shell of Fe atoms in the Fe EXAFS of the $[Fe_6S_6(L)_6(Mo(CO)_3)_2]^{n-}$ adducts.

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Supplementary Material Available: Tables S2-S5, listing positional coordinates for all non-hydrogen atoms, thermal parameters, and the derived hydrogen positions for I-IV (13 pages); tables of calculated and observed structure factors for I-IV (47 pages). Ordering information is given on any current masthead page.

Notes

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Protic Acid Behavior of Phosphine-Triborane(7)

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The protic acid behavior of phosphine-borane(3) ($BH_3 \cdot PH_3$) has been demonstrated by its reaction with ammonia to give $NH_4^+[BH_3 \cdot PH_2]^-$[1,2] and by its reaction with $NaBH_4$ in dimethoxyethane at 0 °C to produce hydrogen gas and $Na^+[BH_3 \cdot PH_2 \cdot BH_3]^-$.[3] In the reaction with ammonia a further change occurs at a higher temperature (-45 °C) to finally give an ammonium salt of the $[BH_3 \cdot PH_2 \cdot BH_3]^-$ anion.[1,2]

The observed enhanced protonic character of the PH_3 hydrogen atoms is due to the coordination of a BH_3 fragment to the PH_3. Since the B_3H_7 fragment is a stronger acid than BH_3,[4,5] phosphine-triborane(7) ($B_3H_7 \cdot PH_3$) is expected to behave as a stronger protic acid than $BH_3 \cdot PH_3$. Conversely, the conjugate base $[B_3H_7 \cdot PH_2]^-$ should be weaker than $[BH_3 \cdot PH_2]^-$. In this paper, the reactions of $B_3H_7 \cdot PH_3$ with ammonia and with $NaBH_4$ are described and compared with those of $BH_3 \cdot PH_3$ in order to delineate the similarities and differences between the acid-base reaction chemistries of $B_3H_7 \cdot PH_3$ and $BH_3 \cdot PH_3$.

Results

A. Formation of $[B_3H_7 \cdot PH_2]^-$. $B_3H_7 \cdot PH_3$ in Liquid Ammonia. In Figure 1, the ^{11}B and ^{31}P NMR spectra of a liquid ammonia solution of $B_3H_7 \cdot PH_3$ (ca. 0.5 M) are shown and compared with those of $B_3H_7 \cdot PH_3$ in dichloromethane. The ^{31}P spectra indicated that, upon dissolution of $B_3H_7 \cdot PH_3$ in liquid ammonia, the PH_3 moiety of the molecule was converted to a PH_2^- unit: the ^{31}P signal is a 1:2:1 triplet of a 1:1:1:1 quartet. The ^{11}B spectra indicated that the B_3H_7 group remained intact: the two ^{11}B signals appear in a 2:1 intensity ratio. These spectra remained unchanged as long as the solution was kept below -45 °C. When the solvent ammonia was removed by pumping, the original $B_3H_7 \cdot PH_3$ was recovered as residue. Thus, the following equation is appropriate for the interaction of $B_3H_7 \cdot PH_3$ with ammonia:

$$B_3H_7 \cdot PH_3 + NH_3 \rightleftharpoons NH_4^+ + [B_3H_7 \cdot PH_2]^- \qquad (1)$$

At -45 °C, the solute underwent a slow change to give the $[B_3H_7 \cdot PH_2 \cdot BH_3]^-$ anion (see part B of this section) and as yet unidentified species indicated by the ^{11}B signals at -15 and -48 ppm.

Isolation of the Sodium Salt of $[B_3H_7 \cdot PH_2]^-$. Phosphine-triborane(7) could be deprotonated by the use of $Na^+[HB(sec\text{-}Bu)_3]^-$ (eq 2 and 3). The salt $Na^+[B_3H_7 \cdot PH_2]^-$, which was soluble in tetrahydrofuran and dichloromethane, could be isolated as a white solid by pumping out the solvent and $B(sec\text{-}Bu)_3$ from the reaction mixture below -45 °C. The major features of the NMR spectra of $Na^+[B_3H_7 \cdot PH_2]^-$ are identical with those of the ammonia solution of $B_3H_7 \cdot PH_3$ in Figure 1. The spectral data of this anion and its related compounds are summarized in Table I. Treatment of a solution of this salt with anhydrous hydrogen chloride regenerated $B_3H_7 \cdot PH_3$. The $B_3H_7 \cdot PH_2^-$ anion decomposed above -45 °C to form the $[B_3H_7 \cdot PH_2 \cdot BH_3]^-$ anion and another unidentified species, which showed a broad ^{11}B signal ($\nu_{1/2}$ = 250 Hz) centered at -35.5 ppm.

$$B_3H_7 \cdot PH_3 + Na^+[HB(sec\text{-}Bu)_3]^- \xrightarrow{-45\ ^\circ C \text{ in THF}} Na^+[B_3H_7 \cdot PH_2 \cdot B(sec\text{-}Bu)_3]^- + H_2 \qquad (2)$$

$$Na^+[B_3H_7 \cdot PH_2 \cdot B(sec\text{-}Bu)_3]^- \xrightarrow{-B(sec\text{-}Bu)_3/\text{pumping}} Na^+[B_3H_7 \cdot PH_2]^- + B(sec\text{-}Bu)_3 \qquad (3)$$

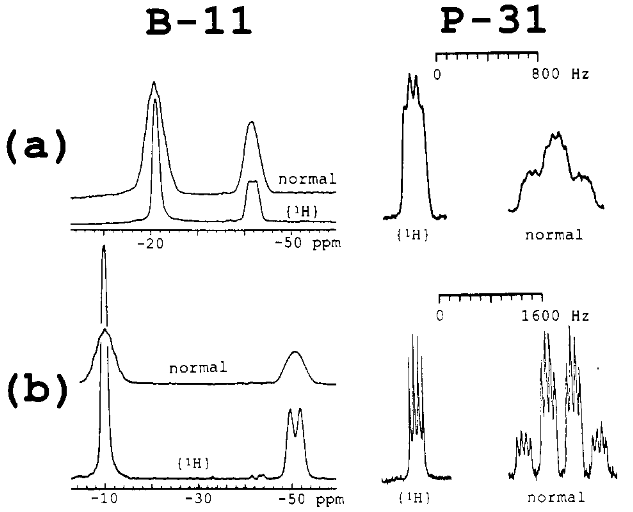

Figure 1. ^{11}B and ^{31}P NMR spectra (32.1 and 40.5 MHz, respectively) of $B_3H_7 \cdot PH_3$ (a) in ammonia at -70 °C and (b) in CH_2Cl_2 at +25 °C, recorded on a Varian XL-100-15 spectrometer.

Several attempts were made to prepare the salts of the $[B_3H_7 \cdot PH_2]^-$ anion by other methods, which included treatments of ammonia solutions of $B_3H_7 \cdot PH_3$ with sodium metal and with $NaNH_2$, treatment of $B_3H_7 \cdot PH_3$ with *n*-butyllithium in hexane, and treatment of $B_3H_7 \cdot THF$ with KPH_2 in diglyme. These attempts, however, were unsuccessful. Apparently, the B_3H_7 moiety undergoes reactions with these reagents. The $B_3H_7 \cdot PH_2^-$ ion could be detected only in the ^{11}B NMR spectrum of the $NaNH_2$ reaction solution (usually in <40% yields). It, however, could not be isolated from the product mixture.

B. Formation of $[B_3H_7 \cdot PH_2 \cdot BH_3]^-$. Phosphine-triborane(7) reacted with $NaBH_4$ in tetrahydrofuran at -45 °C according to eq 4. The product $Na^+[B_3H_7 \cdot PH_2 \cdot BH_3]^-$, a white solid, was very

$$B_3H_7 \cdot PH_3 + Na^+BH_4^- \rightarrow Na^+[B_3H_7 \cdot PH_2 \cdot BH_3]^- + H_2 \qquad (4)$$

soluble in tetrahydrofuran, and was stable at room temperature in the absence of air. The anion $[B_3H_7 \cdot PH_2 \cdot BH_3]^-$ was inert toward strong Lewis bases, as prolonged contact with $P(CH_3)_3$ at ambient temperature left the anion unchanged.

The formation of the $[B_3H_7 \cdot PH_2 \cdot BH_3]^-$ anion was accompanied by a side reaction that produced NaB_3H_8 (eq 5). At -45 °C the

$$B_3H_7 \cdot PH_3 + Na^+BH_4^- + THF \rightarrow Na^+B_3H_8^- + BH_3 \cdot THF + PH_3 \qquad (5)$$

participation of this side reaction was slight. At higher temperatures, however, it became increasingly significant. When

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Table I. NMR Data for Phosphine–Triborane(7) and Related Compounds[a]

		chem shift, ppm			
		B_3H_7		BR₃ or BH₃	
compd	solvent/temp, °C	$^{11}B_1$	$^{11}B_{2,3}$	^{11}B	^{31}P
$B_3H_7{\cdot}PH_3$[b]	CH_2Cl_2/+25	–51.3 J_{BP} = 70 Hz	–10.2		–96.3 J_{PH} = 400 Hz J_{PB} = 70 Hz
$B_3H_7{\cdot}PH_3$	liquid NH_3/–70	–42.2 J_{BP} = 45 Hz	–21.6		–193.6 J_{PH} = 180 Hz
$Na^+B_3H_7{\cdot}PH_2^-$	THF/–70	–42.2 J_{BP} = 45 Hz	–21.3		
	CH_2Cl_2/–45	–42.4 J_{BP} = 55 Hz	–20.4		–187 J_{PH} = 220 Hz
$Na^+B_3H_7{\cdot}PH_2{\cdot}BR_3^-$	THF/+25	–45.2	–18.5	+25.7	–76.5[c] J_{PH} = 300 Hz $J_{PB} \approx$ 80 Hz
$Na^+B_3H_7{\cdot}PH_2{\cdot}BH_3^-$	THF/+25	–46.6 J_{BP} = 88 Hz	–16.4	–40.2 J_{BP} = 50 Hz J_{BH} = 92 Hz	–85 J_{PH} = 400 Hz $J_{PB(1)}$ = 88 Hz
$BH_3{\cdot}PH_3$[d]	neat/+37			–42.8 J_{BP} = 28 Hz J_{BH} = 103 Hz	–113 J_{PH} = 380 Hz
$BH_3{\cdot}PH_2^-$[e]	ND_3			–37.6 J_{BP} = 29 Hz J_{BH} = 90 Hz	–202.6[f] J_{PH} = 175 Hz
$NH_4^+BH_3{\cdot}PH_2{\cdot}BH_3^-$[g]	H_2O			–41.8 J_{BP} = 60 Hz J_{BH} = 91.2 Hz	–117 J_{PH} = 320 Hz

[a] Reference standards are $BF_3{\cdot}O(C_2H_5)_2$ for boron and 85% orthophosphoric acid for phosphorus. [b] Reference 5. [c] At –60 °C. [d] Reference 16. [e] Reference 2. [f] $BH_3{\cdot}PH_3$ in liquid ammonia at –60 °C; this work. [g] Reference 1.

diethyl ether was used as the solvent, the formation of $B_3H_8^-$ was the major reaction.

The ^{11}B and ^{31}P NMR data of the $[B_3H_7{\cdot}PH_2{\cdot}BH_3]^-$ anion are included in Table I. The large triplet feature of the ^{31}P signal and a multiplet fine structure in the ^1H-spin-decoupled ^{31}P signal (a 1:1:1:1 quartet of a 1:1:1:1 quartet) confirm the presence of a H_2PBB' moiety, and the doublet of a quartet structure of the ^{11}B signal to –40.2 ppm shows the presence of a BH_3 group that is bonded to the phosphorus atom. The relative intensity ratio of the signal at –16.4 ppm and the doublet signal at –46.6 ppm is 2:1, suggesting the presence of a triborane(7) fragment attached to the phosphorus atom.

Anhydrous hydrogen chloride reacted with $Na^+[B_3H_7{\cdot}PH_2{\cdot}BH_3]^-$ in tetrahydrofuran at 0 °C according to eq 6.

$$Na^+[B_3H_7{\cdot}PH_2{\cdot}BH_3]^- + HCl + THF \rightarrow B_3H_7{\cdot}PH_2{\cdot}BH_2{\cdot}THF + NaCl + H_2 \quad (6)$$

Trimethylamine displaced tetrahydrofuran from $B_3H_7{\cdot}PH_2{\cdot}BH_3{\cdot}THF$ at –45 °C to give $B_3H_7{\cdot}PH_2{\cdot}BH_3{\cdot}N(CH_3)_3$. At higher reaction temperatures, the strong base cleaved the B_3H_7 moiety.

Discussion

Reaction of $B_3H_7{\cdot}PH_3$ with Ammonia. The protic acid behavior of $B_3H_7{\cdot}PH_3$ shown in eq 1 is similar to that of $BH_3{\cdot}PH_3$ (eq 7).[1,2]

$$BH_3{\cdot}PH_3 + NH_3 \rightleftharpoons NH_4^+ + [BH_3{\cdot}PH_2]^- \quad \text{(below –45 °C)} \quad (7)$$

Both $B_3H_7{\cdot}PH_3$ and $BH_3{\cdot}PH_3$ are weak protic acids and can be recovered from the solutions by pumping out the solvent. However, at higher temperatures the reactions that occur are different for the two systems.

Parry and co-workers[1,2] showed that the formation of the $[BH_3{\cdot}PH_2{\cdot}BH_3]^-$ anion is the result of the PH_3 displacement from $BH_3{\cdot}PH_3$ by a strong Lewis base, $[BH_3{\cdot}PH_2]^-$.

$$[BH_3{\cdot}PH_2]^- + BH_3{\cdot}PH_3 \rightarrow [BH_3{\cdot}PH_2{\cdot}BH_3]^- + PH_3 \quad \text{(at –45 °C)} \quad (8)$$

The reaction that is extrapolated to the $B_3H_7{\cdot}PH_3$ system would be

$$[B_3H_7{\cdot}PH_2]^- + B_3H_7{\cdot}PH_3 \rightarrow [B_3H_7{\cdot}PH_2{\cdot}B_3H_7]^- + PH_3 \quad (9)$$

However, because of the strongly acidic character of the B_3H_7 fragment, the B–P bond is stronger in $B_3H_7{\cdot}PH_3$ than in $BH_3{\cdot}PH_3$, the Lewis basicity of $[B_3H_7{\cdot}PH_2]^-$ is weaker than that of $[BH_3{\cdot}PH_2]^-$, and the activity of $B_3H_7{\cdot}PH_3$ in the ammonia solution is weaker than that of $BH_3{\cdot}PH_3$ in ammonia. All of these factors make the PH_3 displacement in the triborane system (eq 9) less favorable than that in the monoborane system (eq 8). Instead, the Lewis base $[B_3H_7{\cdot}PH_2]^-$ ruptures the B_3H_7 moiety.

Reaction of $B_3H_7{\cdot}PH_3$ with $NaBH_4$. The formation of $Na^+[B_3H_7{\cdot}PH_2{\cdot}BH_3]^-$ by the reaction of $B_3H_7{\cdot}PH_3$ with $NaBH_4$ parallels that of $Na^+[BH_3{\cdot}PH_2{\cdot}BH_3]^-$ from $BH_3{\cdot}PH_3$ and $NaBH_4$. As expected from its stronger protic acidity, the $B_3H_7{\cdot}PH_3$ reacts with $NaBH_4$ in tetrahydrofuran at a much lower temperature than $BH_3{\cdot}PH_3$ does in the same solvent.

The side reaction that was observed (eq 5) is explained by the formation of $B_3H_7{\cdot}THF$ followed by its facile reaction with $NaBH_4$[6] (eq 10 and 11). The participation of the side reaction

$$B_3H_7{\cdot}PH_3 + THF \rightleftharpoons B_3H_7{\cdot}THF + PH_3 \quad (10)$$

$$B_3H_7{\cdot}THF + Na^+BH_4^- \rightarrow Na^+B_3H_8^- + BH_3{\cdot}THF \quad (11)$$

is more pronounced at higher temperatures because of a higher rate of the $B_3H_7{\cdot}THF$ formation and, therefore, a higher concentration of $B_3H_7{\cdot}THF$. The preferential formation of NaB_3H_8 in diethyl ether probably indicates that the rate of the $[B_3H_7{\cdot}PH_2{\cdot}BH_3]^-$ formation is dependent on the solubility of $NaBH_4$ in the solvent used. Sodium borohydride is less soluble in diethyl ether than in tetrahydrofuran: solubilities of $NaBH_4$ at 20 °C are 0.02 g/100 g of $(C_2H_5)_2O$[7] vs 0.1 g/100 g of C_4H_8O.[8]

The $B_3H_8^-$ anion that is produced as the side product might further react with $B_3H_7{\cdot}PH_3$ and even with $BH_3{\cdot}PH_3$ (which would be formed by the reaction of the liberated PH_3 and $BH_3{\cdot}THF$[10])

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to give $[B_3H_7\cdot PH_2\cdot B_3H_7]^-$ and $[BH_3\cdot PH_2\cdot B_3H_7]^-$, respectively. However, the ^{11}B NMR spectrum of the reaction solution did not give evidence for the $[B_3H_7\cdot PH_2\cdot B_3H_7]^-$ anion.

The $[B_3H_7\cdot PH_2\cdot BH_3]^-$ Anion. The $[B_3H_7\cdot PH_2\cdot BH_3]^-$ anion is unique in that it contains two different borane fragments attached to the same phosphorus atom. The two J_{BP} values for P–B(B_3H_7) and P–B(BH_3) are 88 and 50 Hz, respectively, and are consistent with those expected from the relative acid strength of the two borane fragments.[5,10] Furthermore, the P–B(BH_3) coupling constant for $[B_3H_7\cdot PH_2\cdot BH_3]^-$ (50 Hz) is smaller than that for $[BH_3\cdot PH_2\cdot BH_3]^-$ (60 Hz),[1] reflecting the weakly basic nature of $[B_3H_7\cdot PH_2]^-$ relative to that of $[BH_3\cdot PH_2]^-$. The reaction of the $[B_3H_7\cdot PH_2\cdot BH_3]^-$ anion with HCl provided a unique testing case for the relative hydridic character of hydrogen atoms in the BH_3 and B_3H_7 fragments.

Reactivity of B_3H_7 Moieties toward Lewis Bases. The inertness of the $[B_3H_7\cdot PH_2\cdot BH_3]^-$ anion toward strong Lewis bases compares with that of the $B_3H_8^-$ anion. The coordination of the strongly basic $PH_2\cdot BH_3^-$ ligand and the overall negative charge render the B_3H_7 moiety inert toward strong bases. When the negative charge is removed as in $B_3H_7\cdot PH_2\cdot BH_2\cdot L$ [L = THF or $N(CH_3)_3$], the inertness is lost and its B_3H_7 moiety is cleaved readily by strong bases in the manner that was previously discussed.[11]

The instability of $[B_3H_7\cdot PH_2]^-$ is notable. Apparently, this negatively charged base is strong enough to rupture the B_3H_7 group of the anionic adduct $[B_3H_7\cdot PH_2]^-$. A complete study of the $[B_3H_7\cdot PH_2]^-$ anion decomposition has not been performed. However, the observation that one of the two decomposition products was the $[B_3H_7\cdot PH_2\cdot BH_3]^-$ anion and another product showed a rather simple ^{11}B NMR spectrum suggests that the B_3H_7 moiety may have been cleaved into BH_3 and B_2H_4 units. Thus, the decomposition of $NH_4^+[B_3H_7\cdot PH_2]^-$ in liquid ammonia and the decomposition of $Na^+[B_3H_7\cdot PH_2]^-$ in tetrahydrofuran might be expressed by

$$2NH_4^+[B_3H_7\cdot PH_2]^- + NH_3 \rightarrow NH_4^+[B_3H_7\cdot PH_2\cdot BH_3]^- + NH_4^+\text{“}[H_3N\cdot B_2H_4\cdot PH_2]^-\text{”} \tag{12}$$

$$2Na^+[B_3H_7\cdot PH_2]^- \rightarrow Na^+[B_3H_7\cdot PH_2\cdot BH_3]^- + Na^+\text{“}[(1/n)(B_2H_4\cdot PH_2)_n]^-\text{”} \tag{13}$$

Experimental Section

(a) General Procedures and Chemicals. Conventional vacuum-line techniques were used throughout for the handling of volatile compounds. Generally, reactions were performed in 9- or 10-mm-o.d. Pyrex tubes, each equipped with a stopcock. The tube containing a reaction mixture was placed in the probe of a Varian XL-100-15 or a FT-80A NMR spectrometer for recording of ^{11}B NMR spectra to monitor the reaction. Chemicals: B_2H_6, obtained from Callery Chemical Co.; B_4H_{10}, laboratory stock[12] or prepared by the hydrolysis of laboratory stock B_5H_{11};[13] PH_3, prepared by the pyrolysis of phosphorus acid;[14] $B_3H_7\cdot PH_3$, prepared by the reaction of B_4H_{10} and PH_3 in CH_2Cl_2;[5] $NaBH_4$, Alfa Products, purified by extracting with ammonia; $NaHB(sec\text{-}Bu)_3$, obtained from Aldrich Chemical Co. (N-Selectride), 1 M in tetrahydrofuran; HCl, Matheson Gas Products.

(b) Reaction of $B_3H_7\cdot PH_3$ with $Na^+[HB(sec\text{-}Bu)_3]^-$. A 0.50-mmol sample of $B_3H_7\cdot PH_3$ was condensed at the bottom of a 10-mm-o.d. Pyrex reaction tube, which was equipped with a ₸19/38 inner joint and, in parallel with the joint, a stopcock at the upper end of the tube.[15] A 0.6-mL sample of the N-Selectride solution was syringed in above the frozen sample of $B_3H_7\cdot PH_3$ through the ₸19/38 joint while an outward flow of nitrogen gas was maintained. The joint was then capped, the tube was evacuated, and the mixture was held at −45 °C for 2 h. The hydrogen gas evolved was collected in a Toepler pump system. The ^{11}B NMR spectrum of the reaction solution showed the signals of $Na^+[B_3H_7\cdot PH_2\cdot B(sec\text{-}Bu)_3]^-$. Weak signals of $B_3H_8^-$ and $[HB(sec\text{-}Bu)_3]^-$ were present in the spectrum also. The reaction tube was then pumped on for 30 h at −45 °C to remove $B(sec\text{-}Bu)_3$ and tetrahydrofuran. Dichloromethane was then added to the tube, and the ^{11}B and ^{31}P NMR spectra of $Na^+[B_3H_7\cdot PH_2]^-$ were recorded.

To a CH_2Cl_2 solution of $Na^+[B_3H_7\cdot PH_2]^-$ was added a slight excess of anhydrous HCl at −197 °C. The mixture was stirred at −80 °C and then allowed to warm to −45 °C. A white precipitate formed in the solution. The ^{11}B NMR spectrum of the solution indicated that $B_3H_7\cdot PH_3$ had been regenerated.

(c) Reaction of $B_3H_7\cdot PH_3$ with $NaBH_4$. In Tetrahydrofuran. A jump-stirrer rod was placed in a reaction tube similar to that described in part b. A 0.50-mmol sample of $B_3H_7\cdot PH_3$ and about 1 mL of tetrahydrofuran were condensed at the bottom of the reaction tube. A slight excess of $NaBH_4$ was then added through the joint. The tube was evacuated, and the mixture was stirred at −45 °C for 18 h. After the evolved hydrogen gas (0.43 mmol) was pumped out, the resulting solution was filtered into a 10-mm-o.d. NMR tube to record ^{11}B and ^{31}P spectra. In the sample thus prepared, only a trace of $B_3H_8^-$ was present. When the reactions were run at higher temperatures, increasingly larger amounts of $B_3H_8^-$ were found as the side product and smaller amounts of hydrogen gas were evolved.

In Diethyl Ether. A 0.24-mmol sample of $B_3H_7\cdot PH_3$ was allowed to react with 0.25 mmol of $NaBH_4$ for 30 min in diethyl ether at room temperature. The volatile components were pumped out, and a fresh portion of diethyl ether was condensed in the tube to obtain the ^{11}B NMR spectrum, which indicated the formation of $B_3H_8^-$ and a trace of $[B_3H_7\cdot PH_2\cdot BH_3]^-$.

(d) Supplementary Observation on $BH_3\cdot PH_3$ Reactions. Reaction with Ammonia. A 0.95-mmol sample of $BH_3\cdot PH_3$ was prepared in a 9-mm-o.d. tube and was dissolved in about 1.5 mL of liquid ammonia at −75 °C. The ^{11}B NMR spectrum of the solution showed the formation of $NH_4^+[BH_3\cdot PH_2]^-$. The ammonia was pumped out at −80 °C, and the residue was dissolved in CH_2Cl_2. The ^{11}B spectrum of the solution was that of $BH_3\cdot PH_3$.

Reaction with $NaBH_4$ in Tetrahydrofuran. A 0.48-mmol sample of $BH_3\cdot PH_3$ was prepared in a reaction tube and was allowed to react with 0.45 mmol of $NaBH_4$ in about 1 mL of tetrahydrofuran. At 0 °C no overt signs of reaction were noted. When the mixture was allowed to stand at room temperature for a few weeks, its ^{11}B NMR spectrum indicated that approximately 80% of the original $BH_3\cdot PH_3$ had been converted to $[BH_3\cdot PH_2\cdot BH_3]^-$.

(e) Reaction of $[B_3H_7\cdot PH_2\cdot BH_3]^-$ with HCl. A tetrahydrofuran solution of $Na^+(B_3H_7\cdot PH_2\cdot BH_3]^-$ was treated with a slight excess of anhydrous HCl at 0 °C for 45 min. The evolved hydrogen gas was pumped out, and the precipitate of NaCl (identified by its X-ray powder pattern) was filtered off. The ^{11}B NMR spectrum of the filtrate showed the signals attributable to $B_3H_7\cdot PH_2\cdot BH_2\cdot THF$: −5.0 ppm (t of d, $J_{BH} \approx 100$ Hz, $J_{BP} = 70$ Hz), BH_2; −14.0 (s) and −49.8 ppm (d, $J_{BP} = 80$ Hz), intensity ratio 2:1, B_3H_7. The ^{31}P signal appeared at −103.5 ppm.

When the tetrahydrofuran solution of $B_3H_7\cdot PH_2\cdot BH_2\cdot THF$ was treated with an excess of trimethylamine at −45 °C, the solution gave a set of ^{11}B signals which was interpreted as that of $B_3H_7\cdot PH_2\cdot BH_2\cdot N(CH_3)_3$: −11.0 ppm (t of d, $J_{BH} \approx 100$ Hz, $J_{BP} \approx 70$ Hz), BH_2; −13.7 (s) and −49.2 ppm ($J_{BP} = 78$ Hz), intensity ratio 2:1, B_3H_7.

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