, J. L. and CAVA, M. P. (1963) *Lloydia* **26**, 254.

<sup>3</sup> KNAPP, J. E., HUSSEIN, F. T., BEAL, J. L., DOSKOTCH, R. W. and TOMIMATSU, T. (1967) *J. Pharm. Sci.* **56**, 139.

<sup>4</sup> ALJADIN, M., BRYCE, T. A., CLAYTON, E., MARTIN-SMITH, M. and SUBRAMANIAN, G. (1955) *J. Chem. Soc.* 4611.

**Neutral fraction** Chromatography over silicic acid and elution with light petrol gave an alkane fraction which crystallized from MeOH (75 mg), m p 65–65.5°,  $\nu_{\max}^{\text{KBr}}$  2930, 2860, 1460, 725 and 715  $\text{cm}^{-1}$  GLC on a 160 cm column of 0.8% OV-17 on Gas Chrom Q (80–100 mesh) showed the mixture to be composed primarily of  $\text{C}_{25}$  to  $\text{C}_{35}$  *n*-alkanes,  $\text{C}_{25}$  (1%),  $\text{C}_{26}$  (2),  $\text{C}_{27}$  (6),  $\text{C}_{28}$  (4),  $\text{C}_{29}$  (20),  $\text{C}_{30}$  (5),  $\text{C}_{31}$  (25),  $\text{C}_{32}$  (4),  $\text{C}_{33}$  (19),  $\text{C}_{34}$  (2),  $\text{C}_{35}$  (1) The identity was confirmed by GC-MS Elution with light petrol– $\text{C}_6\text{H}_6$  (2/3) gave a sterol mixture which crystallized from MeOH (400 mg), m p 136–137°,  $\nu_{\max}^{\text{KBr}}$  3450, 2960, 1460, 1375 and 1060  $\text{cm}^{-1}$  GLC on a 160 cm column of 0.8% OV-17 on Gas Chrom Q (80–100 mesh) showed the mixture to be composed of *sitosterol* (55%), *campesterol* (32), *stigmasterol* (9) and *cholesterol* (4) The identity was confirmed by GC-MS The spectra were consistent with those of authentic samples

**Basic fraction** Refrigeration of the acidic solution gave an alkaloidal precipitate (157 g) Chromatography over silicic acid and elution with  $\text{CHCl}_3$ –MeOH afforded an alkaloid fraction (1.2 g) Treatment with saturated methanolic KI gave *palmatine iodide* (60 mg), m p 228–229°,  $\lambda_{\max}^{\text{MeOH}}$  227 nm ( $\log \epsilon$  4.58), 270 (4.42), 350 (4.41) and 439 (3.71)  $\nu_{\max}^{\text{KBr}}$  1600, 1360, 1270 and 1020  $\text{cm}^{-1}$  MS  $\text{M}^+$  *m/e* 352 (8%), 351 (17), 337 (95) and 142 (100) Reduction with  $\text{NaBH}_4$  gave *tetrahydropalmatine* m p 142° (MeOH)  $\lambda_{\max}^{\text{MeOH}}$  209 ( $\log \epsilon$  4.02) and 282 (3.78) MS  $\text{M}^+$  355 (40%), 339 (40), 164 (100) and 149 (50) Direct comparison (m p, m m p, IR, MS) with an authentic sample of the compound and derivative confirmed the identity The previously isolated compounds, *jatrorrhizine*,<sup>2</sup> *oxyacanthine*<sup>3</sup> and *obamegine*<sup>3</sup> were re-isolated and identified by m p, m m p, IR, MS and optical rotation

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## ALCALOIDES DE *BAUERELLA BAUERI*

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**Key Word Index**—*Bauerella baueri*, Rutaceae, acridones, acronycine, melicopidine

*La plante Bauerella baueri* (Schott) Engler Cette espèce a été récoltée le 10 septembre 1968 à Pouembout (Nouvelle Calédonie) par Sevenet et McKee Elle fait encore l'objet