A HYDROXYCOUMARIN ARSCOTIN FROM Artemisia scotina

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We have continued the separation of the combined lactones from Artemisia scotina Nevski [1] by chromatography on neutral alumina (activity grade III). Elution with benzene and with benzene–methanol (9 : 1 and 8 : 2) has given a substance with the composition $C_{11}H_{10}O_5$, mp 192–193°C (methanol), R_f 0.16 [benzene–methanol (9 : 1) system; Silufol], mol. wt. 222, which we have called arscotin.

IR spectrum, ν_{max}, cm^{-1}: 3350 (OH), 1710 (C=O of a lactone), 1610, 1575, 1500 (aromatic ring).

UV spectrum: λ_{max} 258, 312 nm (log ε 3.63, 3.95), which is characteristic for trisubstituted hydroxycoumarins.

The NMR spectrum (taken on a JNM 4H-100/100 MHz instrument in CF_3COOH, chemical shifts given on the δ scale from the signal of HMDS taken as 0) has two three-proton singlets at 3.58 and 3.55 ppm ($Ar-OCH_3$); two one-proton doublets at 7.52 and 6.12 ppm with J = 9.8 Hz (protons at C_4 and C_3); and a singlet at 6.34 ppm (proton at C_8).

Acetic anhydride in pyridine formed acetylarscotin with mp 145–146°C [benzene–hexane (1 : 1)], R_f 0.5 [benzene–methanol (9 : 1)]. The action of dimethyl sulfate on arscotin in absolute acetone with the addition of potassium carbonate gave a methyl ether with mp 100–101°C [benzene–hexane (1 : 1)], R_f 0.77 [benzene–methanol (9 : 1)].

The NMR spectrum of acetylarscotin showed a new three-proton singlet at 2.22 ppm, and the other signals had shifted downfield. The three-proton singlets were shifted by 0.09 and 0.04 ppm, the signals of the protons at C_3 and C_4 by 0.15 and 0.17 ppm, and the one-proton singlet by 0.42 ppm. The latter circumstance shows the presence of a free proton adjacent to the OH group. The shift of the signals of the protons at C_3 and C_4 shows their para arrangement relative to the acetyl group, i.e., the OH is present at C_7, position C_8 is free, and the methoxy groups are therefore located at C_5 and C_6.

Because of the more acidic nature of this hydroxyl, in the UV spectrum with sodium acetate there is a bathochromic shift from λ_{max} 258 to 287 nm and from λ_{max} 312 to 331 nm (log ε 3.63, 3.76, 3.95, and 3.17, respectively).

On the basis of the results obtained, it is most probable that arscotin has the structure of 7-hydroxy-5,6-dimethoxycoumarin.

LITERATURE CITED

1. M. I. Yusupov and G. P. Sidyakin, Khim. Prirodn. Soedin., 667 (1972).

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