Note that norbornene and 2-butyne *will* react with $Ta(CHCMe_3)(TIPT)_3(SEt_2)$, so *if* they also bind trans to the alkylidene ligand, the lifetime of the olefin or acetylene complex must be sufficiently long to permit rearrangement of the initial adduct to one in which the alkylidene ligand is cis to the substrate, in order that a metallacycle can form. (We should note that again there is no proof that the mechanisms of reactions between $Ta(CHCMe_3)(TIPT)_3(SEt_2)$ and a variety of substrates are all fundamentally the same.) A similar conclusion was arrived at in order to explain the inactivity of alkylidyne complexes containing bulky arenethiolate ligands for the metathesis of internal acetylenes.[13]

An important question is whether the metal is significantly less electrophilic in the thiolate complexes than it is in analogous phenoxide complexes. One could intuit that σ donation by a thiolate ligand should be greater than that in a phenoxide ligand and may overpower what appears to be a reduced amount of π donation to yield a more electron-rich metal overall.[13] However, there are virtually no data in the literature that bear on this important point. Another unknown is the exact nature of the initial metal/olefin interaction. It is assumed at this stage to have primarily σ character, but a component of π character cannot be discounted a priori. Under these circumstances attribution of a lower reactivity for the thiolate alkylidene complexes to a reduced metal electrophilicity, as was stated with more certainty in the case of the alkylidyne complexes,[13] is unjustified.

Acknowledgment. R.R.S. thanks the National Science Foundation (Grant CHE 88-22508) for support, and K.C.W. thanks the Central Research Department of DOW Chemical Co. for a predoctoral fellowship. We also thank the U.S. Department of Energy, Division of University and Industry Programs, for funds to purchase the X-ray diffractometer (Grant DE-FG05-86ER 75292).

Supplementary Material Available: An ORTEP drawing and a fully labeled drawing of $Ta(CH\text{-}t\text{-}Bu)(TIPT)_3(SEt_2)$ and a table of final positional and thermal parameters (8 pages); a listing of final observed and calculated structure factors (90 pages). Ordering information is given on any current masthead page.

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Contribution from the Department of Chemistry,
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Formation of the Dihydrogenphosphide-Bis[tetraborane(8)] Anion. Protic Acid Behavior of Phosphine-Tetraborane(8)

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Received September 11, 1989

The reactions of $BH_3\cdot PH_3$ and $B_3H_7\cdot PH_3$ with $NaBH_4$ have been reported in the literature.[1,2] These phosphine adducts behaved as protic acids and produced the $PH_2\cdot 2BH_3^-$ and $B_3H_7\cdot PH_2\cdot BH_3^-$ anions, respectively, when reacted with $NaBH_4$. The reaction of $B_3H_7\cdot PH_3$ was faster than that of $BH_3\cdot PH_3$. This reactivity difference was attributed to the stronger borane activity of the B_3H_7 fragment compared to the BH_3 fragment. The tetraborane(8) (B_4H_8) fragment being a stronger borane acid than B_3H_7,[3] the protic acid character of the recently reported $B_4H_8\cdot PH_3$[4] was expected to be stronger than that of $B_3H_7\cdot PH_3$. The reaction of $B_4H_8\cdot PH_3$ with $NaBH_4$ was studied, and the results are described in this note.

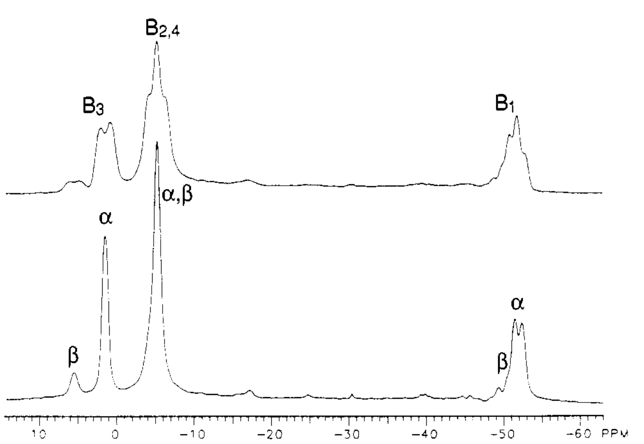

Figure 1. ^{11}B NMR (96.2 MHz) spectra of the $PH_2\cdot 2B_4H_8^-$ ion: upper spectrum, normal; lower spectrum, proton-spin decoupled.

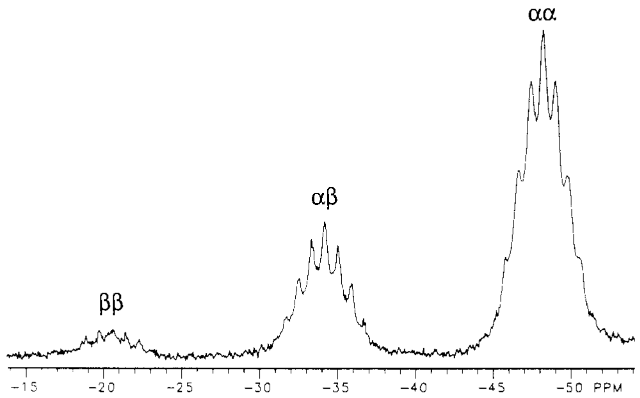

Figure 2. ^{31}P NMR (121.4 MHz) spectrum of the $PH_2\cdot 2B_4H_8^-$ ion, proton-spin decoupled.

Table I. NMR Data for the $PH_2\cdot 2B_4H_8^-$ Ion:[a] Shift, ppm (J_{XY}, Hz)

Boron-11[b]

α form			β form		
B_3	$B_{2,4}$	B_1	B_3	$B_{2,4}$	B_1
+1.4 (J_{BH} = 119)	-5.3 (J_{BH} = 105)	-52.0 (J_{BH} = 93) (J_{BP} = 90)	+5.4 (J_{BH} = 108)		-50.0 (J_{BH} = 107) (J_{BP} = 102)

Phosphorus-31[c]

αα form	αβ form	ββ form
-48.3 (J_{BP} = 95)	-34.2 (J_{BP} = 100)	-20.6

[a] Solvent: diethyl ether. [b] At -10 °C. Shift reference: $BF_3\cdot O(C_2H_5)_2$; external. [c] At -20 °C. Shift reference: 85% orthophosphoric acid; external.

Results

Reaction of $B_4H_8\cdot PH_3$ with $NaBH_4$. Phosphine-tetraborane(8) reacted with sodium borohydride in tetrahydrofuran, slowly at -80 °C and rapidly at -45 °C, according to the equation

$$2B_4H_8\cdot PH_3 + Na^+BH_4^- + THF \xrightarrow[\text{in THF}]{-80\ ^\circ C} Na^+[PH_2\cdot 2B_4H_8]^- + H_2 + PH_3 + BH_3\cdot THF \quad (1)$$

Although $BH_3\cdot THF$ was the major adduct product of BH_3, the $B_2H_7^-$ anion and $BH_3\cdot PH_3$ were present in small quantities in the reaction solution. The product salt, $Na[PH_2\cdot 2B_4H_8]$, was soluble in tetrahydrofuran and diethyl ether and could be isolated as a white solid that was unstable at room temperature.

NMR Spectra of $PH_2\cdot 2B_4H_8^-$. The ^{11}B and ^{31}P NMR spectra of the anion are shown in Figures 1 and 2, respectively, and the chemical shift data are listed in Table I. The adducts of B_4H_8 are considered to exist in two isomeric forms: endo and exo with respect to the position of the ligand relative to the hinge-shaped

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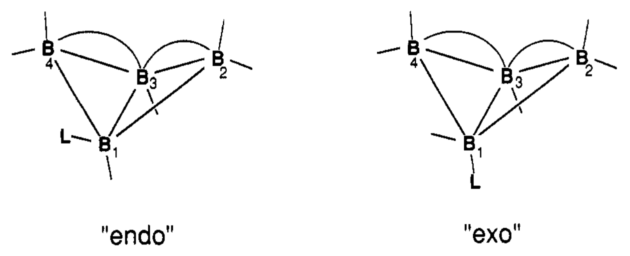


Figure 3. Endo and exo forms of the B_4H_8 adducts, $B_4H_8{\cdot}L$.

tetraborane framework.[4,5] See Figure 3. The ^{11}B spectra show the general feature of the B_4H_8 adducts, consisting of three major signals due to the B_1 (−52.0 ppm), $B_{2,4}$ (−5.3 ppm), and B_3 (+1.4 ppm)[6] atoms of one of the two conformers (α). In addition, weak signals of the B_1 and B_3 atoms of another conformer (β) are seen on the low-field sides of the respective major signals. The $B_{2,4}$ signal of the β conformer is overlapped by the corresponding signal of the α conformer.

Because of the two possible conformations for the B_4H_8 structure, the $PH_2{\cdot}2B_4H_8^-$ anion can exist in three isomeric forms: namely, $\alpha\alpha$, $\alpha\beta$, $\beta\beta$. Thus, the ^{31}P NMR spectrum of the anion contains three signals at −48.3, −34.2, and −20.6 ppm. The ^{11}B shift values of the B_4H_8 moiety depend upon its endo–exo conformation but appear to be virtually independent of the conformation of the other B_4H_8 moiety that is bonded to the same phosphorus atom. Thus, only two sets of B_4H_8 signals are observed in the ^{11}B spectrum.

Signal intensity measurements gave further support for the above consideration of isomeric forms. The intensity ratio for the three ^{31}P signals were measured to be 2.7:1.0:0.2. If the three signals were assigned to $\alpha\alpha$, $\alpha\beta$, and $\beta\beta$ isomers, respectively, then the population ratio between the α and β conformers of B_4H_8 would have to be 6.4:1.4, or simply 4.6:1.0. The intensity ratio of the $\alpha(B_3)$ and $\beta(B_3)$ signals in the ^{11}B spectrum was measured to be 4.7:1.0.[7] As in the case of several other B_4H_8 adducts, the assignment of the α and β conformation to the endo or exo form has yet to be accomplished.[4]

Discussion

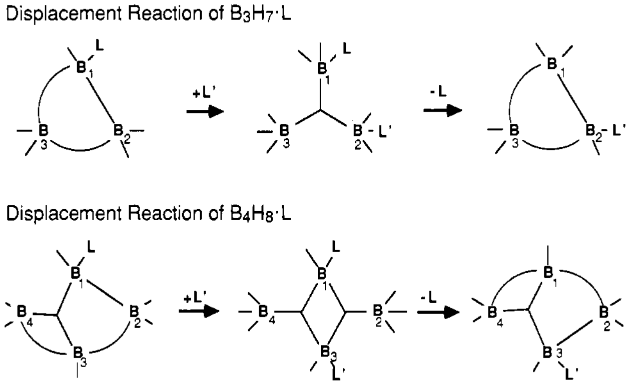
Formation of the $PH_2{\cdot}2B_4H_8^-$ Anion. The reactions of $BH_3{\cdot}PH_3$ and $B_3H_7{\cdot}PH_3$ with sodium borohydride are expressed by the equations

$$BH_3{\cdot}PH_3 + Na^+BH_4^- \xrightarrow[\text{in glyme}]{\text{room temp}} Na^+BH_3{\cdot}PH_2{\cdot}BH_3^- + H_2\,\text{[1]} \tag{2}$$

$$B_3H_7{\cdot}PH_3 + Na^+BH_4^- \xrightarrow[\text{in THF}]{-45\ ^\circ\text{C}} Na^+B_3H_7{\cdot}PH_2{\cdot}BH_3^- + H_2\,\text{[2]} \tag{3}$$

The ease of reaction increases in the order of the BH_3, B_3H_7, and B_4H_8 adducts. The PH_3 hydrogen atoms in $B_4H_8{\cdot}PH_3$, therefore, appear to be the most protonic among the three adducts. This trend is in keeping with the increasing borane acidity with the size of borane fragments.[3] The same trend has been observed for the B–P coupling constant values of the PH_3 adducts of BH_3, B_3H_7, and B_4H_8.[4] In spite of the regular reactivity trend that was observed for the PH_3 adducts of BH_3, B_3H_7, and B_4H_8, the anions produced by the $NaBH_4$ reactions are not of the same type: Neither the $PH_2{\cdot}2B_3H_7^-$ anion was formed, nor was the $B_4H_8{\cdot}PH_2{\cdot}BH_3^-$ anion produced.

Scheme I



It is likely that the initial step of these $NaBH_4$ reactions is the deprotonation of the phosphine adducts to form the PH_2^- adducts of the same boranes. For the $B_3H_7{\cdot}PH_3$ reaction, the PH_2^- adduct formed by the deprotonation (eq 4) combines with the "BH_3" that has been produced from the BH_4^- ion (eq 5): reaction pathway i

$$B_3H_7{\cdot}PH_3 + BH_4^- \rightarrow B_3H_7{\cdot}PH_2^- + H_2 + \text{“}BH_3\text{”} \tag{4}$$

$$B_3H_7{\cdot}PH_2^- + \text{“}BH_3\text{”} \rightarrow B_3H_7{\cdot}PH_2{\cdot}BH_3^- \tag{5}$$

For the $B_4H_8{\cdot}PH_3$ reaction, the PH_2^- adduct acts as a base to displace PH_3 from the PH_3 adduct (eqs 6 and 7): reaction pathway ii

$$B_4H_8{\cdot}PH_3 + BH_4^- \rightarrow B_4H_8{\cdot}PH_2^- + H_2 + \text{“}BH_3\text{”} \tag{6}$$

$$B_4H_8{\cdot}PH_2^- + B_4H_8{\cdot}PH_3 \rightarrow PH_2{\cdot}2B_4H_8^- + PH_3 \tag{7}$$

The choice of reaction pathway (i or ii) appears to be determined by the electrophilicity of the PH_3 adduct and by the basicity of the PH_2^- adduct.

The displacement of ligand from the B_3H_7 and B_4H_8 adducts is considered to proceed through the initial formation of a bis(ligand) intermediate, as illustrated in Scheme I.[4,8] The sites of the second Lewis base attack are the B_2 (or B_3) atom of the $B_3H_7{\cdot}PH_3$ molecule and the B_3 or B_2 (or B_4) atom of the $B_4H_8{\cdot}PH_3$ molecule. The B_4H_8 fragment being a stronger borane acid than the B_3H_7 fragment, these attack sites on the B_4H_8 adduct would be more electrophilic, or more reactive toward Lewis bases, than those on $B_3H_7{\cdot}PH_3$. The displacement of PH_3 from $B_4H_8{\cdot}PH_3$ by the $B_4H_8{\cdot}PH_2^-$ anion is thought to be facilitated because of this strong interaction of the B_4H_8 moiety with the $B_4H_8{\cdot}PH_2^-$ anion.[9] Furthermore, the order of basicity of the PH_2^- adducts (conjugate bases of the PH_3 adducts) is $B_4H_8{\cdot}PH_2^- < B_3H_7{\cdot}PH_2^- < BH_3{\cdot}PH_2^-$. Since the basicity of the $B_4H_8{\cdot}PH_2^-$ ion is weaker than that of the $B_3H_7{\cdot}PH_2^-$ ion, the $P\rightarrow BH_3$ bond of $B_4H_8{\cdot}PH_2{\cdot}BH_3^-$ would be weaker than that of $B_3H_7{\cdot}PH_2{\cdot}BH_3^-$. Consequently, the $B_4H_8{\cdot}PH_2^-$ ion is more readily available for the displacement reaction than the $B_3H_7{\cdot}PH_2^-$ anion.

Experimental Section

Chemicals and Equipment. Conventional vacuum-line techniques were used throughout for the handling of volatile compounds. Air-sensitive

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(6) In the ^{11}B spectrum of the reaction solution, which was recorded on a lower field NMR spectrometer, the B_3 signal was overlapped severely by the signal of $BH_3{\cdot}THF$ at −0.6 ppm. Earlier, a false intensity was measured for this signal because of the overlap, and thus the product was once wrongly thought to be "$B_5H_{10}{\cdot}PH_3^-$" (Jock, C. P. Master's Thesis, University of Utah, Salt Lake City, UT, 1987).

(7) When the original reaction solution containing the product, $Na[PH_2{\cdot}2B_4H_8]$, had never been allowed to warm above −23 °C, the $\beta\beta$-isomer signal (^{31}P) was barely detectable, and the relative intensities of the β-conformer signals (^{11}B) were much weaker than those found for solutions that had been warmed to room temperature. Apparently, at low temperatures, the α conformation is retained during the formation of the $PH_2{\cdot}2B_4H_8^-$ anion, and at room temperature, conversion of α to β occurs for an overall equilibrium. An isomerization process related to this has been observed for $B_4H_8{\cdot}PH_3$.[4]

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(9) Consideration of the mechanism of $PH_2{\cdot}2BH_3^-$ formation was not included in this part of the discussion because this anion could be formed by either one of the two reaction pathways (i and ii) under the conditions of experiment that was reported,[1] and currently no evidence is available to identify the pathway that the reaction assumed.

solids were handled under an atmosphere of nitrogen gas. Samples of $B_4H_8{\cdot}PH_3$ were prepared in reaction tubes, as needed, by the published method.[4] A commercial sample of $NaBH_4$ (Alfa Products) was purified by extracting with ammonia. Tetrahydrofuran and diethyl ether were refluxed and distilled over $LiAlH_4$ and stored over molecular sieves. A Varian XL-300 NMR spectrometer was used for the ^{11}B (96.2 MHz) and ^{31}P (121.4 MHz) spectral acquisitions. The ^{11}B chemical shift values for $B_4H_8{\cdot}PH_3$,[4] $BH_3{\cdot}PH_3$,[10a] $B_2H_7^-$,[11] $BH_3{\cdot}THF$,[10b] and BH_4^-[10c] are found in the respective references cited, and these were used to identify the components in the reaction mixtures.

Reaction of $B_4H_8{\cdot}PH_3$ with $NaBH_4$. A 1.12-mmol sample of B_5H_{11} was treated with a 4.88-mmol sample of PH_3 in a 22 mm o.d. Pyrex reaction tube, which was equipped with a Teflon valve, to prepare a sample of $B_4H_8{\cdot}PH_3$. Then, a 3-mL sample of tetrahydrofuran was condensed into the tube to dissolve the $B_4H_8{\cdot}PH_3$ at -80 °C. The solution was frozen at -197 °C, nitrogen gas was admitted into the tube, and a 4.63-mmol sample of $NaBH_4$ was introduced into the reaction tube through a side arm that had been provided at the upper end of the tube. The side arm was then sealed, and the tube was reevacuated and allowed to warm. At -80 °C, slow bubbling of a gas (H_2) was seen. At -45 °C, the bubbling was rapid. When the bubbling stopped (total 30 min), a total of 0.52 mmol of H_2 gas was collected. Volatile components in the tube were then distilled out, first at -45 °C and finally at 0 °C, to leave a white solid residue. To the volatile components that were collected was added a sample of $N(CH_3)_3$ (in a large excess). The mixture was warmed to 0 °C to convert any BH_3 adduct in the mixture to $BH_3{\cdot}N(CH_3)_3$. Phosphine and $BH_3{\cdot}N(CH_3)_3$ were separated from this mixture, and their quantities were measured to be 0.53 and 0.58 mmol, respectively. The solid residue in the original reaction tube was leached with diethyl ether. From the leachate, 85.9 mg of a white solid [0.54 mmol as $Na[PH_2{\cdot}2B_4H_8]$] was obtained by pumping out the solvent ether. The diethyl ether insoluble component was $NaBH_4$.

The NMR spectra of the reaction mixtures at various stages of the reaction process were obtained by preparing the reaction mixtures in 12-14 mm o.d. Pyrex tubes. These tubes were placed in the cold probe of the spectrometer for spectrum recording. The amounts of $B_4H_8{\cdot}PH_3$ used for these experiments were 0.3-0.5 mmol. The diethyl ether solution of $Na[PH_2{\cdot}2B_4H_8]$ for the NMR study was prepared by leaching the initial product residue with diethyl ether directly into a 12 mm o.d. Pyrex tube.

Acknowledgment. This work was supported by the U.S. Army Research Office through Grants DAAG 29-81-K-0101 and 29-85-K-0034.

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Magnetic Properties of Linear Chromium(III) Trimeric Clusters: $[(C_6H_5)_4P]_3[Cr_3Te_{24}]$ and $[(C_6H_5)_4P]_3[Cr_3Se_{24}]$

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and Charles J. O'Connor*,[1a]

Received March 30, 1989

The chemistry of binary metal polyselenides and polytellurides is considerably less well-known than that of the metal polysulfides.[2] However, recent work has shown that reaction of metal salts with polychalcogen sources under anhydrous conditions can generate a wide variety of soluble binary metal polyselenides and polytellurides.[3-7]

Following the recent report of the preparation and characterization of salts of two novel clusters having the formulas $[Cr_3Se_{24}]^{3-}$ and $[Cr_3Te_{24}]^{3-}$,[8] we have undertaken the magnetic characterization of these compounds. Both compounds have three chromium(III) ions linked in a linear fashion by bridging tetrachalcogenide chelates. These compounds are structurally similar to another linear cluster containing three chromium(III) ions and bridging chelate ligands.[9] Clusters such as these are under scrutiny as potential precursors to metal chalcogenide polymers.[10]

Given the current interest in systems with several magnetically active metal centers bridged by chalcogen atoms,[11,12] as well as the lack of data on selenide and telluride bridging groups, we have undertaken magnetochemical measurements of these systems. In this paper, we report the results of low-temperature magnetic measurements of these systems using a SQUID susceptometer, as well as analysis of this data using the linear trimer Heisenberg exchange model.

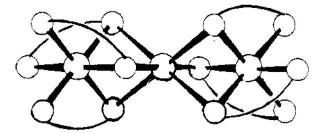
Experimental Section

Magnetic susceptibility data were recorded with an SHE Corp. VTS superconducting SQUID susceptometer. The sample bucket was fabricated from an Al-Si alloy obtained from the SHE Corp. The magnetic susceptibility of the sample bucket was measured independently over the temperature range 8-300 K, and the magnetic data for all samples were then corrected for the bucket contribution. Magnetic data were recorded on polycrystalline samples weighing 50 mg or so, which were packed into the buckets with tightly sealed caps in an argon-filled glovebox containing less than 1 ppm of oxygen. The samples were measured at 5 kOe. Measurement and calibration procedures are reported elsewhere.[13]

The magnetic susceptibility corrected for diamagnetism by using Pascal's constants is plotted as trimer susceptibility (i.e., three Cr^{III} ions per molecule) as a function of temperature. The diamagnetic corrections for the selenide and telluride complexes are 0.001 239 and 0.001 582 emu/mol, respectively.

Results and Discussion

The preliminary crystal structure data published previously[8] has shown the selenide and telluride analogues ($[Cr_3X_{24}]^{3-}$) have the same molecular structure. A ball and stick diagram that shows the molecular geometry as well as the coordination of the six Te_4^{2-} chelates is shown in **1**. The selenide and telluride analogues are similar, although they crystallize in distinctly different unit cells. Each structure contains a linear arrangement of three chromium(III) ions. The terminal metals are octahedrally chelated by three E_4^{2-} groups while a coordinated atom from each E_4^{2-} chain



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