Crystal Structure of Tridecaborane(19)

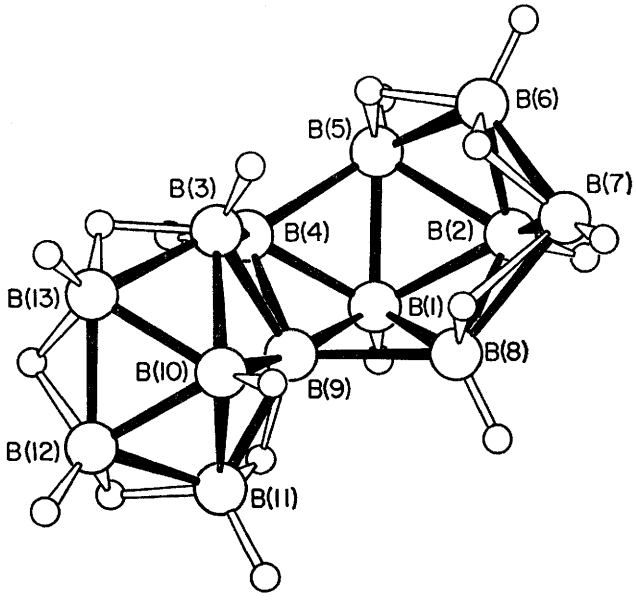
By JOHN C. HUFFMAN,* DAVID C. MOODY, JEROME W. RATHKE, and RILEY SCHAEFFER
(*Department of Chemistry, Indiana University, Bloomington, Indiana* 47401)

Summary The crystal structure of tridecaborane(19), a minor product of the pyrolysis of B_6H_{10} can be described as a *n*-B_9H_{15} cage which shares the B(3)–B(9) positions with two basal borons of B_6H_{10}.

YELLOW crystals (m.p. 44°) of $B_{13}H_{19}$ occur as a minor product of the pyrolysis of B_6H_{10}.[1] A single crystal was characterized and oriented on a Picker FACS-1 diffractometer equipped with a highly orientated graphite monochromator and liquid nitrogen cooling system[2] using a standardized reciprocal lattice search procedure.[3]

The crystals are monoclinic, $P2_1/c$ with $a = 9{\cdot}217(2)$, $b = 6{\cdot}498(3)$, $c = 19{\cdot}719(5)$ Å, $\beta = 97{\cdot}68(3)°$, and $D_c = 0{\cdot}91$ gm/cm^3 for $Z = 4$ at *ca.* −130°. Redundant data were collected using a standard θ—2θ scan technique to $\sin\theta/\lambda = 0{\cdot}538$, and averaged to yield a final set of 1763 unique reflections, of which 729 were greater than $2{\cdot}33\sigma$ (I) based on counting statistics. The latter were used in all subsequent calculations. All boron atoms were located by direct methods, and hydrogens were located using standard difference Fourier techniques. Isotropic least-squares refinement using unit weights gives an overall R-factor of 0·062 at this stage.

The structure (Figure) can be described as a *n*-B_9H_{15} cage sharing two borons with a B_6H_{10} cage. Interesting features are the lack of a terminal hydrogen on the seven-co-ordinate B(9), and an abnormally long B(7)–B(8) distance of 2·08(2) Å. The latter distance is significantly larger than the B(5)–B(10) distances of 1·987 Å reported[4] for $B_{10}H_{14}$ and 2·037 Å reported[5] for $B_{10}H_{13}^-$. The B_9 skeleton lacks the m symmetry present in *n*-B_9H_{15}, and is best described geometrically as a $B_{10}H_{14}$ cage fragment. All other boron distances and angles appear normal.



FIGURE

We acknowledge support from the National Science Foundation.

(*Received, 12th March* 1973; *Com.* 330.)

[1] J. Rathke and R. Schaeffer, to be published in *Inorg. Chem.*
[2] J. C. Huffman, J. M. Mueller, and W. E. Streib, unpublished data.
[3] J. C. Huffman, to be published in *J. Appl. Cryst.*
[4] Von R. Brill, H. Dietrich, and H. Dierks, *Acta Cryst.*, 1971, **B27**, 2003.
[5] L. G. Sneddon, J. C. Huffman, R. O. Schaeffer, and W. E. Streib, *J.C.S. Chem. Comm.*, 1972, 474.