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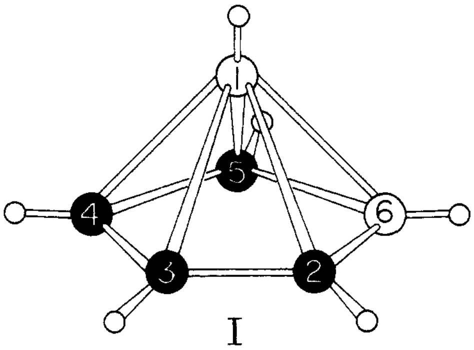
Preparation of the Pentagonal Pyramidal Carborane, 2,3,4,5-Tetracarba-*nido*-hexaborane(6)

Sir:

We wish to report the synthesis of 2,3,4,5-tetracarba-*nido*-hexaborane(6), I (Figure 1), from 1,2-tetramethylenediborane(6). The conversion was accomplished at 550° using a high-vacuum system which permits a rapid flow of gaseous reactant[1,2] through the hot zone at low pressures. In addition to the carborane, $C_4B_2H_6$, in low yield, a number of other carboranes are also produced. The experimental details of this reaction and the nature of the side products will appear in a longer dissertation at a later date.



I

Figure 1. Ball and stick model of $C_4B_2H_6$.

The evidence used to establish the formula and structure of this parent four-carbon two-boron carborane is outlined.

(a) The mass spectrum of the product purified by repeated vacuum fractional distillation exhibits a sharp cutoff at *m/e* 76 (calcd for $^{12}C_4{}^{11}B_2H_6$). When the parent envelope of peaks of the polyisotopic spectrum is subjected to a monoisotopic boron analysis a good fit is found for a compound consisting of two boron atoms.

(b) The ^{11}B nmr at 32.1 MHz exhibits two sharp doublets in an area ratio of 1:1 with chemical shifts (parts per million relative to boron trifluoride ethyl etherate) and coupling constants of +60.8 ppm (202 Hz) and −10.4 ppm (144 Hz). This is consistent with two B–H groups, with the high-field doublet assigned to one of these groups in an apical environment of a pyramid and the low-field doublet assigned to a basal position.[3]

(c) The proton nmr taken at both 100 and 220 MHz consist of two slightly broadened singlet resonances at (relative to τ 10.00 ppm for TMS) τ 5.38 and 4.24 ppm of equal area which are assigned to the two different kinds of carbon-attached hydrogens of I, and also two equal-area 1:1:1:1 quartets at 6.08 (J = 141 Hz) and 10.59 ppm (J = 205 Hz) which are assigned to H–^{11}B(6) and H–^{11}B(1), respectively.[4] The area of each quartet is almost one-half of the area of each H–C singlet. The chemical shifts of all of the protons are reasonably within the range of values found for other pyramidal systems containing a different number of carbon and boron atoms in the series $C_nB_{6-n}H_{10-n}$ (*i.e.*, n = 0–3).[3]

(d) The most informative portions of the infrared spectrum are consistent with the assigned structure, *i.e.*, bands at 2950 (C–H) and 2570 cm^{-1} (B–H) and an absence of peaks in the B–H–B bridge regions.

In the pentagonal-pyramidal series of isoelectronic compounds $C_nB_{6-n}H_{10-n}$, the parent and/or monomethyl derivatives of those with n = 0, 1, 2, or 3 have been previously prepared and reasonably well structurally characterized. The present work adds to the parent compounds in the series a compound in which n = 4. It should be pointed out, however, that the permethyl[5] and perphenyl[6] derivatives of $C_4B_2H_6$ have been previously reported and the structures tentatively, but inconclusively, assigned. The data we present, however, leave little doubt about the identification and structural characterization of the parent $C_4B_2H_6$.

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Solubilization of Alkali Metals in Tetrahydrofuran and Diethyl Ether by Use of a Cyclic Polyether

Sir:

We wish to report a new technique for dissolving alkali metals in solvents in which they are ordinarily either insoluble or only slightly soluble. This method may extend the range of solvents in which the properties of relatively stable solutions of solvated electrons and other species common to metal–amine solutions[1,2] can be studied. Of particular interest would be the ability to make extended comparisons with the properties of solvated electrons produced by pulse radiolysis.

The basis for this increased solubility is the ability of certain cyclic polyethers to complex alkali metal cations.[3,4] Noting that stabilization of the cations

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