

transitions ( ${}^2T_{2g} \rightarrow {}^2A_{2g}$  and  ${}^2T_{2g} \rightarrow {}^2T_{1g}$ ) which are of almost equal energy.

Above 26 kK lies a broad, intense absorption with maxima at 28.3, 32.1, and 37.1 kK. By analogy with similar low-spin  $d^5$  systems, for example<sup>11</sup>  $\text{IrCl}_6^{2-}$ , the bands at 28.3 and 37.1 kK most probably represent allowed charge-transfer transitions from filled ligand  $\pi$  orbitals to the  $t_{2g}$  level ( $t_{1u} \rightarrow t_{2g}$  and  $t_{2u} \rightarrow t_{2g}$ ). Although the assignment of the 37.1-kK band as  $\pi \rightarrow e_g$  cannot be entirely ruled out, the high  $Dq$  value renders this rather improbable. Gaussian analysis of the curve in this region is of dubious validity, but the peak at 32.1 kK appears thereby to be quite weak and is therefore more likely to represent a d-d transition than a further charge-transfer band. The peak in fact corresponds

(11) C. K. Jørgensen, *Mol. Phys.*, **2**, 309 (1959).

almost exactly with the calculated position of a higher  ${}^2T_{2g} \rightarrow {}^2E_g$  excitation. It is possible that the peak at 24.4 kK assigned as  ${}^2T_{2g} \rightarrow {}^2T_{1g}$  could also be due to a  $\pi \rightarrow t_{2g}$  transition, but, here again, the small  $F_R$  value makes the d-d assignment more acceptable.

We have derived as before<sup>1</sup> approximate values for the extinction coefficients and oscillator strengths of the d-d bands, and the values thus obtained are given in Table I. The intensities of the  ${}^2A_{2g}$  and  ${}^2T_{1g}$ ,  ${}^2T_{2g}$ , and  ${}^2T_{1g}$  bands are all reasonable for Laporte-forbidden transitions, and the  ${}^4T_{1g}$  and  ${}^4T_{2g}$  bands are as weak as expected for spin-forbidden excitations. The actual intensities of the spin-forbidden bands are rather less in  $\text{CoF}_6^{2-}$  than in  $\text{NiF}_6^{2-}$ , possibly owing to the greater energy gap between the interacting states in the cobalt compound.

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## Phosphorus-Phosphorus Spin-Spin Coupling in Metal Carbonyl Complexes of Phosphorus Trifluoride

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Fluorine nmr parameters are reported for  $\text{M}(\text{CO})_6\text{PF}_3$ , *cis*- $\text{M}(\text{CO})_4(\text{PF}_3)_2$ , and *trans*- $\text{M}(\text{CO})_4(\text{PF}_3)_2$  (where M = Cr, Mo, and W) and for  $\text{Fe}(\text{CO})_4\text{PF}_3$ ,  $\text{Fe}(\text{CO})_3(\text{PF}_3)_2$ , and  $\text{Ni}(\text{CO})_2(\text{PF}_3)_2$ . Values of  ${}^2J_{\text{PF}}$  for the disubstituted complexes are discussed in terms of the relatively large degree of s character expected in the phosphorus-metal bond compared to that in analogous complexes of other trivalent phosphorus ligands.

### Introduction

In another paper<sup>2</sup> we report  ${}^{31}\text{P}$ - ${}^{31}\text{P}$  coupling constants for over 30 complexes containing two phosphorus ligands. Because ligands such as  $\text{P}(\text{N}(\text{CH}_3)_2)_3$ ,  $\text{P}(\text{CH}_3)_3$ ,  $\text{P}(\text{OCH}_3)_3$ , and  $\text{P}(\text{OCH}_2)_3\text{CCH}_3$  used in that study contained from 6 to 18 protons, the  ${}^1\text{H}$  nmr spectra of the complexes could be classed as  $\text{X}_n\text{AA}'\text{X}'_n$  systems where  $n$  is the number of protons and X and A represent hydrogen and phosphorus, respectively. In all these systems except  $\text{P}(\text{CH}_3)_3$  the coupling constants  $J_{\text{XX}'}$  and  $J_{\text{AX}'}$  could be assumed to be zero owing to the three-bond separation of the A and X nuclei in the ligands and the magnitude of  ${}^2J_{\text{PF}}$  was calculated from the  ${}^1\text{H}$  nmr spectra using a computer program to simulate the observed envelope of the unresolved lines.

Recent interest<sup>3</sup> in the unusual ligand properties of  $\text{PF}_3$  in metal carbonyls made it attractive to determine the  ${}^{183}\text{W}$ - ${}^{31}\text{P}$  coupling constant in  $\text{W}(\text{CO})_6\text{PF}_3$  reported elsewhere<sup>4</sup> and the  ${}^{31}\text{P}$ - ${}^{31}\text{P}$  spin-spin interaction constants herein reported, inasmuch as the magnitude of such couplings is undoubtedly related to the nature of

the bonding between the coupling nuclei. The  ${}^{19}\text{F}$  nmr spectra of the  $\text{PF}_3$  complexes are still representative of the  $\text{X}_n\text{AA}'\text{X}'_n$  system where  $n = 3$  but the separation of the X (fluorine) nuclei from the A (phosphorus) nucleus by only one bond allows significant coupling of fluorines on one  $\text{PF}_3$  ligand with those on the other ( $J_{\text{XX}'}$ ) as well as with the phosphorus on the second ligand ( $J_{\text{AX}'} = J_{\text{XA}'}$ ).

### Experimental Section

The compounds were prepared and separated by simple variations on procedures used to prepare these compounds earlier.<sup>5-7</sup> The iron compounds were prepared by the high-pressure thermal reaction between  $\text{Fe}(\text{CO})_5$  and  $\text{PF}_3$  and separated by glpc on a  $7\text{ m} \times 1/4$  in. 40% DC-702 silicone column on Chromosorb P. The group VI compounds<sup>6</sup> were prepared by the irradiation of  $\text{M}(\text{CO})_6$  and  $\text{PF}_3$  in about 1:2 molar ratios in a sealed Pyrex vessel which was heated to about 80-100°. Irradiation with an AH-6 1000-W lamp for 2-3 hr served to convert 1-2 g of  $\text{M}(\text{CO})_6$  into a mixture of liquid intermediates. Through the use of this technique, the distribution of species could be controlled in overall composition to approximately  $\text{M}(\text{CO})_{4.5}(\text{PF}_3)_{1.5}$ . The complexes were separated on the 7-m silicone column. Alternately, shorter columns were used to obtain compositional cuts of mono-

(1) Alfred P. Sloan Fellow.

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and disubstituted complexes, and the cuts of disubstituted complexes were separated with a second pass on the longer column. The chromium compounds are considerably more difficult to separate than the heavier analogs. Data for the *cis*- and *trans*-bis(trifluorophosphines) were obtained from mixtures of these two species. The nickel complexes<sup>7</sup> were prepared thermally from PF<sub>3</sub> and Ni(CO)<sub>4</sub> and isolated on the 7-m silicone column. Since the nickel complexes readily disproportionate, they were kept at -196° until immediately before the spectra were run.

The <sup>19</sup>F spectra were obtained on Varian HA-60 and HR-60 spectrometers at 56.4 MHz. The samples were either liquids at room temperature or low-melting solids that would dissolve in small amounts of solvent. Samples available in adequate quantities were run neat using microcells while samples of insufficient volume were diluted with cyclohexane. CFC<sub>3</sub> was used as an internal standard. The <sup>31</sup>P-<sup>31</sup>P coupling constants were calculated from the <sup>19</sup>F spectra using the method of Harris and Woodman.<sup>8</sup>

### Results and Discussion

Although the changes in the <sup>19</sup>F chemical shifts among the complexes of the same metal are relatively small, a consistent decrease in shielding is observed in all cases from monosubstituted to *trans* to *cis*. Because of the subtle factors capable of affecting such small changes in the dominant paramagnetic contribution to <sup>19</sup>F shielding,<sup>9</sup> it is presently not possible to interpret this trend.

While the <sup>19</sup>F spectra of the monosubstituted complexes are characterized by a simple doublet due to P-F coupling, the general appearance of all of the spectra of the disubstituted metal carbonyls is illustrated for the case of *cis*-W(CO)<sub>4</sub>(PF<sub>3</sub>)<sub>2</sub> in Figure 1. From the sep-

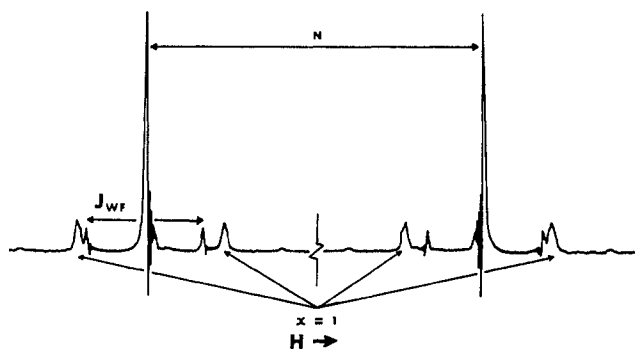


Figure 1.—The <sup>19</sup>F nmr spectrum of *cis*-W(CO)<sub>4</sub>(PF<sub>3</sub>)<sub>2</sub>.

arations of the major peaks (*N*) and the most intense four secondary line groups ( $\chi = 1$ ), it was possible to calculate <sup>2</sup>J<sub>PF</sub>, <sup>1</sup>J<sub>PF</sub>, and <sup>3</sup>J<sub>PF</sub>. Theoretically there are two more sets of four line groups corresponding to  $\chi = 2$  and  $\chi = 3$ . These are much less intense and in many cases cannot be seen. If there is long-range fluorine-fluorine coupling, then each of the four line groups corresponding to  $\chi = 1$  should be a quartet. In many of the complexes reported here such long-range coupling was observed but the quartet could not be re-

solved sufficiently to calculate <sup>4</sup>J<sub>FF</sub>. Thus we can set an upper limit to <sup>4</sup>J<sub>FF</sub> in these complexes of 1 Hz.

The nmr data for the complexes are given in Table I. Although the relative signs of <sup>1</sup>J<sub>PF</sub> and <sup>3</sup>J<sub>PF</sub> could be calculated directly from the <sup>19</sup>F spectrum, an attempt to determine the signs of <sup>1</sup>J<sub>WP</sub> and <sup>2</sup>J<sub>WF</sub> relative to <sup>1</sup>J<sub>PF</sub> in W(CO)<sub>5</sub>PF<sub>3</sub> by double-resonance inord techniques was unsuccessful because our frequency synthesizer did not generate sufficient power at 2.47 MHz to irradiate <sup>183</sup>W nor was a suitable amplifier available. The relative signs of <sup>1</sup>J<sub>WP</sub> and <sup>2</sup>J<sub>WF</sub>, however, were determined from a double-resonance inord experiment. Thus while the spectrometer remained at the frequency of the lowest field member of the four-line <sup>19</sup>F resonance due to <sup>183</sup>W, the <sup>31</sup>P region was scanned with the irradiating frequency. A similar experiment involving the highest field member of the <sup>19</sup>F spectrum showed that the relative signs were the same.

Although only the relative signs of <sup>1</sup>J<sub>PF</sub> and <sup>3</sup>J<sub>PF</sub> can be determined from the spectra, it is probable that <sup>1</sup>J<sub>PF</sub> is negative.<sup>10</sup> The magnitudes of <sup>2</sup>J<sub>PF</sub> of the PF<sub>3</sub> complexes and those of the corresponding phosphine and phosphite complexes<sup>2</sup> fall in the general order PR<sub>3</sub> < P(OR)<sub>3</sub> < PF<sub>3</sub>. A similar trend has been noted for <sup>1</sup>J<sub>WP</sub> in W(CO)<sub>6</sub>L complexes.<sup>4,11</sup> These results can be rationalized on the proposal that the dominant spin-spin coupling mechanism involves bonding orbitals having nonzero spin density at the nucleus. Thus substantial evidence has been reported, both theoretical<sup>12-16</sup> and experimental,<sup>14,15</sup> that the greater the s character in the bond, the greater the coupling between the bonded nuclei. These arguments have been applied<sup>15</sup> to trends in directly bonded phosphorus nuclei in diphosphorus compounds and we believe it valid to extend them here to phosphorus atoms separated by a metal.

The increase in s character in the order PR<sub>3</sub> < P(OR)<sub>3</sub> < PF<sub>3</sub> can be understood using the arguments of Bent<sup>15</sup> which consider the increase in electronegativity and lower steric requirement of fluorine compared to the R or OR phosphorus substituents. Indeed the CPC bond angle in P(CH<sub>3</sub>)<sub>3</sub> (98.6°)<sup>17a</sup> is larger than the recently determined FPF angle in PF<sub>3</sub> (97.8°).<sup>17b</sup> Inasmuch as we are dealing with four-coordinate phosphorus in these complexes, it would appear perhaps more appropriate to examine what few data are available for ligands of this type attached to an acceptor. In complexes of the type PR<sub>3</sub>, CPC bond angles are found to

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TABLE I  
 NMR DATA FOR PF<sub>3</sub> COMPLEXES

Chem shift relative to CFCls, ppm	<sup>1</sup> J <sub>PF</sub> , Hz	<sup>2</sup> J <sub>PF</sub> , Hz	<sup>2</sup> J <sub>PP</sub> , <sup>a</sup> Hz
Cr(CO) <sub>5</sub> PF <sub>3</sub>	2.06	1315	
<i>cis</i> -Cr(CO) <sub>4</sub> (PF <sub>3</sub> ) <sub>2</sub>	-0.09	±1306	78 (78.5) <sup>b</sup>
<i>trans</i> -Cr(CO) <sub>4</sub> (PF <sub>3</sub> ) <sub>2</sub>	1.28		(34.0) <sup>b</sup>
Mo(CO) <sub>5</sub> PF <sub>3</sub>	4.70	1310	
<i>cis</i> -Mo(CO) <sub>4</sub> (PF <sub>3</sub> ) <sub>2</sub>	3.03	±1306	55 (55.5) <sup>b</sup>
<i>trans</i> -Mo(CO) <sub>4</sub> (PF <sub>3</sub> ) <sub>2</sub>	3.99	±1320	312
W(CO) <sub>5</sub> PF <sub>3</sub> <sup>c</sup>	7.89	1245	
<i>cis</i> -W(CO) <sub>4</sub> (PF <sub>3</sub> ) <sub>2</sub> <sup>d</sup>	5.96	±1281	38 (39.0) <sup>b</sup>
<i>trans</i> -W(CO) <sub>4</sub> (PF <sub>3</sub> ) <sub>2</sub> <sup>e</sup>	6.97	±1286	315
Fe(CO) <sub>4</sub> PF <sub>3</sub>	6.22	1329	
Fe(CO) <sub>3</sub> (PF <sub>3</sub> ) <sub>2</sub>	5.59	±1322	98
Ni(CO) <sub>2</sub> (PF <sub>3</sub> ) <sub>2</sub>	18.87	±1357	38 (39) <sup>b</sup>

<sup>a</sup> Error limits ±1 Hz. <sup>b</sup> J. F. Nixon, private communication. Error limits ±0.5 Hz except for the last compound for which none was specified. <sup>c</sup> <sup>2</sup>J<sub>WF</sub> = 31 Hz. <sup>d</sup> <sup>2</sup>J<sub>WF</sub> = 30 Hz. <sup>e</sup> <sup>2</sup>J<sub>WF</sub> = 32 Hz.

range from 101 to 111°<sup>18,19</sup> In compounds of the type XPR<sub>3</sub> (where X = S, Se, or O) these angles are between 106 and 108°.<sup>18</sup> Structural data for phosphites coordinated to metals are restricted to [Ni(P(OCH)<sub>2</sub>(CH<sub>2</sub>)<sub>3</sub>)](ClO<sub>4</sub>)<sub>2</sub>.<sup>20</sup> This complex contains five bicyclic phosphites in which the average OPO bond angle is 104°. Magnitudes for this angle in the bicyclic systems O=P(OCH<sub>2</sub>)<sub>3</sub>CCH<sub>3</sub><sup>21a</sup> and S=P(OCH)<sub>3</sub>(CH<sub>2</sub>)<sub>3</sub><sup>21b</sup> are 104 and 105°, respectively, while in O=P(OCH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>(OH)<sup>22</sup> it is 104°. No FPF angles for metal PF<sub>3</sub> complexes have appeared as yet but their sizes in O=PF<sub>3</sub>, S=PF<sub>3</sub>, (F<sub>2</sub>B)<sub>3</sub>BPF<sub>3</sub>,<sup>23a</sup> and H<sub>3</sub>BPF<sub>3</sub><sup>23b</sup> are 102.5,<sup>16</sup> 100.3,<sup>16</sup> 101.7,<sup>23a</sup> and 99.8°,<sup>23b</sup> respectively. If the angles between the substituent atoms in the chalcogenide derivatives can be taken as indicative of the trend in bond angles in metal coordination compounds, the order of angles might then reasonably be PR<sub>3</sub> > P(OR)<sub>3</sub> > PF<sub>3</sub> in an analogous series of complexes. If this is indeed the case, the order of s character in the phosphorus-metal bond is MPR<sub>3</sub> < MP(OR)<sub>3</sub> < MPF<sub>3</sub> which parallels the increasing order of <sup>31</sup>P-<sup>31</sup>P coupling constants.

An additional and perhaps dominating influence in the rise of <sup>2</sup>J<sub>PP</sub> with the electronegativity of the phosphorus substituent is that attributable to the increase in phosphorus nuclear charge in the order MPR<sub>3</sub> < MP(OR)<sub>3</sub> < MPF<sub>3</sub>. Grant and Litchman<sup>24</sup> have pointed out on theoretical grounds that the effective nuclear charge appears as a cubed term in the coupling equation for a <sup>13</sup>C-H bond whereas that due to the per cent s

character is only squared. The work of Roberts and coworkers<sup>25</sup> on <sup>13</sup>C couplings in organometallic compounds supports this assertion quite well. Because the per cent s character and effective charge arguments complement one another, further work is necessary before the relative importance of these effects can be evaluated in coordination compounds of phosphorus ligands. A serious complicating factor is the present lack of an adequate theory for couplings in transition metal complexes of second-row elements.

Although π bonding very probably exists in the phosphorus-metal bond, we presently favor the s character interpretation of the spin-spin coupling data. The synergic effect of π bonding in strengthening the σ bond between the phosphorus lone-pair orbital and the metal may well be important<sup>11</sup> but available data do not permit its assessment. The question of the possible role of π bonding in the spin-spin coupling mechanism<sup>11</sup> is discussed elsewhere.<sup>2,4</sup>

Where both *cis* and *trans* isomers are known, <sup>2</sup>J<sub>PP</sub> has always been found to be larger in the *trans* isomer except for Cr(CO)<sub>4</sub>L<sub>2</sub>.<sup>2</sup> We note that PF<sub>3</sub> apparently also follows this "rule" although it is not yet clear why the reversal with chromium takes place with all of the ligands studied so far. The observation that <sup>2</sup>J<sub>PP</sub> *trans* is greater than <sup>2</sup>J<sub>PP</sub> *cis* for the molybdenum and tungsten PF<sub>3</sub> complexes is perhaps best explained by the fact that the σ-bonding lone pairs of *trans* phosphorus nuclei share the same metal p<sub>σ</sub> molecular orbital whereas those *cis* do not. Thus <sup>31</sup>P-<sup>31</sup>P coupling may be enhanced by overlap of the two phosphorus lone-pair orbitals with a common metal orbital in the *trans* case.

The one case in which <sup>2</sup>J<sub>PP</sub> for PF<sub>3</sub> is substantially less than that for the corresponding complex with a phosphite is the iron compound. The value for <sup>2</sup>J<sub>PP</sub> in *trans*-Fe(CO)<sub>3</sub>(P(OCH<sub>2</sub>)<sub>3</sub>CC<sub>2</sub>H<sub>5</sub>)<sub>2</sub> is 300 ± 40 Hz which contrasts sharply with the value of 98 Hz for Fe(CO)<sub>3</sub>(PF<sub>3</sub>)<sub>2</sub>. However, the iron spectra are almost certainly time averaged because of equivalence of the PF<sub>3</sub> ligands. Infrared evidence<sup>5,26,27</sup> clearly indicates

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that all possible trigonal-bipyramidal isomers are present, although nmr work has shown no sign of  $PF_3$  nonequivalence.<sup>28</sup> On the basis of the calculated 21% *cis*, 23% *trans*, and 56% *vic* isomer composition,<sup>16</sup> an average angle of about 128° is estimated to correspond to the 98 Hz  $^{31}P$ - $^{31}P$  coupling.

**Acknowledgments.**—The authors thank Dr. J. F. Nixon for making available to us the results of his in-

dependent calculations of  $^2J_{PP}$  in several of the  $PF_3$  complexes and R. D. Bertrand for obtaining some of the spectra. F. O. and J. G. V. thank the National Science Foundation for support of this research in the form of grants toward the purchase of the nmr spectrometers. R. J. C. acknowledges the financial assistance of the Atomic Energy Commission.

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## The Polyhedral $B_6C_2H_8$ , $B_7C_2H_9$ , and $B_8C_2H_{10}$ Carboranes and Their C-Monomethyl and C-Monophenyl Derivatives

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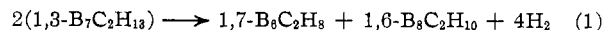
The pyrolysis of 1,3- $B_7C_2H_{13}$  at 215° in the absence of diborane yields the  $B_6C_2H_8$ ,  $B_7C_2H_9$ , and 1,6- $B_8C_2H_{10}$  carboranes, while in the presence of diborane the yield of 1,6- $B_8C_2H_{10}$  is considerably enhanced. Isomerization of 1,6- $B_8C_2H_{10}$  to the 1,10- $B_8C_2H_{10}$  isomer occurs at 300°. Pyrolysis of 1,3- $NaB_7C_2H_{12}$  at 200° yields 1,7- $B_7C_2H_9$ . The C-monomethyl and C-monophenyl derivatives of these carboranes are also described.

Recently, the preparation and characterization of the C,C'-dimethyl derivatives of 1,7-dicarba-*closo*-octaborane(8), 1,7- $B_6C_2H_8$ , 1,7-dicarba-*closo*-nonaborane(9), 1,7- $B_7C_2H_9$ , 1,6-dicarba-*closo*-decaborane(10), 1,6- $B_8C_2H_{10}$ , and 1,10-dicarba-*closo*-decaborane(10), 1,10- $B_8C_2H_{10}$ , were reported.<sup>1,2</sup> The results of X-ray diffraction studies and theoretical treatments of the C,C'-dimethyl derivatives of 1,7- $B_6C_2H_8$  and 1,7- $B_7C_2H_9$  have been reported<sup>3,4</sup> by Lipscomb and coworkers. Dunks and Hawthorne<sup>5</sup> have reported the low-pressure pyrolysis of 1,3- $B_7C_2H_{13}$  at 360° to give a 60% yield of 1,7- $B_6C_2H_8$ . This paper describes the preparation and characterization of the 1,7- $B_6C_2H_8$ , 1,7- $B_7C_2H_9$ , 1,6- $B_8C_2H_{10}$ , and 1,10- $B_8C_2H_{10}$  carboranes and their C-monomethyl and C-monophenyl derivatives.

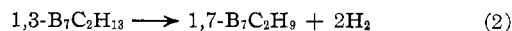
### Results and Discussion

**Pyrolysis of 1,3- $B_7C_2H_{13}$ .**—The thermal decomposition of 1,3-dicarba-*nido*-nonaborane(13) near 215° in diphenyl ether solution produced 1,7- $B_6C_2H_8$ , 1,7- $B_7C_2H_9$ , and 1,6- $B_8C_2H_{10}$  carboranes. The corresponding C-monomethyl derivatives were obtained by the same route while the C-monophenyl derivatives were obtained using benzene solvent. Yield and characterization data are presented in Table I. The almost equivalent yields of 1,7- $B_6C_2H_8$  and 1,6- $B_8C_2H_{10}$  carboranes and the low yield of 1,7- $B_7C_2H_9$  suggests that the pre-

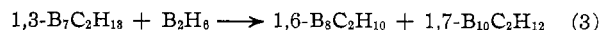
dominant reaction is a disproportionation of 1,3- $B_7C_2H_{13}$  (eq 1) and that 1,7- $B_7C_2H_9$  arises from the elim-



ination of hydrogen from 1,3- $B_7C_2H_{13}$  (eq 2).



When a continuous stream of diborane was passed through a diphenyl ether solution of 1,3- $B_7C_2H_{13}$  at 215°, the yield of 1,6- $B_8C_2H_{10}$  increased at the expense of 1,7- $B_6C_2H_8$  and 1,7- $B_7C_2H_9$ . With excess diborane, 1,6- $B_8C_2H_{10}$  was the predominant product along with only trace amounts of 1,7- $B_6C_2H_8$  and 1,7- $B_7C_2H_9$  and a low yield of 1,7- $B_{10}C_2H_{12}$  (eq 3). Hydroboration of pure



1,6- $B_8C_2H_{10}$  under the same conditions produced 1,7- $B_{10}C_2H_{12}$  in moderate yield<sup>1</sup> and this may account for its appearance among the pyrolysis reaction products. The yield data for the pyrolysis of 1,3- $B_7C_2H_{13}$  and its C-monomethyl and C-monophenyl derivatives in the presence of diborane are presented in Table I. 1- $C_6H_5$ -1,6- $B_8C_2H_9$  could not be successfully prepared by pyrolysis of 1- $C_6H_5$ -1,3- $B_7C_2H_{12}$  in the presence of added diborane.

Thermal rearrangement of 1,6- $B_8C_2H_{10}$  occurs above 300° to give a nearly quantitative yield of the 1,10- $B_8C_2H_{10}$  isomer.<sup>1</sup> The yield and characterization data for 1,10- $B_8C_2H_{10}$  and its C-monomethyl and C-monophenyl derivatives are presented in Table I.

**Isomers of Substituted 1,6- $B_8C_2H_{10}$  Carboranes.**—The infrared and  $^1H$  nmr spectra of 1,7- $B_6C_2H_8$ , 1,7- $B_7C_2H_9$ , 1,6- $B_8C_2H_{10}$ , and 1,10- $B_8C_2H_{10}$  and their C-monomethyl

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