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ISODIHYDROCYCLOMICROPHYLLINE A FROM *Buxus sempervirens*

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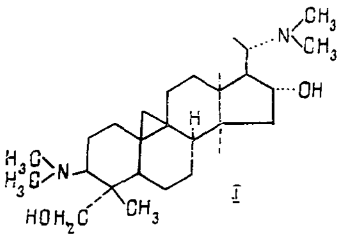
As the result of a further study of the alkaloids of *Buxus sempervirens* L. cultivated in the environs of Tashkent [1] we have isolated a new alkaloid, which we have called isodihydrocyclomicrophylline A, composition $C_{28}H_{50}O_2N_2$ (I), mp 215-217°C (ethanol) $[\alpha]_D$ −64.27°C (c 0.527; chloroform).

The IR spectrum of (I) showed characteristic absorption bands at 3368, 3047, and 1452 cm^{-1} (methylene of a cyclopropane ring and active hydrogens).

In the NMR spectrum of (I), signals of protons were observed in the form of nine-proton singlets at 1.15, 1.06, and 0.92 ppm, respectively, from three $-C-CH_3$ groups [sic], a twelve-proton singlet at 2.32 and 2.18 ppm from two N-dimethyl groups [sic], a three-proton doublet a 0.97 ppm (J = 7.52 Hz) from a $C-CH_3$ group, and a one-proton multiplet at 4.09 ppm from a methine proton geminal to a hydroxy group, $-C\underline{H}-OH$, which are characteristic for the 16β-proton of a series of *Buxus* alkaloids, and in the form of a two-proton quartet at 3.25 and 3.02 ppm from a $-CH_2-OH$ group [sic].



I

The mass spectrum of the alkaloid (I) had the main peaks of ions with m/z 57, 58, 70, 71, 72 (100%), 84, 98, 342, 383, 424 and 466 (M^+).

The maximum peak of the ion with m/z 72 in the mass spectrum arose as a result of the cleavage of the bond between C_{17} and C_{20} and confirmed the presence of a dimethylamino group in the C_{20} position of the pregnane nucleus [2, 3].

The acetylation of (I) with acetic anhydride in pyridine formed O,O′-diacetylisodihydrocyclomicrophylline A with the composition $C_{32}H_{54}O_4N_2$ (II), mp 187−189°C (acetone), $[\alpha]_D$ −89.61° (c 0.905; chloroform).

In the IR spectrum of (II)the absorption bands of active hydrogen had disappeared and absoption bands had appeared at 1743 cm^{-1} (O-acetyl).

The NMR spectrum of (II) had resonance signals at (ppm) 0.86; 1.02; 1.13 (s, 9H, $-C-CH_3$); 0.94 (d, 3H, $C-CH_3$; J = 6.51 Hz), 2.24; 2.06 (s, 12H, $2(CH_3)_2$); 5.14 (m, 1H, −HC−O-acetyl), 3.49 and 3.32 (quartet, 2H, with its center at 3.40; J = 11.82 Hz, $-H_2CO$-acetyl).

The main peaks in the mass spectrum of (II) had m/z 70, 71, 72 (100%), 84, 371, 386, 442, 512, 530 (M^+).

A comparison of the NMR results and the mass spectra of the alkaloid (I) and its acetyl derivative (II) with those of dihydromicrophylline A led to the structure of dihydrocyclomicrophylline. However, (I) and (II) differed sharply in the magnitude and sign of their specific rotations and in their melting points from dihydrocyclomicrophylline and its acetate, and the chemical shits of the protons of the secondary and tertiary methyl groups also differed. On the basis ofwhat has been said above, alkaloid (I) may be an iso product of dihydrocyclomicrophylline A, probably at one of the asymmetric centers C-4 and C-20.

Thus, it may be concluded that (I) differs from dihydrocyclomicrophylline A [4] and has the most probable structure and configuration 3β,20α-bisdimethylamino-16α-hydroxy-4α-hydroxymethyl-4β,14α-dimethyl-9β,198-cyclo-5α-pregnane.

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