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# Formation of the Decahydro(trimethylphosphine)pentaborate(1−) Anion and an Isomer of Trimethylphosphine–Pentaborane(11)

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The hydride ion reacts with borane compounds as a Lewis base that either undergoes addition/displacement reactions with the boranes or combines with a protonic bridge-hydrogen atom to abstract the proton from the borane. Examples of the first scenario are represented by eqs 1 and 2. A representative example of the

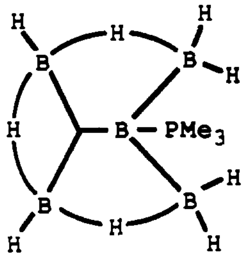
$$B_2H_6 + 2H^- \rightarrow 2BH_4^{-1} \qquad (1)$$

$$B(CH_3)_3 + H^- \rightarrow HB(CH_3)_3^{-1} \qquad (2)$$

second case is seen in the reaction of pentaborane(9) (eq 3).

$$B_5H_9 + H^- \rightarrow B_5H_8^- + H_2{}^3 \qquad (3)$$

The trimethylphosphine adduct of pentaborane(9), $B_5H_9{\cdot}P(CH_3)_3$, is known to combine with a second Lewis base to form a bis(base) adduct such as $B_5H_9{\cdot}2P(CH_3)_3$[4] or $B_5H_9{\cdot}P(CH_3)_3{\cdot}N(CH_3)_3$,[4,5] and as seen in the illustration, its molecule contains bridge-hydrogen atoms[4] that may be protonic enough to be abstracted by the hydride ion. It was of interest to find the course of $B_5H_9{\cdot}P(CH_3)_3$ reaction with the hydride ion. The recently formulated, facile preparative method for $B_5H_9{\cdot}P(CH_3)_3$[5] prompted us to undertake this investigation.



$B_5H_9{\cdot}P(CH_3)_3$

## Results

**A. Reaction of $B_5H_9{\cdot}P(CH_3)_3$. (a) With KH.** When $B_5H_9{\cdot}P(CH_3)_3$ and excess KH were mixed in tetrahydrofuran at room temperature, a slow reaction occurred according to eq 4 to give a pentaborane complex anion, $B_5H_{10}{\cdot}P(CH_3)_3^-$.

$$B_5H_9{\cdot}P(CH_3)_3 + KH \rightarrow K^+[B_5H_{10}{\cdot}P(CH_3)_3^-] \qquad (4)$$

**(b) With $NaHB(sec\text{-}C_4H_9)_3$.** When $B_5H_9{\cdot}P(CH_3)_3$ and $NaHB(sec\text{-}C_4H_9)_3$ were mixed in a 1:1 molar ratio in tetrahydrofuran at room temperature, a hydride-transfer reaction (eq 5) occurred to form the pentaborane complex anion.

$$B_5H_9{\cdot}P(CH_3)_3 + NaHB(sec\text{-}C_4H_9)_3 \rightarrow Na^+[B_5H_{10}{\cdot}P(CH_3)_3^-] + B(sec\text{-}C_4H_9)_3 \qquad (5)$$

**B. Characterization of the $B_5H_{10}{\cdot}P(CH_3)_3^-$ Ion. (a) General Description.** Sodium and potassium salts of the $B_5H_{10}{\cdot}P(CH_3)_3^-$ ion are white solids stable at room temperature in the absence of air. The anion remained unchanged even when a tetrahydrofuran solution of the sodium salt was heated to 60 °C. The salts are slightly soluble in diethyl ether and insoluble in dichloromethane and hydrocarbons.

**(b) NMR Spectra.** The $^{11}B$ spectrum of $Na^+[B_5H_{10}{\cdot}P(CH_3)_3^-]$ in tetrahydrofuran at 20 °C is shown in Figure 1. The two signals are at −25.1 and −59.6 ppm. A triplet feature due to B–H coupling is noted on the −25.1 ppm signal. This triplet appearance is lost at 50 °C. The $^1H\{^{11}B\}$ NMR spectrum of the compound at −20 °C shows signals at 1.20 (terminal protons), 0.36 (terminal protons) and −2.67 ppm (bridge protons) in an intensity ratio of 2:2:1. These three signals coalesce at 60 °C into a single broad signal centered at 0.16 ppm. The methyl proton signal appears as a doublet ($J_{HP}$ = 11 Hz) at 1.04 ppm. The above observations suggest that the structure of the ion is as illustrated in Figure 1 and that the hydrogen atoms exchange positions rapidly at the higher temperatures. The $^{31}P$ signal appears at +6.2 ppm. It has a partially collapsed quartet structure due to the B–P coupling [$J_{BP} \approx$ 100 Hz].

**(c) Reaction with $BCl_3$.** When $NaB_5H_{10}{\cdot}P(CH_3)_3$ was treated with $BCl_3$ in a 1:1 molar ratio in tetrahydrofuran, $B_5H_9{\cdot}P(CH_3)_3$ was regenerated (eq 6).

$$B_5H_{10}{\cdot}P(CH_3)_3^- + BCl_3 \rightarrow B_5H_9{\cdot}P(CH_3)_3 + HBCl_3^- \qquad (6)$$

**(d) Reaction with HCl.** Treatment of $NaB_5H_{10}{\cdot}P(CH_3)_3$ with 1 equiv of anhydrous HCl in tetrahydrofuran yielded $B_4H_8{\cdot}P(CH_3)_3$ and $BH_3{\cdot}THF$ in a 1:1 molar ratio. The formation of the final products was preceded by an unstable intermediate. The $^{11}B$ NMR signals of the intermediate could clearly be observed in the reaction mixture below −75 °C during the early stage of the reaction (see Figure 2). Equations 7 and 8 are appropriate for this reaction.

$$Na^+B_5H_{10}{\cdot}P(CH_3)_3^- + HCl \rightarrow B_5H_{11}{\cdot}P(CH_3)_3 + NaCl \qquad (7)$$

$$B_5H_{11}{\cdot}P(CH_3)_3 + THF \rightarrow B_4H_8{\cdot}P(CH_3)_3 + BH_3{\cdot}THF \qquad (8)$$

**C. Reaction of $B_5H_9{\cdot}P(CH_3)_3$ with KOH.** When $B_5H_9{\cdot}P(CH_3)_3$ was treated with excess KOH powder in tetrahydrofuran, a rapid reaction occurred at room temperature. The product mixture contained the $B_5H_{10}{\cdot}P(CH_3)_3^-$ ion and $B_4H_8{\cdot}P(CH_3)_3$ in a 1:1 molar ratio. A minor quantity of $B_3H_7{\cdot}P(CH_3)_3$ was also detected.

**D. Reaction of $B_5H_9{\cdot}P(CH_3)_3$ with $KOCH_3$.** When $B_5H_9{\cdot}P(CH_3)_3$ was treated with excess $KOCH_3$ in tetrahydrofuran, the $B_5H_{10}{\cdot}P(CH_3)_3^-$ ion was produced. Other identifiable products were $B(OCH_3)_4^-$ and small quantities of $HB(OCH_3)_3^-$ and $BH_3{\cdot}P(CH_3)_3$. However, *$B_4H_8{\cdot}P(CH_3)_3$ was not found in the product.*

## Discussion

**Formation of $B_5H_{10}{\cdot}P(CH_3)_3^-$.** Like $P(CH_3)_3$ or $N(CH_3)_3$, the hydride ion simply added on to $B_5H_9{\cdot}P(CH_3)_3$ to form the $B_5H_9{\cdot}P(CH_3)_3{\cdot}H^-$ anion. Abstraction of bridge-hydrogen atoms did not occur. The hydride-transfer reactions that were observed

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(1) Finholt, A. E.; Bond, A. C., Jr.; Schlesinger, H. I. *J. Am. Chem. Soc.* **1947,** *69,* 1199.

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(4) (a) Long, J. R. Ph.D. Dissertation, The Ohio State University, Columbus, OH, 1973. (b) Kameda, M.; Kodama, G. *Inorg. Chem.* **1982,** *21,* 1267.

(5) Schwarz, A.; Kodama, G. *Inorg. Chem.* **1993,** *32,* 3970.

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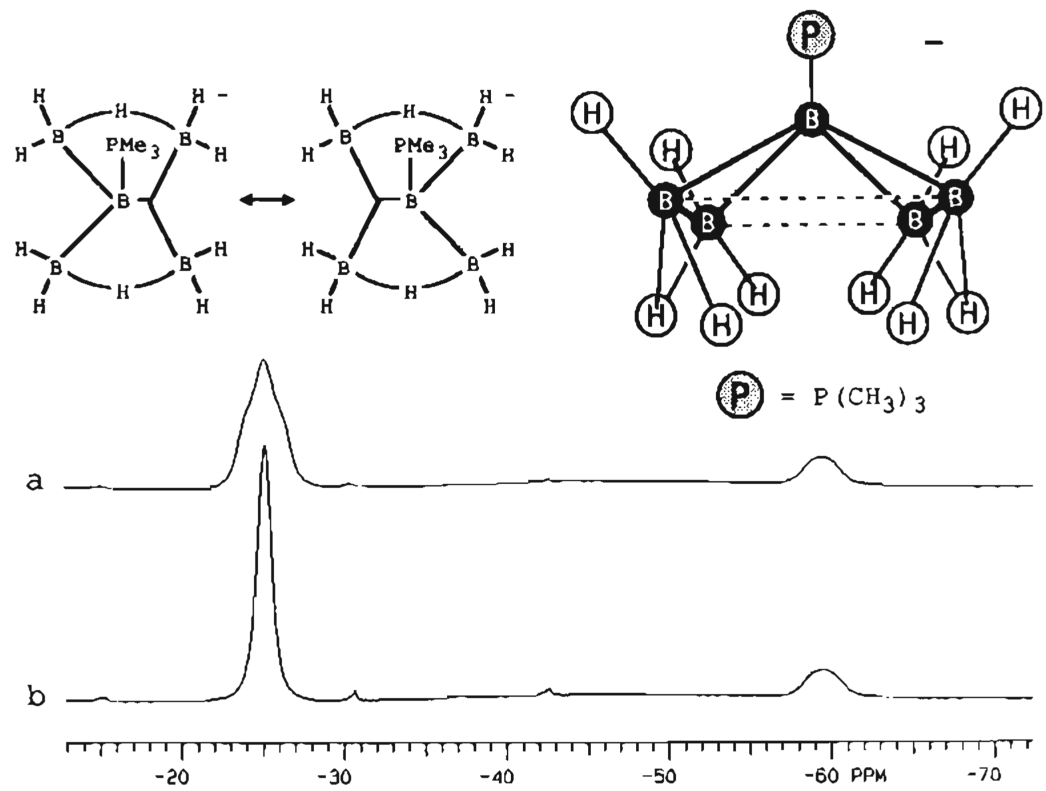

**Figure 1.** $^{11}B$ NMR spectra, proposed structure, and valence-bond representation of the $B_5H_{10}{\cdot}P(CH_3)_3^-$ ion: (a) normal spectrum; (b) proton-spin-decoupled spectrum. Spectral conditions: solvent, tetrahydrofuran-$d_8$; temperature, 20 °C.

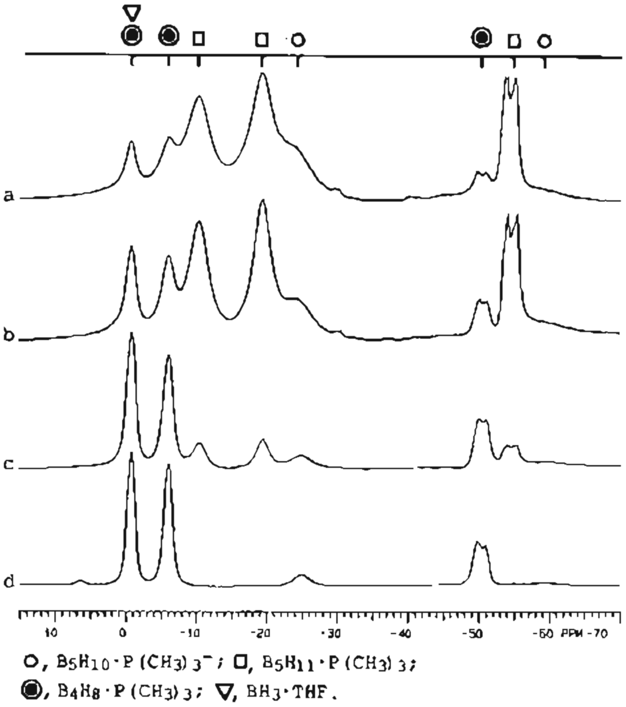

**Figure 2.** $^{11}B$ NMR spectra of a reaction mixture consisting of $B_5H_{10}{\cdot}P(CH_3)_3^-$ and HCl in a 1:1 molar ratio, in tetrahydrofuran: (a) −90 °C; (b) −75 °C; (c) −60 °C; (d) −40 °C.

here (eqs 5 and 6) and elsewhere[5,6] indicated that the hydride affinities of the related compounds increase in the following order: $B(sec\text{-}C_4H_9)_3 < B_5H_9{\cdot}P(CH_3)_3 < B_5H_8{\cdot}2P(CH_3)_3^+ < B_5H_8{\cdot}P(CH_3)_3^+ < BCl_3$.

The observed formation of $B_5H_{10}{\cdot}P(CH_3)_3^-$ in the reaction of $B_5H_9{\cdot}P(CH_3)_3$ with solid KOH is thought to proceed through the addition of $OH^-$ to $B_5H_9{\cdot}P(CH_3)_3$ to form an intermediate "$B_5H_9{\cdot}P(CH_3)_3{\cdot}OH^-$", which then donates its $H^-$ to $B_5H_9{\cdot}P(CH_3)_3$ to produce $B_5H_{10}{\cdot}P(CH_3)_3^-$, as indicated in eqs 9 and 10.

$$B_5H_9{\cdot}P(CH_3)_3 + OH^- \rightarrow \text{“}B_5H_9{\cdot}P(CH_3)_3{\cdot}OH^-\text{”} \quad (9)$$

$$\text{“}B_5H_9{\cdot}P(CH_3)_3{\cdot}OH^-\text{”} + B_5H_9{\cdot}P(CH_3)_3 \rightarrow \text{“}B_5H_8{\cdot}P(CH_3)_3{\cdot}OH\text{”} + B_5H_{10}{\cdot}P(CH_3)_3^- \quad (10)$$

The resulting compound "$B_5H_8{\cdot}P(CH_3)_3{\cdot}OH$" would then be converted into $B_4H_8{\cdot}P(CH_3)_3$ by transferring the protonic OH hydrogen to the apex boron atom and splitting off "HBO", in a manner similar to that in the reaction of $B_5H_9{\cdot}P(CH_3)_3$ with $CH_3OH$, $H_2O$, or HCl[5] (see Scheme 1). Likewise, the reaction with $KOCH_3$ is thought to proceed through the formation of "$B_5H_9{\cdot}P(CH_3)_3{\cdot}OCH_3^-$" which then transfers $H^-$ to $B_5H_9{\cdot}P(CH_3)_3$. The resulting "$B_5H_8{\cdot}P(CH_3)_3{\cdot}OCH_3$", not having a protonic OH hydrogen atom, does not form $B_4H_8{\cdot}P(CH_3)_3$ and appears to undergo yet unidentified decompositions that produce methoxyborates.

**Fluxional Behavior of $B_5H_{10}{\cdot}P(CH_3)_3^-$.** The octahydropentaborate ion ($B_5H_8^-$) and $B_5H_9{\cdot}P(CH_3)_3$ are known to be fluxional in that the bridge- and endo-hydrogen atoms migrate rapidly at the base of the pyramidal structure of the boranes.[3b,4] The exo-hydrogen atoms are bonded rigidly to the basal boron atoms and do not participate in the fluxional motion, whereas in $B_5H_{10}{\cdot}P(CH_3)_3^-$ all hydrogen atoms exchange positions (see Figure 3). The same indiscriminate scrambling of H atoms was observed for $B_5H_9{\cdot}2P(CH_3)_3$,[7] which is isoelectronic with $B_5H_{10}{\cdot}P(CH_3)_3^-$. Stability of the structure that has a dangling $BH_3$ group[8] may be responsible for the endo–exo hydrogen atom exchange (see Scheme 2).

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(6) Kameda, M.; Kodama, G. *Inorg. Chem.* **1987**, *26*, 2011.

(7) Fratini, A. V.; Sullivan, G. W.; Denniston, M. L.; Hertz, R. K.; Shore, S. G. *J. Am. Chem. Soc.* **1974**, *96*, 3013.

(8) An isomeric structure of $B_5H_9{\cdot}2P(CH_3)_3$ that has a dangling $BH_3$ group has been observed by Professor Sheldon G. Shore of The Ohio State University in his X-ray structural study of the compound (private communication).

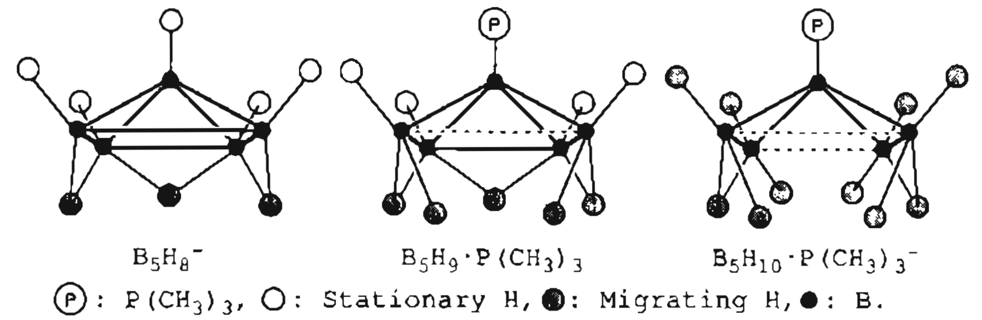
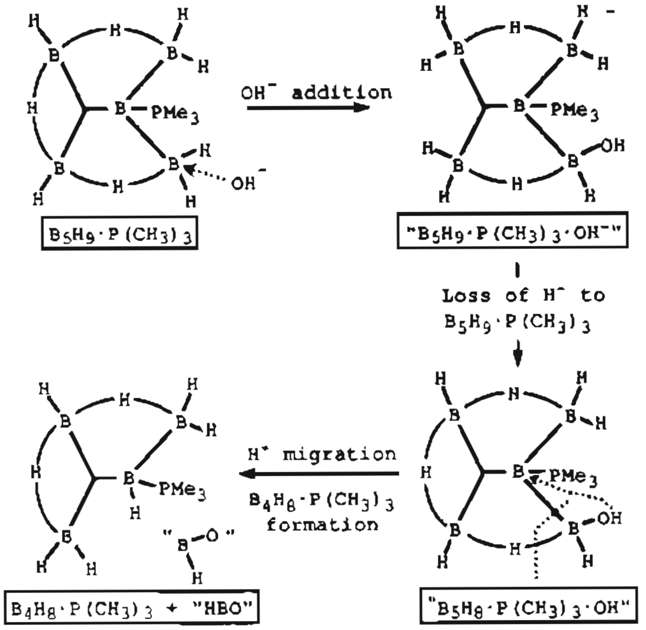


Figure 3. Structures of $B_5H_8^-$, $B_5H_9 \cdot P(CH_3)_3$, and $B_5H_{10} \cdot P(CH_3)_3^-$ indicating the rigid and nonrigid hydrogen atoms.

**Scheme 1**



**Scheme 2**

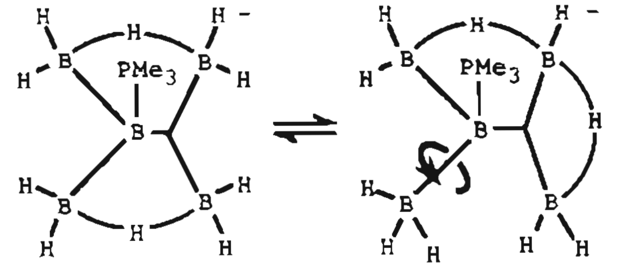


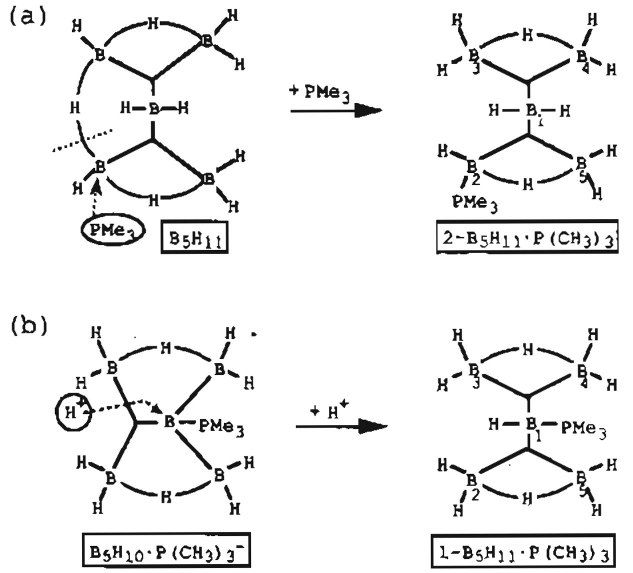
**Table 1.** $^{11}B$ NMR Shift Data for Lewis Base Adducts of $B_5H_{11}$

| adduct | shift [rel intens] (assgnt) | | |
|---|---|---|---|
| $B_5H_{11} \cdot P(CH_3)_3$ (current study) | −9.3 [2] ($B_{4,5 \text{ or } 2,3}$) | −18.3 [2] ($B_{2,3 \text{ or } 4,5}$) | −53.6 [1] ($B_1$) |
| $B_5H_{11} \cdot PH_3$[a] | −6.7 [3] ($B_{3,4,5}$) | −39.4 [1] ($B_2$) | −48.6 [1] ($B_1$) |
| $B_5H_{11} \cdot S(CH_3)_2$[b] | −4.2 [3] ($B_{3,4,5}$) | −19.3 [1] ($B_2$) | −48.8 [1] ($B_1$) |
| $B_5H_{11} \cdot P(CH_3)_3$[c] | −8.8 [3] ($B_{3,4,5}$) | −36.4 [1] ($B_2$) | −53.2 [1] ($B_1$) |

[a] Reference 10. [b] Reference 9. [c] Reference 4a.

**Nature of the Unstable Compound $B_5H_{11} \cdot P(CH_3)_3$.** Several unstable 1:1 Lewis base adducts of $B_5H_{11}$, including $B_5H_{11} \cdot P(CH_3)_3$, have been reported in the literature.[4a,9–11] These compounds were produced by the direct reactions of $B_5H_{11}$ with the respective Lewis bases. Their $^{11}B$ NMR shift data are listed in Table 1. The three signals of each of these adducts are in a 3:1:1 intensity ratio, which is clearly different from that observed for our intermediate, $B_5H_{11} \cdot P(CH_3)_3$.

**Scheme 3**



In the reactions of $B_5H_{11}$ with Lewis bases, the site of base attack is one of the basal boron atoms. Therefore, the structure shown in Scheme 3a was proposed for the earlier adducts,[4a,9,10] and the compounds should be termed as *2-(Lewis base)-pentaboranes (11)*. On the other hand, the addition of a proton to $B_5H_{10} \cdot P(CH_3)_3^-$ is expected to occur at the electron-rich apex boron atom to which $P(CH_3)_3$ is bonded. Therefore, the structure shown in Scheme 3b is proposed for the current isomer of $B_5H_{11} \cdot P(CH_3)_3$ and is described as *1-trimethylphosphine-pentaborane (11)*.

These $P(CH_3)_3$ adducts of $B_5H_{11}$ are isoelectronic with the $B_5H_{12}^-$ anion, which was reported by Shore *et al.* as the product from the reaction of $B_4H_9^-$ with diborane.[12] On the basis of its $^{11}B$ and $^1H$ NMR spectra, a structure similar to those illustrated in Scheme 3 was proposed for the anion. This $B_5H_{12}^-$ anion may be considered to be the $H^-$ adduct of $B_5H_{11}$. Indeed, when treated with HCl, the anion yielded a $H^-$ ion to the acid to form $H_2$ and $B_5H_{11}$.[12]

## Experimental Section

**Procedures and Chemicals.** Conventional high-vacuum-line techniques were used throughout. Reactions were performed in Pyrex tubes equipped with stopcocks. Nonvolatile samples were introduced into the tube, against

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(9) Ishii, M.; Kodama, G. *Inorg. Chem.* **1990**, *29*, 2986.

(10) Jock, C. P.; Kodama, G. *Inorg. Chem.* **1988**, *27*, 3431.

(11) Compounds with the formula $B_5H_{11} \cdot OR_2$, where $OR_2$ = $O(CH_3)_2$, $O(C_2H_5)_2$, or $OC_4H_8$, have also been reported (Kodama, G.; Saturnino, D. J. *Inorg. Chem.* **1975**, *14*, 2243). The $^{11}B$ NMR signals for these species appeared at −17 and −59 ppm in an intensity ratio of 4:1. This is interpreted as a case of base-induced fluxionality of $B_5H_{11}$; high lability of the $B_5H_{11}$–$OR_2$ complexes is thought to be responsible for the equivalency of the four basal boron atoms.

(12) Remmel, R. J.; Johnson, H. D., II; Jaworiwsky, I. S.; Shore, S. G. *J. Am. Chem. Soc.* **1975**, *97*, 5395.

a flow of nitrogen gas, through a side arm which was located just below the stopcock, and then the side arm was sealed with torch. In order to monitor the reaction progress, the tube was placed in the probe of a Varian FT80A or XL-300 NMR spectrometer, and $^{11}B$ spectra were recorded. NMR shift standards were $BF_3{\cdot}O(C_2H_5)_2$ and 85% orthophosphoric acid for $^{11}B$ and $^{31}P$, respectively.

Samples of $B_5H_9{\cdot}P(CH_3)_3$ were prepared by the literature method.[5] Potassium hydride powder was laboratory stock, and its active purity was 62%. Potassium hydroxide powder was prepared by pulverizing reagent grade KOH pellets in a blender. Potassium methoxide was prepared by treating potassium metal with excess anhydrous methyl alcohol in the vacuum line and then pumping out the remaining alcohol at room temperature. The sodium salt of $HB(\textit{sec}\text{-}C_4H_9)_3^-$ (N-Selectride) was obtained from Aldrich Chemical Co. as a 1 M solution in tetrahydrofuran, and its active concentration was determined to be 0.85 M. Solvents were rigorously dried and stored in airtight containers.[13]

**Preparation of $B_5H_{10}{\cdot}P(CH_3)_3^-$ Salts. (a) Reaction of $B_5H_9{\cdot}P(CH_3)_3$ with KH.** A solution containing 163.3 mg (1.17 mmol) of $B_5H_9{\cdot}P(CH_3)_3$ in 3 mL of tetrahydrofuran was prepared in a 20 mm o.d. Pyrex tube, and a 117.6-mg sample of potassium hydride was added onto the frozen solution. The tube was evacuated, sealed off, and shaken for 74 h on a shaker. Then the reaction mixture was filtered, and the solvent was removed from the filtrate by pumping. The resulting solid residue was washed with $CH_2Cl_2$ and then leached with tetrahydrofuran. Removal of tetrahydrofuran from the leachate gave 81 mg (0.45 mmol) of $KB_5H_{10}{\cdot}P(CH_3)_3$ which was contaminated with $KB_3H_8$ (ca. 15%). The above reaction of $B_5H_9{\cdot}P(CH_3)_3$ with KH did not proceed in a toluene solution.

**(b) Reaction of $B_5H_9{\cdot}P(CH_3)_3$ with $NaHB(\textit{sec}\text{-}B_4H_9)_3$.** A 1.13-mL portion of the N-Selectride solution, corresponding to 0.96 mmol of $NaHB(\textit{sec}\text{-}B_4H_9)_3$, was mixed with 7.5 mL of tetrahydrofuran in a 20 mm o.d. Pyrex tube. Then, a 150-mg (1.1 mmol) sample of $B_5H_9{\cdot}P(CH_3)_3$ was placed into the tube above the frozen Selectride solution. The tube was then evacuated, and a 1.5-mL quantity of tetrahydrofuran was condensed in. The mixture was stirred for 2 h at room temperature. During this process no noncondensable gas evolved. The volatile components (tetrahydrofuran and $B(\textit{sec}\text{-}B_4H_9)_3$) were pumped out. The solid residue that resulted was washed with $CH_2Cl_2$ and then leached with tetrahydrofuran. From the leachate 111 mg (0.68 mmol, 71% yield based on the Selectride used) of $NaB_5H_{10}{\cdot}P(CH_3)_3$ was obtained by pumping out the solvent. The product was contaminated with a small amount of $NaB_3H_8$, which could be removed by washing with a small amount of diethyl ether.

The reaction was also run in dichloromethane. The solvent tetrahydrofuran was pumped out from a 1.32-mL sample of the N-Selectride solution in a reaction tube, and the resulting white solid was dissolved in 5 mL of dichloromethane. Then, a 101.9-mg (0.732-mmol) sample of $B_5H_9{\cdot}P(CH_3)_3$ was introduced into the tube while the solution was frozen, and the mixture was allowed to stand at room temperature for 35 min. No noncondensable gas could be detected during this entire process. Formation of a white solid was visible within 5 min at room temperature. The solid was filtered out, washed with $CH_2Cl_2$, and leached with tetrahydrofuran. From the leachate 74 mg (0.45 mmol) of $NaB_5H_{10}{\cdot}P(CH_3)_3$ was obtained. Yield: 62% based on the $B_5H_9{\cdot}P(CH_3)_3$ used.

**Reactions of $NaB_5H_{10}{\cdot}P(CH_3)_3$. (a) With $BCl_3$.** A 28.7-mg (0.245-mmol) sample of $BCl_3$ was condensed into a 9 mm o.d. reaction tube which contained a 38.9-mg (0.238-mmol) sample of $NaB_5H_{10}{\cdot}P(CH_3)_3$ in 3 mL of tetrahydrofuran. The tube was then placed in a −95 °C bath, shaken to mix the reactants, and placed in the cold (−80 °C) probe of the FT80A spectrometer. The $^{11}B$ signals of $B_5H_9{\cdot}P(CH_3)_3$ began to appear when the temperature of the probe was raised to 0 °C. At 20 °C the reaction was complete in a few minutes; the final spectrum consisted of the signals of $B_5H_9{\cdot}P(CH_3)_3$ and a very weak signal of $BCl_3$. A white precipitate (presumably $NaHBCl_3$) was formed in the reaction tube.

**(b) With HCl.** A solution containing 32.3 mg (0.198 mmol) of $NaB_5H_{10}{\cdot}P(CH_3)_3$ in about 3 mL of tetrahydrofuran was frozen in a 14 mm o.d. tube, and a 0.196-mmol sample of anhydrous HCl was condensed into the tube. The tube was sealed, placed in a −95 °C bath, and shaken to mix the reactants. It was then placed in the cold (−90 °C) probe of Varian XL-300 spectrometer, and a series of spectra were recorded as the temperature was increased stepwise. (See Figure 2.) At −60 °C the intermediate disappeared within 15 min, leaving the signals of $B_4H_8{\cdot}P(CH_3)_3$ and $BH_3{\cdot}THF$ and a weak signal of $B_5H_{10}{\cdot}P(CH_3)_3^-$. By this time a white precipitate was visible in the solution. Overlapping of the $BH_3{\cdot}THF$ signal with the low-field signal of $B_4H_8{\cdot}P(CH_3)_3$ was confirmed by separating $BH_3{\cdot}THF$ from the reaction mixture. When excess HCl was used, $B_3H_7{\cdot}P(CH_3)_3$, $BH_2Cl{\cdot}THF$, and a small amount of $BHCl_2{\cdot}THF$ were produced.

**Formation of the $B_5H_{10}{\cdot}P(CH_3)_3^-$ Anion. (a) From $B_5H_9{\cdot}P(CH_3)_3$ and KOH.** A 0.27-g quantity of powdered potassium hydroxide and about 4 mL of tetrahydrofuran were placed in a 9 mm o.d. tube, and a 52.8-mg (0.379-mmol) sample of $B_5H_9{\cdot}P(CH_3)_3$ was added to the frozen mixture of KOH–THF. Then the reaction mixture was shaken at room temperature. The $B_5H_{10}{\cdot}P(CH_3)_3^-$ ion and $B_4H_8{\cdot}P(CH_3)_3$ formed steadily in a 1:1 molar ratio, and the change became unnoticeable within 50 min. In addition to the two products, small amounts of $B_3H_7{\cdot}P(CH_3)_3$ and $BH_3{\cdot}P(CH_3)_3$ were detected.

**(b) From $B_5H_9{\cdot}P(CH_3)_3$ and $KOCH_3$.** A 51.6-mg (0.371-mmol) sample of $B_5H_9{\cdot}P(CH_3)_3$ was treated with a large excess (>1 mmol) of $KOCH_3$ in a manner similar to that described above for the KOH reaction. The reaction was complete within 30 min. The major products were $B_5H_{10}{\cdot}P(CH_3)_3^-$ and $B(OCH_3)_4^-$ [$\delta(^{11}B)$ 3.3 ppm] in about 1.0:1.4 molar ratio. Small quantities of $HB(OCH_3)_3^-$ [$\delta(^{11}B)$ 7.9 ppm], $B_3H_8^-$, and $BH_3{\cdot}P(CH_3)_3$ were also present in the product mixture.

**Acknowledgment.** We are grateful to the German Academic Exchange Service (DAAD) for financial support to P.G. for his study abroad at the University of Utah.

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(13) Kameda, M.; Shimoi, M.; Kodama, G. *Inorg. Chem.* **1984**, *23*, 3705. See also ref 5.