7-OXOBAICALINE — A NEW ALKALOID FROM Thalictrum baicalense

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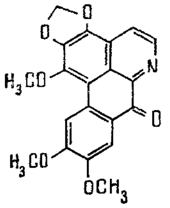
We have investigated the alkaloid composition of the stems of Thalictrum baicalense Turcz., collected on June 25, 1982 in the Skovorodino region of the Amur province. When a concentrated chloroform extract was treated with 10% sulfuric acid, berberine sulfate (I) (0.043% on the weight of the air-dry stems) was obtained. The filtrate after the separation of (I) was washed with benzene and with ether and was then made alkaline with concentrated ammonia solution and the bases were extracted with chloroform. The combined bases (0.13% on the weight of the stems) were separated into phenolic and nonphenolic fractions (A and B). The chromatography of fraction B on a column of silica gel (with elution by chloroform and chloroform—methanol gave glaucine, baicaline [1], and base (II), and fraction A gave thalbaicaline [2]. Base (II) crystallized from methanol in the form of large needles with mp 240°C (decomp.).

The UV spectrum of (II) — $\lambda_{\max}^{\text{ethanol}}$ 250, 289, 380, 500 nm — did no change in an alkaline medium. A bathochromic shift took place in an acid medium. The IR spectrum (KBr) had the band of a conjugated carbonyl group with $\nu_{\max}$ 1650 cm^{-1}. These facts permitted (II) to be assigned to the oxoaporphine alkaloids.

The PMR spectrum of (I) CF_3COOH, δ scale, ppm, HMDS = 0) contained singlets at 3.73 (6 H), 3.80 (3 H), ($3 \times OCH_3$), and 6.18 (2 H, CH_2O_2). Two one-proton singlets at 7.60 and 8.45 were due to protons at C-8 and C-11, respectively. The signals from H-4 and H-5 were observed in the 8.20-8.40 ppm region. The mass spectra of (II) had the peaks of ions with m/z 365 (M^+, 100%) 350, 349, 336, 320, 307, 279, 223, and 185.5^{2+}. The facts presented, and also its common presence in the plant with baicaline permitted the structure of 7-oxobaicaline to be proposed for (II). The reduction of (II) with zinc and sulfuric acid gave (±)-baicaline. The oxidation of baicaline with chromium trioxide and potassium permanganate gave 7-oxobaicaline, identical with (II) in coloration, R_f value, melting point, and IR and PMR spectra.

Thus, the stems of T. baicalense, not investigated previously, have yielded berberine, glaucine, baicaline, thalbaicaline, and the new base 7-oxobaicaline.



II

LITERATURE CITED

1. S. Kh. Maekh, S. Yu. Yunusov, É. V. Boiko, and V. M. Starchenko, Khim. Prir. Soedin., 227 (1982).
2. S. Kh. Maekh, S. Yu. Yunusov, É. V. Boiko, and V. M. Starchenko, Khim. Prir. Soedin., 537 (1983).

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