X-Ray Crystal and Molecular Structure Determination of a Novel Bicyclic System. 2-*p*-Bromophenyl-1,3-Diazabicyclo[3,1,0] Hexane

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Summary The crystal and molecular structure as well as the conformation of 2-*p*-bromophenyl-1,3-diazabicyclo-[3,1,0]hexane $C_{10}H_{11}N_2Br$, have been determined by *X*-ray structure analysis; the 1,3-diazabicyclo[3,1,0]-hexane fragment of the molecule has the boat conformation, and the benzene ring orients itself in the *exo*-position to the heterohexane bicycle.

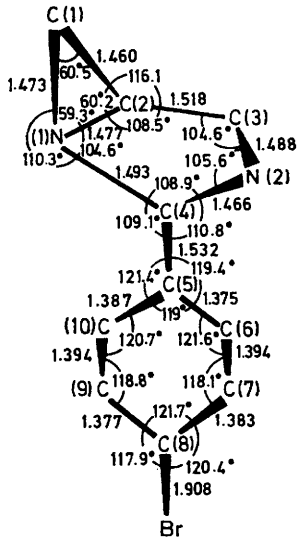
As part of our study on condensed three-membered and five-membered heterocycles with a common nitrogen atom,[1] we now report the *X*-ray structure analysis of 2-*p*-bromophenyl-1,3-diazabicyclo[3,1,0]hexane.

Crystal data. Colourless monoclinic crystals with $a = 10{\cdot}779(2)$, $b = 8{\cdot}638(2)$, $c = 12{\cdot}000(2)$ Å, $\beta = 121{\cdot}263(13)°$, $U = 955{\cdot}0$ Å^3, $M = 239{\cdot}15$, $D_c = 1{\cdot}66$ g cm^{-3}, $Z = 4$, μ (Cu-K_α) = 60·7 cm^{-1}, space group $P2_1/c$.

The three-dimensional range of intensities (1047 independent non-zero reflections) was obtained by the method of $\theta/2\theta$ scanning on a Syntex-automated four-circle diffractometer $P2_1$ using monochromatic cupric radiation.

The structure was solved by direct methods in the on line mode using the system XTL and the program MULTAN;[2] computations used 965 observed reflections with $I \geqslant 1{\cdot}96\sigma$; the model was refined by least-squares to an *R* value of 0·034 (anisotropic approximation).

The molecular geometry is shown in the Figure. The 1,3-diazabicyclo[3,1,0]hexane fragment of the molecule is in the boat conformation slightly curved in the direction of C(2)–C(4). The benzene ring orients itself in the exo-position to the heterohexane bicycle. The angle between the planes N(1)–C(2)–C(1) and N(1)–C(2)–C(4) is 104·7°; between N(2)–C(3)–C(4) and C(2)–C(3)–C(4) 28·9°; between N(1)–C(2)–C(4) and C(2)–C(3)–C(4) 4·5°; and between the benzene ring and the plane N(1)–C(2)–C(3)–C(4) 72·3°. The



FIGURE

structure is a molecular one. Some shorter intermolecular distances such as N(2) . . . Br = 3·2 Å and N(2) . . . N(1) = 3·1 Å, indicate the presence of steric hindrance in the molecular packing.

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[2] G. Germain, P. Main, and M. H. Woolfson, *Acta Cryst.*, 1970, **B26**, 274.