Structure of the $B_{10}H_{13}^-$ Ion

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Summary Three dimensional *X*-ray data collected at −170 °C show that the $B_{10}H_{13}^-$ ion has the same structure as $B_{10}H_{14}$, with one bridging hydrogen removed and a shortening of the corresponding boron–boron bond to 1·65 Å.

OF the known boron hydrides, one of the few structures which has remained in doubt is that of the $B_{10}H_{13}^-$ ion discovered[1] in 1958. Several structures have been proposed, based on various bonding theories[2] and spectral data.[3] The only previous crystallographic work was with etherates of the sodium salt[4] and indicated high thermal motion and/or disorder at ambient conditions.

Preliminary work on samples of the triethylammonium salt indicated excessive thermal motion and radiation damage at 22 °C, as crystals generally decomposed after one day of exposure to *X*-rays. A single crystal was mounted on a Picker automated diffractometer equipped with a highly oriented graphite monochromator and cooled to −170 °C by means of a nitrogen vapour coldstream.[5] Careful searching of a limited hemisphere indicated a triclinic unit cell, in agreement with the findings of preliminary film work. Cell dimensions at −170 °C are $a = 7{\cdot}200(4)$, $b = 14{\cdot}270(10)$, $c = 8{\cdot}452(5)$ Å, $\alpha = 82{\cdot}40(2)$, $\beta = 104{\cdot}28(5)$, and $\gamma = 112{\cdot}73(3)°$, giving a reasonable value of $D_C = 0{\cdot}957$ g cm^{-3} for $Z = 2$.

A complete sphere of data was collected to sin θ/λ 0·650 and averaged to obtain the final set of 1349 intensities greater than the standard error, based on counting statistics, which was used in the refinement. Statistical tests indicated a centrosymmetric structure, and the space group $P\bar{1}$ was assigned. All non-hydrogen atoms were located by direct methods, and the hydrogen atoms by difference Fourier methods. Anisotropic least-squares refinement of the structure gave a final residual of 0·048.

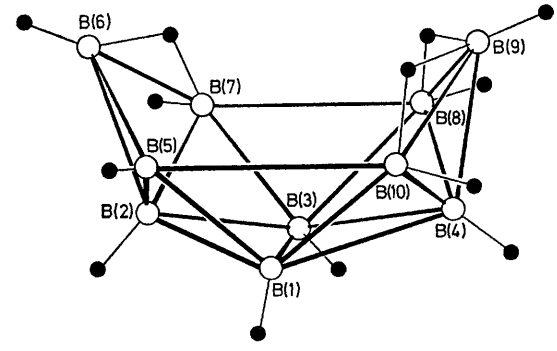

FIGURE. *The structure of the* $B_{10}H_{13}^-$ *ion. Boron–boron distances which differ by more than* 0·02 Å *from the corresponding distance in* $B_{10}H_{14}$ *are:* B(5)–B(6) = 1·65, B(7)–B(8) = 2·04, *and* B(5)–B(10) = 1·86 Å.

The structure, as shown in the Figure, is that of $B_{10}H_{14}$ with the B(5)–B(6) bridging hydrogen removed. Distances in the anion are equivalent to those[6] in $B_{10}H_{14}$ with the exception of those listed in the Figure, which differed by

more than 0·02 Å. It is interesting to note the B(5)–B(6) distance of 1·65 Å is one of the shortest boron–boron bonds reported.[7]†

The authors thank Mr. A. R. Siedle for preparation of the samples.

(*Received*, 28*th February* 1972; *Com.* 324.)

† The boron–boron distance of 1·60 in B_6H_{10} (ref. 7) was found to be 1·63 Å in a recent reinvestigation (J. C. Huffman, L. G. Sneddon, R. O. Schaeffer, and W. E. Streib, to be published).

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