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ISOLATION AND STRUCTURE OF BUXAMINOL-G, AN ALKALOID FROM *BUXUS PAPILOSA*

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Key Word Index—*Buxus papilosa*; Buxaceae; steroidal alkaloids; buxaminol-G.

Abstract—A new alkaloid, buxaminol-G, has been isolated from the alcoholic extract of the leaves of *Buxus papilosa*, and its structure assigned on the basis of spectroscopic studies.

INTRODUCTION

The plants of the family Buxaceae have been used in the indigenous system of medicine as a febrifuge, for the relief of rheumatism and venereal diseases, and they also show antimalarial activity. *Buxus papilosa*, C. K. Schn, Linn. is found in the northern regions of Pakistan. In previous publications we have reported the isolation of several new alkaloids from the leaves of this plant [1–6].

RESULTS AND DISCUSSION

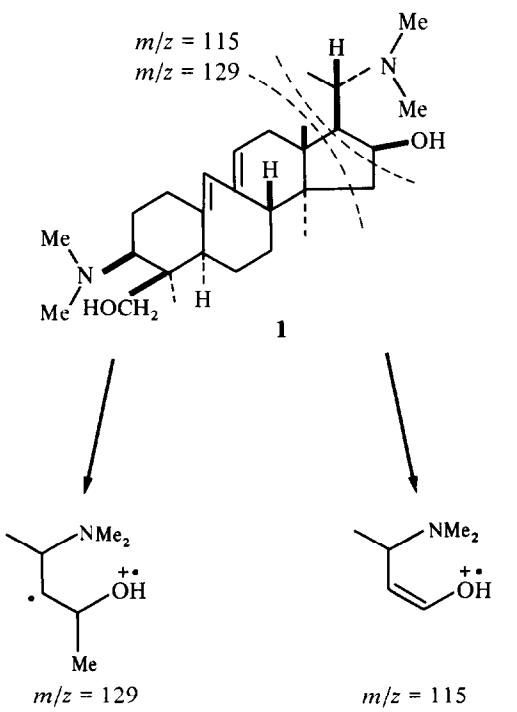
Buxaminol-G, $[\alpha]_D$ $CHCl_3$:83.33°, was isolated as a colourless amorphous solid. The IR spectrum of the substance showed bands at 1380 (C–N), 1550 (C=C) and 3550 cm^{-1} (–OH). The UV spectrum showed maxima at 237 nm (ε 6040), 245 nm (ε 5866) and at 253 nm (ε 4151), characteristic of the presence of a 9,(10 → 19) *abeo*-diene system [7]. The ^1H NMR spectrum showed singlets for three tertiary methyl groups at δ0.74, 0.87 and 0.90, a doublet at 0.72 (J = 5 Hz) for a secondary methyl group and two 6-proton singlets at 2.30 and 2.33 for the two $-NMe_2$ groups at C-3 and C-20 respectively. A set of AB doublets at δ3.52 and 3.84 (J_{AB} = 10 Hz) were assigned to the CH_2OH group at C-4. The presence of the hydroxyl group at C-30 in a β-disposition was deduced by comparison of chemical shifts and coupling constants with other similarly substituted alkaloids [7, 8]. A broad triplet at δ4.21 (1H, J = 7.2 Hz, $W_{1/2}$ = 16 Hz) was assigned to the C-16 proton. The chemical shift, coupling constant and half width of the multiplet is consistent with a tentative C-16β assignment of the hydroxyl group [9, 10]. A singlet at δ5.15 was assigned to the olefinic proton at C-19, and a multiplet at 5.45 to the C-11 olefinic proton.

The mass spectrum of the alkaloid showed the molecular ion peak at m/z 444.3709 in agreement with the formula $C_{28}H_{28}N_2O_2$ (calc. 444.3715). The fragmentation pattern confirmed the presence of a dimethylamino grouping at C-3 (peaks at m/z 58, 71, 84), whereas the peaks corresponding to $[M-15]^+$ and $[M-44]^+$ and the base peak at m/z 72 were indicative of a dimethylamino substituent at C-20 [11]. Peaks at m/z 115.0650 ($C_6H_{13}NO$) and 129.0901 ($C_7H_{15}NO$) indicated the presence of a hydroxyl group on ring D.

The peak at m/z 115 corresponding to the formula $C_6H_{13}NO$ formed by the cleavage of ring D was particularly significant since it established that the hydroxyl group was present on a six-carbon fragment at C-16. The position of the multiplet at δ4.21 was consistent with the known resonance position of the C-16 proton in other compounds bearing a hydroxyl group at this carbon [12] and was in accordance with the biogenetic trends in such compounds. The mass fragmentation pattern also clearly showed that there was no hydroxy group directly bonded to ring A. The fragmentation sequences were ascertained by a combination of high resolution mass spectrometery and linked scan measurements of ion fragmentation pathways. On the basis of the above spectroscopic data structure **1** is assigned to buxaminol-G.

EXPERIMENTAL

An authentic supply of *B. papilosa* was obtained from Gomal University, Baluchistan by courtesy of Prof. G. A. Miana. The



1

m/z = 129 *m/z* = 115

plant was identified by S. I. Ali, Prof. of Botany, University of Karachi.

The EtOH extract of the air-dried leaves (10 kg) of *Buxus papilosa* was evaporated under vacuum to afford a gum. This gum was taken up in 10% HCl and the non-alkaloidal portion was removed by extraction with $CHCl_3$. The aq. acidic layer was made alkaline with NH_3 and extracted with $CHCl_3$ to afford the crude alkaloids (70 g). A portion of the crude alkaloid (30 g) was chromatographed on a neutral alumina column, which was successively eluted with increasing polarities of mixtures of hexane, $CHCl_3$, and MeOH. The buxaminol-G containing fraction was eluted with 20% $CHCl_3$ and 80% MeOH. Buxaminol-G in 7.0 mg yield was isolated as a colourless gum $[\alpha]_D$ $CHCl_3$: 83.33°; IR $\nu_{max}^{CHCl_3}$ cm^{-1} 1380 (C–N), 1550 (C=C), 3550 (–OH); UV λ_{max}^{MeOH} nm: 237 (ε6040), 245 (ε5860), 253 (ε4151); ^{1}H NMR ($CDCl_3$): δ0.74 (3H, *s*, CH_3), 0.90 (3H, *s*, CH_3), 0.72 (3H, *d*, J = 5 Hz, H-21), 2.30 [6H, *s*, $-NMe_2$], 2.33 [6H, *s*, $-NMe_2$], 3.52 and 3.84 (2H, *dd*, J_{AB} = 10 Hz, $-CH_2OH$), 4.21 (1H, *m*, $W_{1/2}$ = 16 Hz, H-16), 5.15 (1H, *s*, H-19) δ5.45 (1H, *m*, H-11). MS *m/z*:444.3709 (1.28) $[M]^+$ ($C_{28}H_{28}N_2O_2$), 129.0901 (1.98, $C_7H_{15}NO$), 115.0650 (1.38, $C_6H_{13}NO$), 84.0904 (13, $C_5H_{10}N$), 72.0817 (100, $C_4H_{10}N$), 71.0740 (27, C_4H_9N), 58.065 (11, C_3H_8N).

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