THE ALKALOID CYCLOPROTOBUXINE-C

FROM Buxus hyrcana. II

A. M. Aliev and G. M. Orazmuradov

We have previously reported the isolation and identification of buxtauine from the leaves of <u>Buxus</u> hyrcana Pojark [1]. In the present communication we give the results of a study of a second base.

The total alkaloids were obtained by chloroform extraction. The ether-soluble fraction was passed through a column of alumina (activity grade II). The benzene eluate (fractions 1-40) was repurified on a column and was eluted with petroleum ether. The residue after the elimination of the solvent was treated with acetone. This yielded a base with the composition $C_{27}H_{48}N_2$, mp 201-202.5°C (acetone), $[\alpha]_D^{20} + 76.3^\circ$ (c 0.27; chloroform), Rf 0.66 (TLC; here and below, alumina, activity grade II) the solvent system here and below being chloroform—ethanol (9:1); M⁺ 400.

IR spectrum, cm⁻¹: 1460 and 3060 [cyclopropane ring $(C_9 - C_{19})$] (here and below, UR-20 spectrometer,

paraffin oil). NMR spectrum, ppm: singlets at 0.76 $(-C-CH_3, 3H)$; doublet with its center at 0.82 ppm (=CH-CH₃, 3 H, J=6 Hz), 2.17 [N(CH₃)₂ at C₂₁]; 2.44 (NHCH₃ at C₃); 0.92 and 0.95 [=C (CH₃)₂ at C₄], one of these singlets (0.95) apparently being superposed on the signals of the CH₂ groups of the molecule (CDCl₃, HMDS). In the mass spectrum the main peaks had m/e 58 (12%), 70 (15%), 72 (100%), 84 (8%), 356 (17%), 385 (12%) and 400 (20%) (M⁺).

The peaks with m/e 58 and 72 show the presence in the molecule of a methylamine group (NHCH₃) at C₃ and of a [N(CH₃)₂] grouping at C₂₀.

With acetic anhydride in pyridine the base formed an acetyl derivative with mp 220-225°C (ether), Rf 0.90 (TLC). IR spectrum: λ_{max} 1650 cm⁻¹ (N-acetyl). The N-methyl derivative, obtained by means of formaldehyde and formic acid, had mp 203-205°C (acetone), $[\alpha]_D^{20}$ +30° (c 0.48; chloroform), Rf 0.83 (TLC).

On comparing the facts given above with the literature [2-13] it may be concluded that the base isolated is cycloprotobuxine-C ("alkaloid L"), this being the first time it has been obtained from plants of the flora of the Soviet Union. In view of the absence of an authentic sample, we were unable to perform identification by direct comparison.

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N. Narimanov Azerbaidzhan State Medical Institute. Institute of Chemistry, Academy of Sciences of the Turkmen SSR. Translated from Khimiya Prirodnykh Soedinenii, No. 6, pp. 808-809, November-December, 1974. Original article submitted October 10, 1973.

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