Synthesis and *X*-Ray Structure of 4-Azanonaborane(13) (4-NB_8H_{13})

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Summary Addition of $NaNO_2$ to $B_{10}H_{14}$ in tetrahydrofuran, followed by acidification with HCl afforded the first uncharged 4-NB_8H_{13} azaborane, the structure of which was determined by *X*-ray diffraction.

THE reaction of sodium nitrite with decaborane in tetrahydrofuran followed by acidification with gaseous HCl afforded a new azaborane in *ca.* 30% yield. The azaborane, purified by column chromatography (silica gel, benzene), was an air-sensitive liquid, b.p. 60 °C at 10^{-2} Torr, m.p. *ca.* 0 °C.; ν(NH) 3405 s; $\nu(BH_t)$ 2570vs, 2590vs, and 2610vs; $\nu(BH_{bridge})$ 2220sh,w, 2145m, 2100sh,m, and 2060sh,m cm^{-1}; *m/e* 125·1802 (calc. for NB_9H_{12}, 125·1807). However, the ^{11}B n.m.r. spectrum showed only 8 doublets of relative intensities 1:2:1:2:2 and indicated the formula NB_8H_{13}

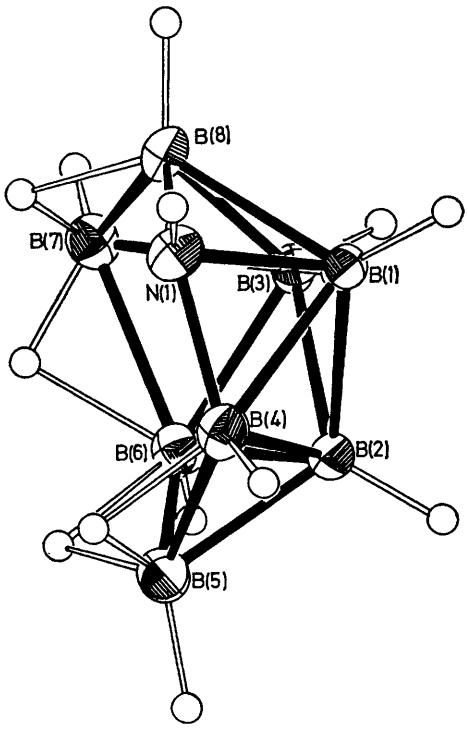

FIGURE. Structure of NB_8H_{13}.

[1] L. E. Todd, personal communication.

rather than NB_9H_{12}. An *X*-ray structure determination confirmed the NB_8H_{13} formation.

Crystal data: NB_8H_{13} crystallizes in the centrosymmetric orthorhombic space group *Pbca*, $a = 10{\cdot}619(5)$, $b = 11{\cdot}943(6)$, $c = 11{\cdot}866(6)$ Å, $Z = 8$. 1331 reflection intensities were collected by the θ–2θ scan technique using graphite-monochromated Mo-K_α radiation on a Picker FACS-1 automated diffractometer for a crystal grown and mounted in a glass capillary. 1252 reflections were considered non-zero, and were used in the subsequent refinement. The N and B atoms were located using direct methods and the H atoms were located using standard difference techniques. Anisotropic least-squares refinement on the heavy atoms (isotropic refinement of H) gives an overall *R*-factor of 0·076. This structure (Figure) was also proposed[1] on the basis of ^{11}B n.m.r. data. The structure can be described as an iso-B_9H_{15} cage with the ninth boron position replaced by a nitrogen and with two bridging hydrogens missing. Each atom has one terminal hydrogen. Four asymmetric bridging hydrogens are found on the open face, the short distances being towards B(5) and B(7).

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