Y-PYRONE DERIVATIVES FROM Halenia corniculata

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From 500 g of the epigeal part of *Halenia corniculata* (L.) Cornaz., collected in the Pribaikal region of the Buryat ASSR in July 1975, by extraction with 70% ethanol followed by treatment with chloroform we have isolated 32.7 g of extractive substances. Chromatography of the chloroform extract on silica gel with elution by chloroform yielded substances (1) and (2) of xanthone nature, and chloroform—ethanol (9:1) yielded luteolin, which was identified by its UV, mass, and PMR spectra and by comparison with an authentic sample [1].

Substance (1) was the predominating component and consisted of bright yellow acicular crystals with the composition $C_{17}H_{16}O_7$, M⁺ 332, mp 145-147°C. UV spectrum (nm); MeOH: 243, 261, 273 infl., 313, 380; AlCl₃: 243, 274, 278 sh, 327, 440. The PMR spectrum of substance (1) (0 - TMS, CDCl₃, 100 MHz) showed the signals of three aromatic protons of ring B at (ppm) 7.8 (dd, 1 H, J = 2.5 Hz, J = 9 Hz, H-8) and 7.28 (m, 2 H, H-6, H-7). There were the signals of four methoxy groups: 3.96 (s, 3 H), 4.02 (s, 6 H), and 4.16 (s, 3 H). The PMR spectrum of the acetate (I) contained a three-proton signal at 2.54 ppm of one acetyl group, showing the presence of an OH group in position 1 of the xanthone nucleus, which was confirmed by the appearance in the UV spectrum with AlCl₃ of bathochromic shifts of the short-wave and long-wave maxima [2].

From its melting point and its UV, PMR, and mass spectra we identified substance (1) as 1-hydroxy-2,3,4,5-tetramethoxyxanthone, which has been isolated previously from species of *Frasera* [3, 4] and *Swertia* [5], and from *Halenia asclepidea* [6].

Substance (2) formed light yellow crystals with mp 191-192°C, M⁺ 302; empirical formula $C_{16}II_4O_6$. UV spectrum of the substance (nm): MeOH: 220, 244, 253 sh, 264 sh, 272, 306, 366; AlCl₃: 220, 244, 266, 283, 339, 420. The UV spectra with AcONa and with AcONa + H₃BO₃ were similar to those of substance (3).

Analysis of the PMR spectrum of substance (3) showed the presence of the signals of four aromatic protons as (ppm): 6.58 (s, 1 H, H-4); 7.28 (m, 2 H, H-6, H-7); and 7.83 (dd, 1 H, J = 2.5 Hz, J = 9 Hz) and the signals of three methoxy groups (3.90, 3.94, and 4.00 ppm (s, 3 H each)).

According to its melting point and mass, UV, and PMR spectra, substance (2) was identical with 1-hydroxy-2,3,5-trimethoxyxanthone [6, 7].

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