# Isomers of $B_{20}H_{26}$: Structural Characterisation by *X*-Ray Diffraction of 2,2′-Bi(*nido*-decaboranyl)

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*Summary* One of the isomers of $B_{20}H_{26}$ obtained by the photolysis of $B_{10}H_{14}$ has been shown by single-crystal *X*-ray structure analysis to be 2,2′-bi(*nido*-decaboranyl).

A NUMBER of isomers of bi(*nido*-decaboranyl), $(B_{10}H_{13})_2$, have been isolated.[1,2] However, only for one of these has it been possible to infer the structure by indirect methods,[2] and there has been no direct proof of the bi(*nido*-decaboranyl) formulation. We summarize here the results of a single-crystal $X$-ray diffraction analysis which confirm that one of these isomers (isomer 1 in the numbering system of ref. 1) indeed has the bi(*nido*-decaboranyl) structure and that it is the 2,2′-isomer.

Isomer 1 was obtained *via* the photolysis of $B_{10}H_{14}$ and was isolated as in ref. 1. It crystallised from diethyl ether as colourless blocks, m.p. 177—178 °C. That the crystal selected for $X$-ray diffraction was representative of the bulk sample was subsequently confirmed by the identity of the $^{11}B$ and $^{11}B\text{-}\{^1H\}$ n.m.r. spectra. The n.m.r. data† are entirely consistent with the structure; the $^{11}B$ quadrupolar relaxation times ($T_1$) are all substantially shorter than those for $B_{10}H_{14}$, as found also for the 6,6′-isomer,[2] and as expected on the basis of increased molecular motion correlation times arising from the approximately two-fold increase in molecular size.

The crystals are tetragonal, space group $I4_1cd$, with $a = b = 11.950(3)$, $c = 23{\cdot}135(4)$ Å, and $Z = 8$. The structure was determined from 755 independent reflections with $5° < 2\theta(\text{Mo}\text{-}K_\alpha) < 50°$ and $I < 3\sigma(I)$. The boron atoms were located using MULTAN 76, and the hydrogen atoms were located on a subsequent difference map. Least-squares refinement with anisotropic thermal parameters for boron atoms and isotropic parameters for hydrogen atoms gave a final $R$ of 0·036.

The crystallographically imposed molecular symmetry is $C_2$ and the structure is shown in the Figure. The compound is 2,2′-bi(*nido*-decaboranyl), and the gross structure of each *nido*-decaboranyl fragment differs from that of decaborane only in having the 2-hydrogen atom replaced by the linkage to the second fragment. The length of the 2-centre, 2-electron apical–apical boron–boron $\sigma$-bond joining the two parts of the molecule is 1·692(3) Å, which may be compared to the length of 1·74(6) Å obtained for the only other such linkage measured, that in 1,1′-bi(*nido*-pentaboranyl), $(B_5H_8)_2$.[3] The cluster shows little distortion from that[4] of $B_{10}H_{14}$, the most significant variation being the lengthening of the five boron–boron nearest-neighbour distances to the substituted 2-boron atom, which are all *ca.* 0·02 Å longer than the corresponding ones to the 4-boron atom. Only 14 neutral boranes have been fully characterized by $X$-ray diffraction techniques, and of these the compound described here is the largest.

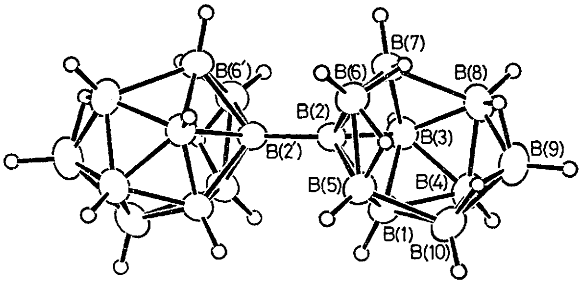

FIGURE. ORTEP drawing of the molecular structure of 2,2′-bi(*nido*-decaboranyl). The molecular $C_2$ axis is vertical, the boron atoms are represented by 50% probability ellipsoids, and the hydrogen atoms have been given artificial temperature factors, B, of 0·05 Å$^2$. In this projection the bridging hydrogen atom in the 9′, 10′-position is obscured.

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† $^{11}B$ N.m.r.: assignment, $\delta(^{11}B)$ ±0·05 p.p.m. to high frequency of $BF_3\cdot Et_2O$, and $T_1$/ms (±20%): B(1,3), +14·0 and 13·5; B(6), *ca.* +11·1 and *ca.* 2·8; B(9), *ca.* +11·1 and *ca.* 2·8; B(5,7), 0·0 and 4·6; B(8,10), +1·9 and 6·0; B(2), −31·6 and 4·8; and B(4), −34·5 and 21·0.

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