(measured as a gas) of trimethylamine was added. The mixture was stirred for 1 day, the solid (NaI) was separated by filtration, and the filtrate was concentrated to a pale yellow solid residue under vacuum. Extraction of this with water and metathesis of the solution with ammonium hexafluorophosphate gave 249 mg (7%) of solid whose melting point (143–144°) and spectral data matched those of material designated as $(CH_3)_3NBH_2CNBH_2N(CH_3)_3^+$ prepared from methyl isocyanide and trimethylamine–iodoborane. The water-insoluble product, 156 mg, was recrystallized from methylene chloride–pentane and identified as $(CH_3)_2NBH_2NCBH_3$ by elemental analyses and spectral data; yield 123 mg, mp 126–128°.

Syntheses of $[(CH_3)_3NBH_2NC]_2BH_2^+PF_6^-$ and $(CH_3)_3NBH_2NCBH_2P(CH_3)_3^+PF_6^-$.—Iodine, 587 mg, in 20 ml of chloroform was added to 516 mg (4.6 mmol) of $(CH_3)_3NBH_2NCBH_3$ in 5 ml of chloroform. Only a very slow discoloration was noticed. After stirring overnight a yellow solid was present in a brownish solution. To this was added 0.6 g of pyridine *N*-oxide in 3 ml of chloroform. Some lightening of the coloration was noticed, but the mixture was still cloudy yellow after 3 days of stirring. A small amount of insoluble material was filtered. The filtrate was evaporated under vacuum and the resulting residue was treated with 20 ml of water which dissolved all but a trace. Metathesis to the hexafluorophosphate salt gave 488 mg of solid later identified as the first title compound by analytical and spectral data. A similar experiment using DMF instead of pyridine oxide gave the same product. When *N*,*N*-dimethylacetamide or pyridine was employed, no PF_6^- salt was isolated. When trimethylphosphine was used as the second base, 64% of the salt $(CH_3)_3NBH_2NCBH_2P(CH_3)_3^+PF_6^-$ was obtained.

Chlorination of $(CH_3)_3NBH_2OCHN(CH_3)_2^+PF_6^-$.—A 313-mg sample of $(CH_3)_3NBH_2OCHN(CH_3)_2^+PF_6^-$ in 5 ml of methylene chloride was treated with chlorine by passing gas into solution for 0.5 hr, resulting in a slurry of a white solid suspended in a yellow liquid. Volatiles were removed under vacuum (at first with water aspirator vacuum) to leave 386 mg (99%), mp 126–127°, of white reflective crystals of $(CH_3)_3NBCl_2OCHN(CH_3)_2^+PF_6^-$ identified by spectral data and analyses.

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CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF VIRGINIA,
CHARLOTTESVILLE, VIRGINIA 22901

Carborane Formation in Alkyne–Borane Gas-Phase Systems. V.[1] Conversion of Two-Carbon to Four-Carbon Carboranes *via* Alkyne Insertion. Nuclear Magnetic Resonance Studies of Tetracarba-*nido*-hexaboranes

BY VERNON R. MILLER AND RUSSELL N. GRIMES*

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The reaction of the square-pyramidal carborane 1,2-$C_2B_3H_7$ with acetylene yields one volatile product, 2,3,4,5-$C_4B_2H_6$, a carborane of pentagonal-pyramidal geometry. The process occurs as a straightforward alkyne insertion into the carborane cage, as shown by the use of deuterium- or carbon-13-labeled acetylene. The reaction of 1,2-$C_2B_3H_7$ with methylacetylene produces 3-$CH_3C_4B_2H_5$ and a lesser quantity of 2-$CH_3C_4B_2H_5$, while the reaction with dimethylacetylene generates predominantly 2,3-$(CH_3)_2C_4B_2H_4$ with a smaller amount of 3,4-$(CH_3)_2C_4B_2H_4$. The results indicate that alkyne insertion into $C_2B_3H_7$ occurs primarily at C–B bonds without C–C cleavage but that insertion into the carborane C–C bond also occurs to a significant extent. The proton and boron-11 nmr spectra of the $C_4B_2H_6$ derivatives are correlated with structural features, and the spectra of the parent compound are interpreted.

The synthesis of volatile carboranes in reactions of boranes with alkynes has been described in many previous reports.[2] Of the carboranes produced in such reactions, those having two framework carbon atoms are overwhelmingly predominant, although one- and three-carbon species have been isolated in some cases. A logical extension of these syntheses would be the insertion of acetylene into a two-carbon carborane to generate a four-carbon system, but such reactions have been unknown prior to the present work. However, the new compound 1,2-dicarbapentaborane(7), 1,2-$C_2B_3H_7$,[3] has been found to react with acetylene under mild conditions (50°) to yield 2,3,4,5-tetracarbahexaborane(6), $C_4B_2H_6$ (Figure 1).[4] This latter molecule, the only known four-carbon carborane system, was previously characterized as a peralkylated derivative by Binger[5] and has recently been isolated in parent form by Onak and Wong[6] from the 550° pyrolysis of tetramethylenediborane. Since the formation of $C_4B_2H_6$ from $C_2B_3H_7$ and C_2H_2 implied a simple alkyne–carborane insertion, our interest in the mechanism of this process has stimulated further studies involving labeled acetylenes and higher alkynes.

Results

Reaction of $C_2B_3H_7$ with $^{13}C_2H_2$ and C_2D_2.—As reported elsewhere,[4] the gas-phase reaction of $C_2B_3H_7$ with C_2H_2 at 50° yields a white nonvolatile solid and a single volatile product, $C_4B_2H_6$. The ^{11}B and ^1H nmr spectra of this material establish that only the 2,3,4,5 isomer, containing four basal carbons in a pentagonal-pyramidal cage, is present. The apparent insertion of a C_2H_2 unit has been confirmed by allowing ^{13}C-enriched acetylene to react with normal $C_2B_3H_7$. The mass spectrum of the $C_4B_2H_6$ product establishes

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[1] Part IV: D. A. Franz and R. N. Grimes, *J. Amer. Chem. Soc.*, **93**, 387 (1971).

[2] Recent reviews: R. E. Williams, *Progr. Boron Chem.*, **2**, 37 (1970); R. N. Grimes, "Carboranes," Academic Press, New York, N. Y., 1970.

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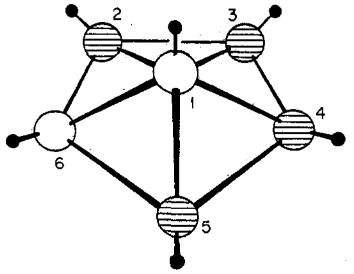

Figure 1.—Structure and numbering system for $C_4B_2H_6$. Boron atoms are open and carbon atoms are shaded.

the incorporation of two ^{13}C atoms, and no evidence is found for ^{13}C exchange with the $C_2B_3H_7$ starting material. The interaction of isotopically normal $C_2B_3H_7$ with C_2D_2 at 50° yields only dideuterated tetracarbahexaborane ($D_2C_4B_2H_4$), with no evidence in the mass spectrum for species containing other than two deuterium atoms. Moreover, the ^{11}B nmr data, described below, establish that no B–D bonds are present. Making the reasonable assumption, consistent with all available evidence, that cleavage of the C–D and *alkynyl* C–C bonds does not occur under the mild conditions employed, the only possible product isomers are 2,3- and 3,4-$D_2C_4B_2H_4$. Formation of 3,4-$D_2C_4B_2H_4$ implies the insertion of C_2D_2 into the C–C bond of the original 1,2-$C_2B_3H_7$ reactant; interestingly, the ^1H nmr spectrum of the product (see below) does indicate two isomers, the 2,3-D_2 species predominating but with a small contribution from 3,4-$D_2C_4B_2H_4$. The product yields from this and other $C_2B_3H_7$–alkyne reactions are listed in Table I.

TABLE I

VOLATILE PRODUCTS OF $C_2B_3H_7$–ALKYNE REACTIONS[a]

Alkyne reactant	Products (% of volatile products)
C_2H_2	2,3,4,5-$C_4B_2H_6$ (100)
$^{13}C_2H_2$[b]	$^{13}C_2{}^{12}C_2B_2H_6$ (100)
C_2D_2	2,3-$D_2C_4B_2H_4$ (93)[c] 3,4-$D_2C_4B_2H_4$ (7)[c]
$CH_3C{\equiv}CH$	2-$CH_3C_4B_2H_5$ (12)[c] 3-$CH_3C_4B_2H_5$ (88)[c]
$CH_3C{\equiv}CCH_3$	2,3-$(CH_3)_2C_4B_2H_4$ (87)[c] 3,4-$(CH_3)_2C_4B_2H_4$ (13)[c]

[a] Reactions at 50° with isotopically normal $C_2B_3H_7$ used in all cases. [b] Carbon-13 content 60%. [c] Approximate composition, determined from proton and boron-11 nmr spectra.

Reaction of $C_2B_3H_7$ with $CH_3C{\equiv}CH$.—Two C-substituted isomers are possible for the expected CH_3-$C_4B_2H_5$ product, and the nmr spectra clearly indicate the presence of both 2-CH_3- and 3-$CH_3C_4B_2H_5$ with the latter present in larger quantity (Table I). Since either isomer can form without cleavage of the original carborane C–C bond, no direct inferences concerning the alkyne-insertion mechanism can be drawn in this case.[7] However, the predominance of the 3-methyl over the 2-methyl derivative is consistent with the results of the dimethylacetylene reaction described below.

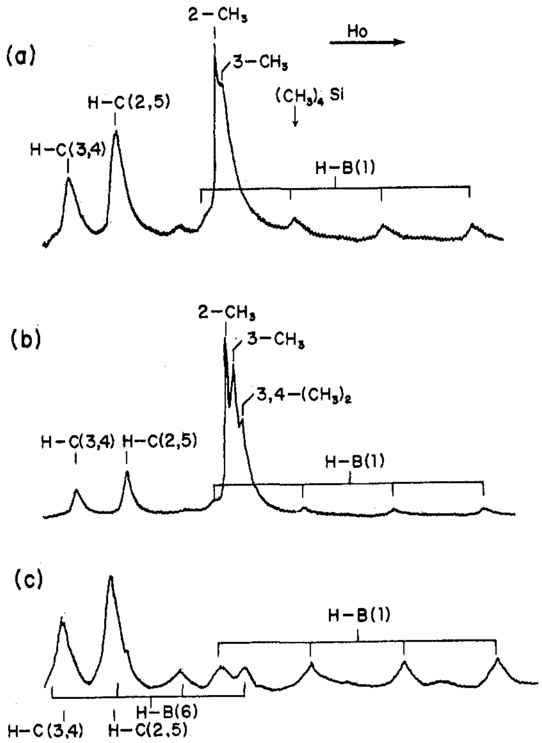

Figure 2.—The 100-MHz ^1H nmr spectra of tetracarbahexaborane derivatives (neat liquid samples): (a) mixture of 2-CH_3-$C_4B_2H_5$ and 3-$CH_3C_4B_2H_5$; (b) mixture of 2,3-$(CH_3)_2C_4B_2H_4$ and 3,4-$(CH_3)_2C_4B_2H_4$; (c) mixture of 2,3-$D_2C_4B_2H_4$ and 3,4-D_2C_4-B_2H_4. Chemical shifts and coupling constants are listed in Table II. Although the dimethyl and monomethyl derivatives are each chromatographically resolvable into the pure isomers, the spectra shown are those of the original product mixtures in order to allow precise comparisons of chemical shifts and peak areas. Spectra were obtained at low power levels to minimize saturation effects, and peak area ratios were reproducible in repeated scans.

Reaction of $C_2B_3H_7$ with $CH_3C{\equiv}CCH_3$.—Dimethylacetylene and $C_2B_3H_7$ interact at 50° to produce two C,C′-dimethyl derivatives of $C_4B_2H_6$ but no other volatile products. Once again, only two isomers are allowed if methyl migration and alkyne C–C cleavage are excluded. The nmr spectra establish that the major isomer is 2,3-$(CH_3)_2C_4B_2H_4$, with a lesser amount of the 3,4-$(CH_3)_2C_4B_2H_4$ isomer as indicated in Table I.

Nmr Spectra of 2,3,4,5-$C_4B_2H_6$ Derivatives.—The ^{11}B nmr spectrum of the isotopically normal parent compound, reported previously[6] and confirmed in our laboratory, consists of two sharp, widely separated doublets at δ +60.8 and −10.4 arising from the two nonequivalent B–H groups. In conformity with the pattern observed for other nido carboranes and boranes, the high-field doublet has been assigned[6] to the apex boron atom. The ^1H nmr spectrum[6] (Table II) contains the expected two C–H singlets and two B–H quartets, but specific assignments of the C–H resonances were not possible prior to the present work. Such correlations can now be made with reasonable confidence, based on the 100-MHz ^1H nmr spectra of the $(CH_3)_2C_4B_2H_4$ isomers (Figure 2b). Since the spectrum of the 2,3-$(CH_3)_2$ isomer should contain two different CH_3 resonances and two cage C–H peaks, while the symmetrical 3,4-$(CH_3)_2$ isomer would be expected to exhibit only one C–H and one CH_3 resonance, the larger of the two cage C–H peaks in Figure 2b can be attributed to the "end" (2 and 5) C–H groups,

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[7] Carborane C–C cleavage *may* be involved in the formation of 3-CH_3C_4-B_2H_5, but the same isomer can also form by endwise addition of the alkyne to the carboranyl C–C bond.

TABLE II

100-MHz ^{1}H NMR DATA FOR TETRACARBA-*nido*-HEXABORANE DERIVATIVES[a]

Compound	Assignments, ppm (relative to $(CH_3)_4Si$)					
	H-C(3,4)	H-C(2,5)	2(5)-CH_3	3(4)-CH_3	H-B(6)[b]	H-B(1)[b]
$C_4B_2H_6$	−5.76[c,d]	−4.62[c,d]			−3.92 (141)[c]	+0.59 (205)[c]
$D_2C_4B_2H_4$[e]	−5.53	−4.37			−3.60 (143)	+1.10 (206)
2-$CH_3C_4B_2H_5$	−5.35	−4.26	−1.95		−3.57 (143)	+0.76 (202)
3-$CH_3C_4B_2H_5$	−5.35	−4.26		−1.79	−3.57 (143)	+0.76 (202)
2,3-$(CH_3)_2C_4B_2H_4$	−5.17	−4.03	−1.72	−1.52	−3.40 (141)	+0.98 (202)
3,4-$(CH_3)_2C_4B_2H_4$		−4.03		−1.38	−3.40 (141)	+0.98 (202)

[a] Neat liquid samples. [b] Coupling constants in hertz given in parentheses. [c] Data taken from ref 6. [d] Assignment based on comparison with spectra of methyl derivatives (see text). [e] Mixture of 2,3- and 3,4-$D_2C_4B_2H_4$.

which are common to both isomeric species.[8] The smaller C–H resonance is assigned to the "middle" (3 and 4) C–H units, which exist only in 2,3-$(CH_3)_2C_4B_2H_4$. It is clear that the major species in the product mixture is the unsymmetrical 2,3-$(CH_3)_2C_4B_2H_4$ isomer, and analysis of the area ratio[9] of the two cage C–H resonances indicates the composition given in Table I. Assignment of the CH_3 peaks in the spectrum is straightforward, since the two larger resonances must arise from the major [2,3-$(CH_3)_2$] isomer while the small, high-field peak is due to the minor component, 3,4-$(CH_3)_2C_4B_2H_4$. Similarly, in the ^{11}B nmr spectrum (Figure 3b) the larger doublets are attributed to the major isomer, 2,3-$(CH_3)_2C_4B_2H_4$. The area ratio of the high-field doublets in this spectrum is in reasonable agreement with the isomer composition calculated from the proton nmr spectrum (Table I).

In the nmr spectra of the monomethyl derivatives, the presence of both C-substituted isomers is obvious. Comparison of the proton spectrum (Figure 2a) with that of the dimethyl species discussed above suggests the assignments shown, and from the relative areas[10] of the cage C–H peaks it appears that the 3-CH_3 isomer predominates (Table I). This conclusion is further supported by the observations that (1) the CH_3 resonance of larger area appears at higher field than the weaker peak and (2) the major apex (high-field) doublet in the ^{11}B nmr spectrum (Figure 3a) is centered at lower field than the smaller apex doublet. Both facts are consistent with the assignments in the ^{11}B nmr spectra of the dimethyl derivatives, described above.

The nmr data for $D_2C_4B_2H_4$ may be interpreted similarly. The ^{11}B nmr spectrum is similar to that of normal $C_4B_2H_6$ (Table III) and is uninformative except to confirm that no B-deuteration has occurred. The proton spectrum (Figure 2c) indicates that, statistically, deuterium substitution is more extensive at C(3) than at C(2) locations, in that the higher field cage C–H resonance has the larger area. As before, the relative amounts of the isomers are deduced from the areas of the cage C–H resonances in the proton nmr spectrum.

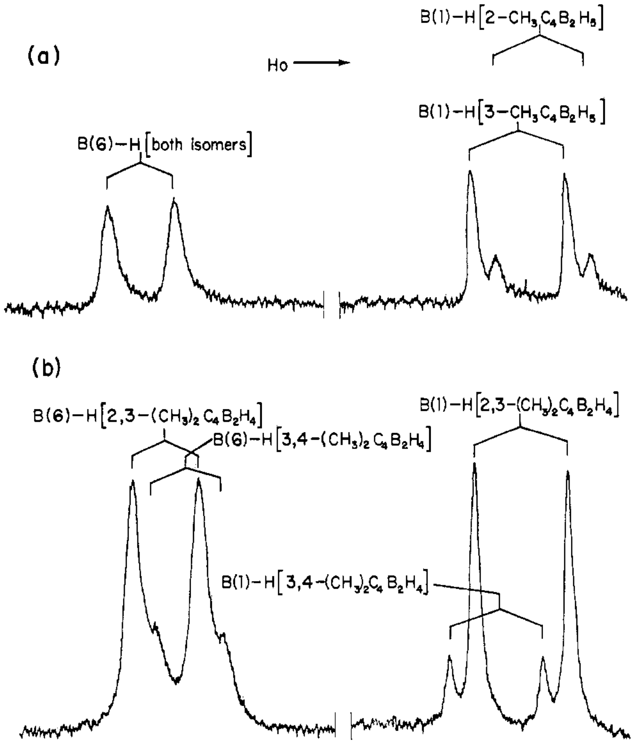

Figure 3.—The 32.1-MHz ^{11}B nmr spectra of the $CH_3C_4B_2H_5$ isomer mixture (a) and the $(CH_3)_2C_4B_2H_4$ isomer mixture (b), obtained under the same conditions as the proton nmr spectra in Figure 2. The high-field doublets in Figure 3b are slightly saturated at the power level employed; at reduced power, the areas of the high-field and low-field doublets are essentially equal. Chemical shifts and coupling constants are listed in Table III.

TABLE III

32.1-MHz ^{11}B NMR DATA FOR TETRACARBA-*nido*-HEXABORANE DERIVATIVES[a]

Compound	B(1)-H	B(6)-H
$C_4B_2H_6$	+60.8 (202)[b]	−10.4 (144)[b]
$D_2C_4B_2H_4$[c]	+59.2 (204)[d]	−9.8 (134)[d]
2-$CH_3C_4B_2H_5$	+56.2 (198)	−10.8 (136)
3-$CH_3C_4B_2H_5$	+54.8 (198)	−10.8 (136)
2,3-$(CH_3)_2C_4B_2H_4$	+54.1 (198)	−10.3 (136)
3,4-$(CH_3)_2C_4B_2H_4$	+52.6 (198)	−9.2 (136)

[a] In ppm relative to boron trifluoride etherate; coupling constants in Hz given in parentheses; neat liquid samples. [b] Data taken from ref 6. [c] Mixture of 2,3- and 3,4-$D_2C_4B_2H_4$. [d] The slight difference between this value and the corresponding chemical shift of the parent compound is close to the expected limits of experimental error and hence may not represent a true isotope effect.

Discussion

Spectroscopic and chemical studies of 1,2-$C_2B_3H_7$ have suggested that the carbon–carbon link in this molecule is of approximately double-bond character.[4] Extensive cleavage of this bond appeared improbable

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(8) It is apparent that the chemical shifts of the C(2,5)–H and C(3,4)–H resonances are relatively insensitive to changes in the number or position of alkyl substituents, so that the corresponding cage C–H peaks in the spectra of two dimethyl isomers are superimposed (Figure 2b), as are those of the monomethyl species (Figure 2a). The broadness of these cage C–H resonances contributes to this effect; in contrast, the much sharper CH_3 peaks are *not* superimposed.

(9) [Area of C(2,5)–H peak]/[area of C(3,4)–H peak] = $(B + 2A)/B$, where A is the mole per cent of 3,4-$(CH_3)_2C_4B_2H_4$ and B is the mole per cent of 2,3-$(CH_3)_2C_4B_2H_4$. Corrections for the B–H resonances underlying the C–H peaks have been made in determining the peak areas.

(10) For the monomethyl derivatives, [area of C(2,5)–H peak]/[area of C(3,4)–H peak] = $(2B + A)/(2A + B)$, where A is the mole per cent of 2-$CH_3C_4B_2H_5$ and B is the mole per cent of 3-$CH_3C_4B_2H_5$. Corrections for B–H contributions have been made.

under mild conditions and was not observed during our earlier work.[4] Thus, alkyne insertion into $C_2B_3H_7$ seemed more likely to involve B–B or B–C cleavage, leaving the C–C link intact. Although the present study essentially substantiates this view, it is significant that in the reactions of dideuterio- or dimethylacetylene with $C_2B_3H_7$, roughly 10% of the tetracarbahexaborane products give evidence of C–C cleavage in the carborane reactant. The formation of two isomers in each reaction implies a common mechanism, with at least two possibilities worth considering. Conceivably, the formation of isomers may result from alkyne attack at different regions of the $C_2B_3H_7$ cage; alternatively, and perhaps more plausibly, one can envision a single unstable intermediate which is capable of rearrangement to both "cleaved" and "noncleaved" tetracarbahexaborane products. Ultimately it may be possible to obtain kinetic evidence for such a process.

Finally, this work suggests that alkyne insertion into other nido carboranes may take place, to generate new carborane systems containing three, four, five, or more carbon atoms. Such possibilities are currently under investigation in our laboratory.

Experimental Section

All work was performed in Pyrex vacuum systems using standard techniques. Gas-phase reactions were conducted in evacuated and degassed Pyrex bulbs equipped with Teflon stopcocks.

Instrumentation.—Infrared spectra were obtained on a Beckman IR-8 spectrophotometer, using a 52-ml gas cell of 12.5-cm path length having NaCl windows. Mass spectra were recorded on a Hitachi Perkin-Elmer RMU-6E spectrometer, and nmr spectra were obtained on a Varian HA-100 instrument.

Materials.—Dicarbapentaborane(7) was prepared as described elsewhere.[3,4] Acetylene (Matheson) was purified by repeated passage through a trap at −128°, methylacetylene (Matheson) by distillation through a −95° trap and collection at −128°, and dimethylacetylene (K & K) by passage through a trap at −63°. Dideuterioacetylene was prepared from D_2O (Strohler Isotopes) and calcium carbide and was purified by distillation through a −128° trap several times. Carbon-13-enriched acetylene (Mallinkrodt and Stohler Isotopes, 60% ^{13}C content) was distilled through a −128° trap prior to use.

Reactions of 1,2-Dicarbapentaborane(7) with Carbon-13- and Deuterium-Labeled Acetylenes.—The reaction with C_2H_2 at 50° has been described earlier.[4] For the $^{13}C_2H_2$ experiment, 0.40 mmol of $C_2B_3H_7$ was allowed to react with 5.21 mmol of $^{13}C_2H_2$ in a 500-ml bulb. After 24 hr at 50° the inside of the reactor was coated with a white deposit. Unreacted $^{13}C_2H_2$ (4.68 mmol) was separated from the other materials by distillation through a −128° trap; a mass spectrum indicated no significant change in ^{13}C content as compared with the starting material. The products condensing at −128° were separated by glpc at 65°, which gave 0.104 mmol of unreacted $C_2B_3H_7$ and 0.019 mmol of $C_4B_2H_6$ (6.4% yield). Similar results were obtained starting with 3.46 mmol of $C_2B_3H_7$ and 18.5 mmol of $^{13}C_2H_2$. The recovered $C_2B_3H_7$ was isotopically identical with the starting material, as shown by mass spectroscopic analysis. The mass spectrum of the $C_4B_2H_6$ product contains a sharp cutoff at *m/e* 78, corresponding to the parent ion, and the fragmentation pattern is in close agreement with that calculated for a species containing two incorporated acetylenic carbons, assuming 60% ^{13}C content in the acetylene.

The reaction with C_2D_2 was conducted under the same conditions and reactant concentrations as in the preceding reaction, and the yields of corresponding products were essentially identical. The mass spectrum of the $D_2C_4B_2H_4$ product contains a sharp cutoff at *m/e* 78, corresponding to the parent ion, and contains no evidence for species having more or fewer than two deuterium atoms.

Reaction of Dicarbapentaborane(7) with Methylacetylene.—A mixture of 4.38 mmol of CH_3C_2H and 0.443 mmol of $C_2B_3H_7$ in a 500-ml bulb was maintained at 45–50° for 27 hr, during which a white nonvolatile solid formed on the walls. Chromatography on a column of 30% Kel-F on Chromosorb W at 67° gave 0.01 mmol of unreacted $C_2B_3H_7$ and 0.084 mmol (20% yield) of the combined $CH_3C_4B_2H_5$ isomers. The isomer mixture from two such reactions (total 0.165 mmol) was separated by repeated glpc at 50° on a column of 30% tricresyl phosphate on Chromosorb W, to give the pure 2-$CH_3C_4B_2H_5$ (0.031 mmol) and 3-$CH_3C_4B_2H_5$ (0.079 mmol.) Some loss of each isomer was experienced in the purification process, so that the ratio of pure isomers differs somewhat from that determined from the nmr spectra (see above). Infrared data (gas phase, cm^{-1}): 2-$CH_3C_4B_2H_5$ (11 Torr), 3090 (m), 2950 (s), 2890 (m), 2655 (vs, sp), 2585 (vs), 1890 (w), 1460 (m, b), 1310 (m), 1260 (w), 1210 (m, b), 1115 (m), 1050 (m, b), 1002 (sh), 996 (vs, sp), 855 (m), 796 (s, sp), 730 (s, b), 718 (s, sp); 3-$CH_3C_4B_2H_5$ (28 Torr), 3083 (s), 2983 (s, sp), 2945 (s), 2900 (m), 2657 (vvs, sp), 2585 (vvs), 1870 (w, b), 1470 (s, b), 1350 (m), 1290 (vs), 1175 (m, b), 1135 (m), 1050 (s), 995 (vvs), 950 (m), 855 (s, sp), 802 (s, sp), 770 (s, sp), 718 (m), 708 (s, sp).

Reaction of Dicarbapentaborane(7) with Dimethylacetylene.—The carborane (0.983 mmol) and $(CH_3)_2C_2$ (8.75 mmol) were allowed to react at 50° in a 500-ml bulb for 40 hr. A viscous, nonvolatile liquid formed during the reaction but no solid was observed. Fractionation gave 4.70 mmol of unreacted alkyne (passed −84° trap) and 0.097 mmol of the combined 2,3-$(CH_3)_2$- and 3,4-$(CH_3)_2C_4B_2H_4$ isomers (condensed at −84°). Chromatography of the mixture on the previously described tricresyl phosphate column at 73° gave 0.050 mmol of 2,3-$(CH_3)_2C_4B_2H_4$ and 0.019 mmol of the 3,4-$(CH_3)_2$ isomer. As before, some material was unavoidably lost during purification. Infrared data: 2,3-$(CH_3)_2C_4B_2H_4$ (12 Torr), 3090 (m), 2983 (s), 2945 (s), 2885 (s), 2655 (s, sp), 2644 (vs, vsp), 2575 (vs), 1990 (w, bd), 1465 (s), 1390 (m), 1305 (m), 1245 (m), 1120 (m) 1040 (m), 985 (vs, sp), 975 (vs, vsp), 853 (m, vsp), 821 (s, sp), 760 (s, sp), 730 (m), 660 (w); 3,4-$(CH_3)_2C_4B_2H_4$ (7 Torr), 3085 (m), 2983 (s, sp), 2943 (s), 2889 (m), 2655 (s, vsp), 2644 (vs, vsp), 2583 (vs), 1460 (m), 1380 (m), 1300 (m), 1240 (m, b), 1125 (m, b), 1050 (m, b), 983 (s), 974 (s, sp), 855 (m), 820 (m), 795 (w), 760 (w).

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