ISORHAMNETIN 7-GLUCOSIDE FROM CNICUS WALLICHI

K. N. SINGH and V. B. PANDEY

Department of Medicinal Chemistry, Institute of Medical Sciences, Banaras Hindu University, Varanasi 221005, India

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Key Word Index-Cnicus wallichi; Compositae; isorhamnetin 7-glucoside; flavonol glycoside.

Abstract—Isorhamnetin 7-glucoside was characterized from Cnicus wallichi. This is the first report of this glycoside in the Compositae and the second in nature.

As part of a chemical investigation of Indian medicinal plants we now report the characterization of isorhamnetin 7-glucoside (1) from *Cnicus wallichi* [1, 2]. This is the first record of 1 in the Compositae although it has been identified previously in six *Coprosma* species (Rubiaceae) [3]; however, mass spectral and ¹H NMR data were not given by that author.

Compound 1 was obtained from a methanolic extract of the whole plant of C. wallichi by column chromatography. Acid hydrolysis gave isorhamnetin and glucose, and the absence of a sodium acetate shift in the UV spectral analysis indicated that the glucose was at the 7position. This evidence, together with the mass spectral and ¹H NMR data given in the Experimental, confirms the characterization of 1 as isorhamnetin 7-glucoside.

EXPERIMENTAL

Whole plants of *Cnicus wallichi* Hook. F. were collected from Faizabad District, U.P., India. Plant material was identified by Professor S. K. Roy, Department of Botany, Banaras Hindu University, and a voucher specimen has been deposited in that department.

Dried, powdered whole plant of C. wallichi (1.5 kg) was defatted and then extracted in a Soxhlet apparatus with MeOH. The MeOH extract was chromatographed over silica gel and eluted with solvents of increasing polarity. The EtOAc-MeOH (1:3) eluant, on crystallization with MeOH, furnished yellow granules, mp 218-220° ([M]⁺, 478); IR v_{max}^{Navi} cm⁻¹: 3300-3400 (OH), 1650 (C=O), 1595; UV λ_{meX}^{MeOH} nm: 253, 267 sh, 306 sh, 355; $\lambda_{max}^{MeOH-NaOMe}$ nm: 240 sh, 273, 290 sh, 405; $\lambda_{max}^{MeOH-NaOMe}$ nm: 240 sh, 273, 290 sh, 405; $\lambda_{max}^{MeOH-NaOMe}$ nm: 253, 267 sh, 304, 355; $\lambda_{max}^{MeOH+NaOAe}$ nm: 253, 270 sh, 306 sh, 355. ¹ H NMR (90 MHz, DMSO-d_6) showed peaks at δ 6.46, 6.82 (1H, d, J = 2 Hz each, H-6 and H-8), 6.94 (1H, d, J = 2 Hz, H-2'), 3.87 (3H, s, -OMe), 9.86, 12.66 (3H, br s, exchangeable with D₂O, 3 × OH), 5.1 (1 H, br s, anomeric H

of glucose), 3.11–3.66 (6H of glucose). MS m/z: 478 [M]⁺, 316 (base peak), 302, 287, 245, 153, 137, 85, 73, 60. Heptaacetate, mp 147–148°; ¹H NMR (90 MHz, CDCl₃): δ 1.98, 2.03, 2.15, 2.17, 2.19, 2.36, 2.43 (3H, s each, 7 × OAc), 4.00 (3H, s, –OMe), 3.69–5.29 (glucosyl-H), 6.88, 6.98 (1H, d, J = 2 Hz each, H-6 and H-8), 7.15 (1H, d, J = 8 Hz, H-5'), 7.58 (1H, dd, J = 2, 8 Hz, H-6'), 7.76 (1H, d, J = 2 Hz, H-2').

Hydrolysis of 1. On acid hydrolysis with 6 % HCl (4 hr), 1 gave glucose (co-PC, *n*-BuOH-HOAc-H₂O, 4:1:5) and an aglycone, mp 301-305°, $C_{16}H_{12}O_7$ ([M]⁺, 316) identified as isorhamnetin by comparison of IR, UV, NMR and MS with lit. values [4, 5]. The aglycone gave a pentamethyl ether, mp 150-151°, which was identical to an authentic sample of quercetin pentamethyl ether and a tetraacetate, mp 200-202° (isorhamnetin tetraacetate; lit. [6], mp 202-204°).

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