ISOLATION OF APOHYOSCINE AND OF 6-HYDROXYATROPINE FROM *Physochlaina alaica*

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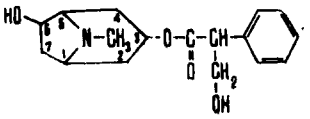
From the roots of Physochlaina alaica E. Korot. collected in the period of the withering of the epigeal part, in addition to compounds found previously [1], we have isolated two alkaloids.

The alkaloid (I), $C_{17}H_{19}NO_3$, has mp 79-80°C (ether), M^+ 285. Its IR spectrum shows absorption bands at 705 and 780 cm^{-1} (monosubstituted benzene ring), 1700 cm^{-1} (conjugated ester carbonyl group), and 1610 cm^{-1} ($>C=C<$). The UV spectrum of the alkaloid has a broad band at λ 240-262 nm like the spectra of hyoscyamine [2]. In the NMR spectrum of (I) (in $CDCl_3$, δ scale, JNM 100/100 MHz, HMDS as internal standard) there is a singlet at 2.40 ppm (3 H, $N-CH_3$), two one-proton doublets at 5.65 and 6.15 ppm ($C=CH_2$), a singlet at 7.22 ppm (5 H, monosubstituted benzene ring), a triplet at 4.95 ppm (C_3H) and a singlet at 3.17 ppm (C_6H and C_7H). The molecular weight of (I) determined mass-spectrometrically differs from that of hyoscine by 18 m/e. The facts given above, and also the absence of a depression of the melting point of a mixture with an authentic sample shows that the base isolated is apohyoscine [3].

The alkaloid (II), $C_{17}H_{23}NO_4$, has mp 68-69°C (carbon tetrachloride), $[\alpha]_D^{20}$ 0° (c 1.9; methanol), M^+ 305. The IR spectrum of (II) has absorption bands at 710 and 745 cm^{-1} (monosubstituted benzene ring), 1730 cm^{-1} (ester carbonyl group) and 3320-3480 cm^{-1} (broad) (hydroxy group). The UV spectrum has a broad band at λ 250-270 nm similar to the spectra of the tropane alkaloids [2]. The NMR spectrum (in CF_3COOH, δ scale) has signals at 2.73 ppm (3 H, singlet, $N-CH_3$), 4.82 ppm (C_3H, triplet), 1.45-2.35 ppm (6 H, protons at C_2, C_4, and C_7), 2.65-2.95 ppm (2 H, protons at C_1 and C_5), 4.50 ppm (C_6H), 3.50-4.20 ppm (5 H, $>CH-CH_2OH$, $-OH$ at C_6), and 6.99 ppm (5 H, singlet, monosubstituted benzene ring). The mass spectrum of (II) has peaks of ions with m/e 305 (M^+), 261, 156, 140, 96, 95, 94, 83, 82, 81, which are characteristic for alkaloids of the tropane group [4].

The results of a comparison of the spectral characteristics of the alkaloid and of 6-hydroxyhyoscyamine, the production of a diacetyl derivative (III) (NMR spectrum: 1.93 ppm, 6 H, singlet, CCl_4), and also the formation of tropane-3α,6β-diol and of d,*l*-tropic acid [5] on its hydrolysis showed that the new base has the structure of 6-hydroxyatropine.



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