The structure of **2** and the reactions of **1** and **2** are under investigation.

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The Formation of CB_5H_9 and Its 1-Methyl Derivative from 1,7-$C_2B_6H_8$

Sir:

We wish to report the preparation of the parent member of the CB_nH_{n+4} carborane series, where $n = 5$, 2-carbahexaborane(9), CB_5H_9,[1] and its 1-methyl derivative, both of which have not been previously reported, although several alkyl derivatives are known.[1-3]

The reaction between 1,7-dicarba-*closo*-octaborane-(8), $C_2B_6H_8$,[4-6] and tetramethylammonium borohydride[7] in diglyme at 100° for 24 hr yields as yet uncharacterized ionic products. Removal of the solvent by vacuum distillation followed by heating at 85° *in vacuo* for 10 hr produced a solvent-free solid. The solid was then treated with an excess of dry HCl, and a mixture of neutral compounds was liberated and separated using standard vpc methods. Three major fractions were collected and characterized.

The mass spectrum of 2-carbahexaborane(9), CB_5H_9 (Figure 1), the major fraction formed in approximately 20% yield, exhibited a cutoff at m/e 76 which corresponds to the $^{12}C^{11}B_5{}^1H_9{}^+$ ion. The 60-Mc/sec ^1H nmr spectrum consisted of a broad peak at τ 4.62 relative to internal tetramethylsilane (TMS). The ^{11}B nmr spectrum (Figure 1) consisted of three sets of doublets of relative area 2:2:1 which is consistent with a species containing five borons in a pentagonal pyramid structure. The doublet at lowest field shows secondary splitting due to bridge hydrogen coupling.

The mass spectrum of 1-methyl-2-carbahexaborane(9), 1-$CH_3CB_5H_8$, exhibited a cutoff at m/e 90 which corresponds to the $^{12}C_2{}^{11}B_5{}^1H_{11}{}^+$ ion. The 60-Mc/sec ^1H nmr spectrum consisted of a broad peak of relative area 3 at τ 10.58 and a broad peak of relative area 1 at τ 4.45 relative to internal TMS. The ^{11}B nmr spectrum (Figure 1) consisted of two sets of doublets and a singlet of relative area 2:2:1, respectively. The high-field singlet indicates substitution at the 1 position.

The third fraction identified from the reaction was 3-methyl-2-carbahexaborane(9), 3-$CH_3CB_5H_8$, which was identical with that reported earlier.[1]

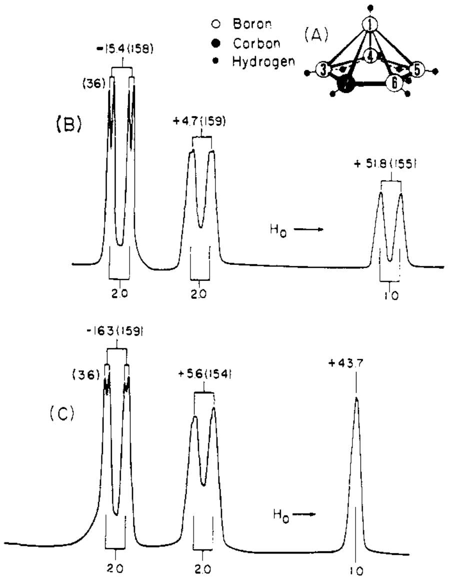

Figure 1. (A) The molecular structure of 2-carbahexaborane(9), CB_5H_9. The 32.1-Mc/sec ^{11}B nmr spectra of CB_5H_9 (B) and 1-$CH_3CB_5H_8$ (C). Chemical shifts (ppm, relative to $BF_3 \cdot O(C_2H_5)_2$) and coupling constants (cps) are indicated. Relative areas appear beneath the peaks.

Even though the removal of boron atoms from carborane cages is well known both by pyrolytic and chemical methods,[6,8-13] this appears to be the first example of the removal of cage carbon atoms from a closed carborane.

The characterization of the ionic species and several minor components also obtained in this reaction, together with the results of the reaction between C-methyl and C,C′-dimethyl-1,7-dicarba-*closo*-octaborane(8) with borohydride ion, will be presented elsewhere.

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The Nuclear Overhauser Enhancement of the Carbon-13 Magnetic Resonance Spectrum of Formic Acid

Sir:

The difficulties associated with detecting the carbon-13 isotope in its 1.1% natural abundance are well known, and therefore it is important to characterize the dramatic improvement in the ratio of signal to noise realized with proton decoupling methods. In addition to the enhancement expected from proton multiplet collapse, application of a proton decoupling radio-