The Crystal Structure of 1,8,10,9-Triazaboradecalin

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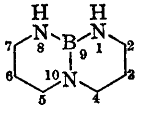
IN aminoboranes and borazoles the B–N bond is regarded as possessing some double-bond character[1] as a result of donation of electrons by the nitrogen atom to a π-bonding orbital between boron and nitrogen. The crystal structure of 1,8,10,9-triazaboradecalin (I) has been determined as part of a programme of studies of compounds containing the boron–nitrogen bond, in order to determine the length and order of the bond, and to compare the BN_3 group in this molecule with the NB_3 group in tri-(1,3,2-benzodioxaborol-2-yl)amine,[2] whose structure is also being determined in this laboratory.

Triazaboradecalin, $C_6H_{14}N_3B$, $M = 102{\cdot}98$. *Monoclinic*, $a = 18{\cdot}009$, $b = 5{\cdot}134$, $c = 8{\cdot}655$ Å, $\beta = 93{\cdot}57°$, $U = 798{\cdot}7$ Å^3, $Z = 4$, $D_c = 1{\cdot}156$ g.cm.$^{-3}$.

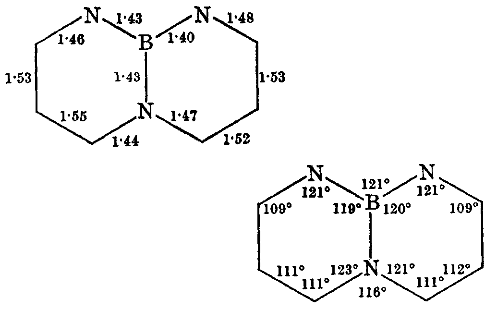


(I)

D_o has not been determined, since the crystals react with water and most organic solvents but D_c is close to the density of naphthalene (1·10), as is reasonable in view of the structure found. Space group $P2_1/n$ (No. 14).

The structure was solved from the sharpened Patterson function. Refinement was by isotropic least-squares analysis based on 1089 reflexions whose intensities were recorded on a Pailred diffractometer, using Mo-K_α-radiation. Hydrogen atoms were located from a difference synthesis, and included in the refinement at an appropriate stage. At present $R = 0·11$. Further refinement using anisotropic temperature factors is in progress.

The bond lengths (Å) and bond angles calculated at this stage of the refinement are indicated below:



Average standard deviations are 0·01 Å for bond lengths and 0·7° for bond angles.

The central part of the molecule comprising atoms 1, 4, 5, 8, 9, and 10 is almost planar [the maximum deviation from the mean plane is 0·045 Å for N(10)] as suggested by Niedenzu *et al.*,[4] but their assumption that the other carbon atoms also lie in this plane proves not to be justified. C(2) and C(7) lie about 0·2 Å, and C(3) and C(6) about 0·5 Å from the plane, the two pairs being on opposite sides, so that the molecular symmetry is approximately $C_s(m)$. The hydrogen atoms attached to nitrogen lie in positions such that the three bonds meeting at each nitrogen are approximately coplanar.

The B–N bond lengths are similar to those in borazole[5] and *B*-trichloroborazole.[6] This, and the coplanarity of the bonds at each nitrogen atom, indicates a contribution from π-bonding between boron and nitrogen, each of these atoms being in an sp^2 hybrid state. This conclusion is supported by the absence of N–H · · · · · N intermolecular hydrogen bonds from this structure; had they been present, N(1) and N(8) would necessarily have been in an sp^3 hybrid state and would have been unable to contribute to the π-bond system.

The research reported herein has been sponsored in part by the European Research Office, United States Army, Frankfurt-am-Main, Germany.

(*Received, May 25th,* 1967; *Com.* 516.)

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[3] Programmes used were (a) A Structure Factor and Diagonal Least-Squares Programme in Fortran IV, by G. J. Bullen and N. H. Clark; (b) A Fortran Crystallographic Least Squares Program, by W. R. Busing, K. O. Martin, and H. A. Levy, I.U.Cr. World List of Crystallographic Computer Programs, No. 360 (modified by D. Hunt of Imperial College).

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