ALKALOIDS OF Buxus sempervirens

B. U. Khodzhaev, R. Shakirov,
and S. Yu. Yunusov

UDC 547.944/945

The alkaloids of Buxus sempervirens L. (common box) cultivated in the USSR have not been studied. We have begun an investigation of the alkaloids of this plant. The alkaloid content in various organs according to the growth site was determined by the chloroform method (Table 1).

The fraction of the combined alkaloids soluble in benzene (171.8 g), isolated from 30 kg of thin flowers and leaves (collected on April 28, 1969) was dissolved in 20% acetic acid and the solution was made alkaline with a saturated solution of sodium carbonate, and was extracted with ether (yield 131.4 g) and chloroform (yield 40 g). When the ethereal residue was treated with acetone, 6 g of a base (I) was isolated with the composition $C_{25}H_{42}N_2O$, mp 235-237° C (from ethanol), $[\alpha]_D + 96.1°$ (c 0.728; chloroform) and R_f 0.25. The IR spectrum of (I) exhibited absorption bands at (cm^{-1}) 3310, 3150 (OH, NH), 2930 (CH_2, CH_3), 1650, 910 (terminal CH_2), 3042, and 1463 (methylene of a cyclopropane ring). The NMR spectrum (δ scale) showed doublets at 4.75 and 4.52 (2H, terminal CH_2; $J < 1$ Hz), a multiplet at 4.03 (1H, $-CH_2-\overset{\overset{OH}{|}}{C}H-CH_2$), singlets at 2.43 and 2.38 (6H, $2N-CH_3$); and 1.06 and 0.91 (6H, $2 \equiv C-CH_3$), and a doublet at 1.02 (3H, $=CH-CH_3$, $J = 6$ Hz). The mass spectrum of (I) exhibits the main peaks of ions with m/e 58 (100%), 328 (11%), 356 (29%), 372 (31%) and 386 (20%) (M^+).

With acetic anhydride in pyridine, substance (I) formed a N,N',O-triacetate (II) with mp 249-251° C, $[\alpha]_D$ 0° (c 2.270; chloroform) and R_f 0.75. The NMR spectrum of (II) exhibited signals at 1.90, 1.95, and 2.03 ppm (3H, $-OCOCH_3$; 6H, $2N-COCH_3$); M^+ 512 (mass spectrometrically). The Hess methylation of (I) gave a N,N'-dimethyl derivative (III) with mp 198-200° C, $[\alpha]_D + 97.7°$ (c 0.522; chloroform) and R_f 0.45. The NMR spectrum of (III) had signals at 2.28 and 2.18 ppm [12H, $2N-(CH_3)_2$]; M^+ 414 (mass spectrometrically). When (I) was hydrogenated in glacial acetic acid according to Adams, the dihydro derivative of (I) (IV) was isolated, with mp 231-233° C $[\alpha]_D + 107.14°$ (c 0.56; chloroform), R_f 0.27.

TABLE 1

Collection site	Date of collection	Plant organs	Combined alkaloids, %
Tashkent	28. IV 1969	First-year stems	1,05
		Leaves and thin flowers	1,00
		Branches several years old	0,7
Village of Khumsan, Tashkent oblast	13. V 1970	Leaves and thin flowers	1,12
Kislovodsk	VIII 1970	The same	1,81
		Yellowed leaves and thin flowers	1,65
		Branches several years old	0,82
		Yellowed branches several years old	0,79

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR. Translated from Khimiya Prirodnykh Soedinenii, No. 4, pp. 542-543, July-August, 1971. Original article submitted March 31, 1971.

The melting points of (I) and (IV), the specific rotations of (II) and (IV), and the IR and NMR spectra of (I) differ slightly from the figures given in the literature [1-6]; nevertheless a mixture of (I) with cyclobuxine D gave no depression of the melting point. On TLC the two substances gave spots with the same R_f values. TLC was performed with silica gel and the butan-1-ol–acetic acid–water (10 : 1 : 3) system. Cyclobuxine D was kindly given to us by I. Tomko.

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