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O-METHYLCYCLOVIROBUXINE-D — A NEW ALKALOID FROM *Buxus sempervirens*

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UDC 547.944/945

As a result of the further study of the alkaloids of *Buxus sempervirens* L. cultivated in the environs of the town of Kobuleti, Adzhar ASSR [1] we have isolated a new alkaloid, which we have called O-cyclomethylvirobuxine-D, with the composition $C_{27}H_{48}N_2O$ (I), mp 231-233°C (ethanol), $[\alpha]_D$ + 83.52° (s 0.903; chloroform).

The IR spectrum of (I) showed absorption bands at 3045 and 1452 cm^{-1} (methylene of a cyclopropane ring) [2] and 2860 and 1275 cm^{-1} (methoxy group). Its NMR spectrum showed signals in the form of two three-proton singlets and a six-proton singlet at 0.69, 1.01 and 0.90 ppm, respectively, from four C—CH_3 groups, a six-proton singlet at 2.34 ppm from two N—CH_3 groups, a singlet at 3.45 ppm from a methoxy group, and a doublet at 1.04 ppm (J = 6 Hz) from a C—CH_3 group. The mass spectrum of alkaloid (I) had the main peaks of ions with m/z 56, 57, 58, (100%), 314, 371, 386, 402, and 416 (M^+).

The m/z value of 58 for the maximum ion peak in the mass spectra confirmed the presence of a methylamine group in the C_{20} position of a pregnane nucleus [3, 4].

When (I) was acetylated with acetic anhydride in pyridine, N,N'-diacetyl-O-methylcyclovirobuxine-D was formed with the composition $C_{31}H_{52}N_2O_3$ (II), mp 242-244°C (acetone—petroleum ether (1:6)), $[\alpha]_D$ −25.51° (s 0.708; chloroform). The IR spectrum of (II) showed an absorption band at 1632 cm^{-1} (N-acetyl group) and its mass spectrum had the main peaks of ions with m/z 57, 58, 100 (100%), 314, 368, 380, 386, 456, 499, and 500 (M^+). This confirmed the formation of a N,N'-diacetyl derivative when (I) was acetylated.

The Hess methylation of (I) gave a N,N'-dimethyl derivative with the composition $C_{29}H_{52}N_2O$ (III), mp 253-255°C (ethanol) $[\alpha]_D$ + 65.18° (s 0.522; chloroform) the IR spectrum of which lacked the absorption band for an NH group. The mass spectrum showed the main peaks of ions with m/z 70, 71, 72 (100%), 84, 342, 358, 370, 399, 414, and 444 (M^+), indicating the complete methylation of the amino groups in (I) [3, 5].

According to a mixed melting point, and also the features of its IR and mass spectra, the N,N'-dimethyl derivative (III) was identical with N,N',O-trimethylcyclovirobuxine-D [6, 7], which was obtained by Kuhn's method.

Consequently, O-methylcyclovirobuxine-D (I) has the structure and configuration of 16α-methoxy-4,4',14α-trimethyl-3β,20α-di(methylamino)-9β,19-cyclo-5α-pregnane.

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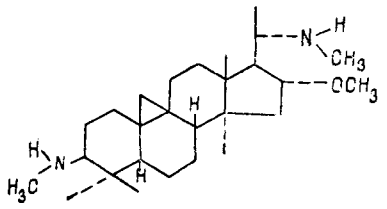

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14-ACETYLKARAKOLINE — A NEW ALKALOID FROM *Delphinium confusum*

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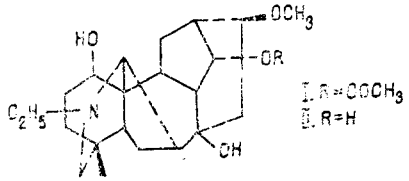
From the epigeal part of *Delphinium confusum* M. Pop., collected in the flowering period in the upper reaches of the R. Talas, in addition to the condelphine isolated previously [1], we have obtained virescenine, 14-acetylvirescenine [2], and a new base (I) with the composition $C_{23}H_{33}NO_6$, M^+ 419, mp 99-100°C (from acetone). Its IR spectrum had the absorption bands of hydroxy groups at 3460 and 3620 cm^{-1}, of ether bonds at 1100 cm^{-1}, and of an ester carbonyl at 1743 cm^{-1}. According to its PMR spectrum, the alkaloid contained a tertiary C-methyl group (three-proton singlet at 0.83 ppm), an N-methyl group (three-proton triplet with J = 7 Hz at 1.06 ppm), a methoxy group (three-proton singlet at 3.22 ppm), and an acetyl group (three-proton singlet at 2.00 ppm).

The mass spectrum of the alkaloid was characteristic for the C_{19}-diterpene alkaloids and had as its maximum peak that of the $(M - OH)^+$ ion, which showed the presence of an α-hydroxy group at C-1 [3]. This was also confirmed by the presence in the mass spectrum of a peak of medium intensity due to the $(M - 56)^+$ ion [4]. A one-proton signal at 4.82 ppm in the form of a triplet with J = 4.5 Hz was connected with the presence of an acetoxy group at C-14 [5]. The facts given make it possible to assume for the alkaloid the structure of 14-acetylkarakoline.

In actual fact, when (I) was saponified a base was obtained which, according to TLC, a mixed melting point, and IR spectroscopy was identical with an authentic sample of karakoline (II) [6].

Thus, the alkaloid that we have isolated is 14-acetylkarakoline, which has not been described in the literature.



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