SCOPOLAMINE AND BELLADONNINE FROM Physochlaina alaica

R. T. Mirzamatov, K. L. Lutfullin,
V. M. Malikov, and S. Yu. Yunusov

UDC 547.944

Continuing an investigation of the alkaloids of Physochlaina alaica E. Korot., by chloroform extraction of roots collected in the fruit-bearing phase in the basin of the R. Shakhimardan, northern slopes of the Alai range, we have isolated 1.4% of combined alkaloids. By separating the mixture of bases according to their basicities and by column chromatography on alumina, in addition to those mentioned previously [1, 2], we have obtained three alkaloids.

An alkaloid $C_{17}H_{21}NO_4$ (I) formed a syrupy liquid $[\alpha]_D^{20}$ −25.6° (c 2.0; chloroform), mol. wt. 303 (mass spectrometrically). From its IR, UV, and NMR, and mass spectra and some chemical properties this alkaloid was identified as *l*-scopolamine [3].

An alkaloid $C_{34}H_{42}N_2O_4$ (II) formed an amorphous substance with mol. wt. 542 (mass spectrometrically). The IR spectrum of the base showed absorption bands at 710 and 770 cm^{-1} (mono- and 1,2-disubstituted benzene ring) and 1725 cm^{-1} (ester carbonyl group). The UV spectrum of (II) showed absorption at 254, 259, and 263 nm (log ε 2.63, 2.67, 2.66), which is characteristic for a benzene ring, as in the spectrum of hyoscyamine. The molecular weight of (II) (M^+ 542 − 65%) found mass spectrometrically corresponds to that for the dimer of apoatropine. The mass spectrum of (II) had, apart from M^+ 542, peaks of ions with m/e 461 (6%), 375 (44%) and 271 (18%), 140 (30%), 124 (100%), 96 (35%), 95 (30%), 94 (40%), which are characteristic of alkaloids of the tropane group [4]. In the NMR spectrum of the base (JNM-4H 100/100 in CCl_4 with HMDS as internal standard) there are two signals in the form of singlets at δ 2.06 ppm and 2.12 ppm due to the protons of two $N-CH_3$ groups, a two-proton multiplet with its center at δ 4.97 ppm (protons at C_3 and C_3'), a one-proton triplet at δ 3.70 ppm from the proton of a methine group, and a four-proton signal at δ 2.74-2.94 ppm (protons at C_1, C_1', C_5, and C_5'). The signals of the aromatic protons are distributed over a broadened signal at δ 7.09 ppm (7H) and a quartet at δ 6.85 ppm (2H), J_{ortho} = 8.0 Hz and J_{meta} = 1.5 Hz.

The above facts permitted the assumption that (II) probably has the structure of β-belladonnine [5]. To confirm this, we obtained β-belladonnine from hyoscyamine. The results of a comparison of the IR, UV, and mass spectra, and also some chemical properties of (II), which we have isolated from this plant for the first time, and of synthetic β-belladonnine showed their identity.

An alkaloid $C_{34}H_{42}N_2O_4$ (III), with mp 128-129°C (ethyl acetate), mol. wt. 542 (mass spectrometrically). The base has the same composition and molecular weight as β-belladonnine and similar IR, UV, and mass spectra. In the NMR spectrum of (III), the signals of the aromatic protons appear in the form of a singlet which is broadened in the base (9H) at δ 7.10 ppm, and the signals of the other protons are close to those for β-belladonnine. What has been said above shows that (III) is apparently α-belladonnine [5-9]. This was confirmed by the results of a comparison of the IR, UV, mass, and NMR spectra, and also by the melting point of a mixture of the base with α-belladonnine, which we obtained from hyoscyamine by a known method [9].

Institute of the Chemistry of Plant Substances, Academy of Sciences of the UzbekSSR. M. I. Kalinin Andizhan State Medical Institute. Translated from Khimiya Prirodnykh Soedinenii, No. 5, p. 680, September-October, 1973. Original article submitted February 27, 1973.

LITERATURE CITED

1. S. Yu. Yunusov and N. V. Plekhanova, Dokl. Akad. Nauk UzbSSR, No. 9, 26 (1952).
2. R. T. Mirzamatov, V. M. Malikov, K. L. Lutfullin, and S. Yu. Yunusov, Khim. Prirodn. Soedin., 493 (1972).
3. N. Mandava and G. Fodor, Can. J. Chem., 46, 2761 (1968).
4. E. C. Blossey, H. Budzikiewicz, M. Ohashi, G. Fodor, and C. Djerassi, Tetrahedron, 20, 3, 585 (1964).
5. Volgländer, Rosenberg, Arch. Pharm., 292, 632 (1959).
6. Hübschman, Jahresb., 376 (1858).
7. K. Kraut, Ber., 13, 165 (1858).
8. J. Merling, Ber., 17, 381 (1884).
9. Küssner, Arch. Pharm., 276, 617 (1938).