

INVESTIGATION OF THE ALKALOIDS
OF *Buxus sempervirens*

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In the present paper we give the results of a further study of the alkaloids of *B. sempervirens* L. cultivated in Tashkent [1].

The combined alkaloids, after the separation of the cyclobuxine-D [1, 2] were dissolved in benzene and separated according to their basicities by means of McIlvaine buffer solutions at pH 8.0-2.2 (in steps of pH 0.2). The fractions with pH 6.6-6.0, 7.2-6.8, and 8.0-7.4 were chromatographed on alumina (separately) and were eluted with ether-ethanol. Three bases were isolated: with mp 198-200°C (ethanol) $[\alpha]_D + 98.88^\circ$ (I); with mp 135-137°C (acetone); $[\alpha]_D + 71.4^\circ$ (II); and with mp 219-221°C (ethanol), $[\alpha]_D + 66.1^\circ$ (III).

Alkaloid (I), $C_{28}H_{50}N_2$, 414 (M^+), IR spectrum: ν_{max} 3050, 1460 cm^{-1} (methylene of a cyclopropane ring). The NMR spectrum had singlets at (δ scale) 0.75 and 0.88 ppm (6 H, $2CH_3$), 0.92 ppm (6 H, $2CH_3$), and 2.14 and 2.26 ppm [$2N(CH_3)_2$] and a doublet at 0.88 ppm (3 H, CH_3).

Alkaloid (II), $C_{26}H_{46}N_2$, 386 (M^+), IR spectrum: ν_{max} 3050, 1460 cm^{-1} (methylene of a cyclopropane ring). The NMR spectrum had singlets at 0.69, 0.85, 0.91, and 0.94 ppm (12 H, CH_3), 2.38 ppm [$2N(CH_3)_2$], and a doublet at 0.88 ppm (3 H, CH_3). The Hess methylation of (II) gave a N,N' -dimethyl derivative with mp 198-200°C, identical with the alkaloid (I) in melting point and IR and mass spectra.

In its physicochemical constants, the alkaloid (I) is close to the known alkaloid cycloprotobuxine A (IV) [3], and the alkaloid (II) to cycloprotobuxine D (V) [4]. We were unable to check the identity of the alkaloids (I) and (II) by direct comparison with the alkaloids (IV) and (V) because we had no authentic samples.

Alkaloid (III), $C_{26}H_{46}N_2O$, 402 (M^+), IR spectrum: ν_{max} 3305, 3150 (OH, NH), 3030, 1460 cm^{-1} (methylene of a cyclopropane ring). The NMR spectrum showed a multiplet at 4.04 ppm (1 H, $CH-OH$), singlets at 2.36 ppm [6 H, $2N(CH_3)_2$], 0.67 and 1.03 ppm (6 H, $2CH_3$), and 0.89 ppm (6 H, $2CH_3$), and a doublet at 1.02 ppm (3 H, CH_3).

The base (III) formed an N,N',O -triacetate with mp 236-238°C, $[\alpha]_D - 68.29^\circ$, 528 (M^+). IR spectrum: ν_{max} 1740 cm^{-1} (O-acetyl) and 1630 cm^{-1} (N-acetyl).

The Hess methylation of (III) gave an N,N' -dimethyl derivative with mp 243-245°C, $[\alpha]_D + 42.5^\circ$, 430 (M^+).

The specific rotations of all the substances were determined in chloroform. The constants given above, and also the features of the IR, NMR, and mass spectra of the base (III) and its derivatives agreed well with those for a known alkaloid-cyclovirobuxine D [5-10].

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