

IDENTIFICATION OF BAUERENOL IN *SOLIDAGO ALTISSIMA*

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Two triterpenes, one of which was identified as bauerenol, were isolated from a methanol extract of the roots of *Solidago altissima* L. together with two acetylenic compounds. Takemoto *et al.* reported the occurrence of ilexol in this plant, while later Lahey and Natori *et al.* showed that ilexol was identical with bauerenol isolated from *Acronychia baueri* (1-4). We report the first direct isolation and identification of bauerenol from *S. altissima*.

EXPERIMENTAL¹

PLANT MATERIAL.—The plant material used in this study was collected in December of 1979 on the campus of this University.

EXTRACTION AND FRACTIONATION.—The air-dried chipped roots (7.3 kg) of *S. altissima* were extracted with methanol (31 liters) at room temperature for 2-4 weeks. The solvent was removed *in vacuo* at 40° until the total volume became ca. 2 liters; this was extracted with diethyl ether (400 ml x 5). Removal of the ether gave oils (100 g) that were dissolved in chloroform (300 ml) and chromatographed on silica gel (Merk Kiesel gel-60). The chloroform and chloroform-diethyl ether fractions gave 2-*cis*-dehydromatricaria methyl ester, mp 115° (2.6 g) (5-7) and 10-angeloyloxy-2-*cis*,8-*cis*-matricaria methyl ester, oil (3.2 g) (8,9), identified by comparison with the spectral data reported in the literature (5-9). From a fraction between these two compounds, a new triterpene ketone, mp 158.3° was obtained: $[\alpha]^{22}_D - 53.1^\circ$ (c = 1.3, CHCl₃); ir (KBr), ν max 1710 cm⁻¹; pmr (CDCl₃) δ , 5.05 (t, -C=CH-CH₂-, J = 7 Hz); ms: *m/z* 426 (28%), 409 (12), and 339 (58).

Anal. calcd for C₃₀H₄₈O: C, 84.84; H, 11.39. Found: C, 84.49; H, 11.65%.

The diethyl ether fraction gave crystals, mp 219° (0.6 g), a mixture from which bauerenol was isolated.

Isolation of bauerenol.—The mixture (84.3 mg) was dissolved in a mixture of pyridine (3 ml) and acetic anhydride (2 ml), heated under reflux for 1 h, and then kept standing at room temperature for 60 h. Workup gave crystals (84.8 mg), which gave, on recrystallization from chloroform-acetone (1:1), colorless needles, mp 281.8°, of bauerenyl acetate, $[\alpha]^{22}_D = -1.4^\circ$ (c = 0.70, CHCl₃) (Lit.: mp 280-282°, $[\alpha]^{20}_D \pm 0^\circ$; mp 293-294°, $[\alpha]_D - 3.7^\circ$; mp 284-287°, $[\alpha]^{20}_D + 1.9^\circ$), identified by comparison of the ir and pmr spectral data reported in the literature (3,4,10,13). The acetate (106 mg) was dissolved in a mixture of benzene (5 ml) and methanol (4 ml) containing sodium hydroxide (10%), and heated under reflux for 6 h. Work-up gave crystals (80 mg), recrystallization of which from acetone gave colorless needles, mp 205.8°, bauerenol, $[\alpha]^{22}_D - 25.9^\circ$ (c = 0.72, CHCl₃) (Lit.: mp 207-208°, $[\alpha]_D - 30^\circ$; mp 204°; mp 206-207°, $[\alpha]_D - 25^\circ$), identified by comparison of the ir, pmr, ms, and uv spectral data reported in the literature (3,4,10-13). The lanthanide shift of the methyl group in the pmr spectrum (CDCl₃) was in the order: 24 > 23 > 25 > 27 > 26 ≈ 28 > 29 ≈ 30.

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¹Full details of the isolation and identification of the compounds are available on request to the senior author.

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CONTRIBUTION TO THE STUDY OF *HIRSCHFELDIA INCANA*. SECOND COMMUNICATION

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Following a previous communication (1) on *Hirschfeldia incana*, we report here, mainly, the isolation of three polyphenolic heterosides.

EXPERIMENTAL¹

PLANT MATERIAL.—Aerial parts of flowered *Hirschfeldia incana* L. (Cruciferae) were collected in May in Traghana (Greece). Voucher of the plant has been deposited at the Botanical Museum of the University of Athens.

METHODS AND RESULTS.—Dried plant material (300 g) was successively extracted with petroleum ether and ethanol 95%. *Petroleum ether extract.* β -Sitosterol was isolated and identified (2); fatty acid composition, determined by gc of methyl esters, indicated presence of myristic, palmitic, oleic, linoleic and linoleic acids (3). *Ethanol extract.* Isolation by preparative pc of quercetin-3,7-0-diglucoside 65 mg, kaempferol-3-0-glucoside 35 mg and caffeic acid rhamno-glucoside 55 mg (4). Polyphenolic heterosides were identified by standard methods (1): pc, color reactions, spectral data (4,5), hydrolysis (aglycones were identified by the same methods and by authentic sample comparison).

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¹Full details of the isolation and identification of the compounds are available on request to the authors.