5.97 kcal mol^{-1}, and $k_{12} = 2.8 \times 10^8\ M^{-1}\ \text{sec}^{-1}$. This calculation undercorrects for ionic strength effects. Using the average of the work terms calculated for the prevailing ionic strength and the $\mu = 0$ limit, we obtain a value of $k_{12} = 5.3 \times 10^6\ M^{-1}\ \text{sec}^{-1}$, in remarkable agreement with the observed value $6.0 \times 10^6\ M^{-1}\ \text{sec}^{-1}$.

In order to test the generality of this treatment of electrostatic factors, we carried out similar calculations for two other reactions between ions of opposite charge. The value of k_{12} for eq 11 (where DMP stands for 4,7-dimethyl-1,10-phenanthroline) is $1.1 \times 10^9\ M^{-1}\ \text{sec}^{-1}$ at 25° and $\mu = 0.10\ M$.[13] The values of k_{11} and k_{22}, the rate constants for the $Fe(DMP)_3^{2+}$–$Fe(DMP)_3^{3+}$ and $IrCl_6^{3-}$–$IrCl_6^{2-}$ exchange reactions, are 3×10^8 and $2.3 \times 10^5\ M^{-1}\ \text{sec}^{-1}$, respectively.[14,15] The value of k_{12} calculated using eq 9 and 10[16] (with the average work term approximation indicated above) is $1.6 \times 10^9\ M^{-1}\ \text{sec}^{-1}$. The value of k_{12} for eq 12 is $2.0 \times 10^9\ M^{-1}\ \text{sec}^{-1}$ at 10° and $\mu = 0.50\ M$.[4] The rate constant calculated as indicated above is $1.3 \times 10^9\ M^{-1}\ \text{sec}^{-1}$.[17,18]

$$IrCl_6^{2-} + Fe(DMP)_3^{2+} \underset{}{\overset{k_{12}}{\rightleftharpoons}} IrCl_6^{3-} + Fe(DMP)_3^{3+} \qquad (11)$$

$$Mo(CN)_8^{3-} + Os(bpy)_3^{2+} \overset{k_{12}}{\rightleftharpoons} Mo(CN)_8^{4-} + Os(bpy)_3^{3+} \qquad (12)$$

It must be pointed out, in view of the arbitrary averaging procedure used above, that the unusually good agreement between measured and calculated rate constants is probably largely fortuitous.[19] Nevertheless, since the comparisons involve complexes of different coordination numbers, coordination spheres, and self-exchange rates, the above calculations do illustrate the importance of electrostatic effects in determining reaction rates between metal complexes of different charge type. Neglect of the work terms for the systems under consideration results in discrepancies of more than 10^3 between observed and calculated rates. Although the importance of correcting for differences in the work terms is most readily seen for cross reactions involving ions of opposite charge, the work term differences are not necessarily negligible for reactions between ions of the same charge type since these terms also include nonelectrostatic contributions. Thus, it has been proposed[20] that contributions of the latter type could be responsible for the poor agreement between observed and calculated rates found for reactions of certain similarly charged ions. Evidently, the work terms must be considered carefully when comparing reaction rates in different systems.

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Registry No. $Fe(CN)_6^{3-}$, 13408-62-3; $Co(phen)_3^{2+}$, 16788-34-4; $Fe(CN)_6^{4-}$, 13408-63-4; $Co(phen)_3^{3+}$, 18581-79-8.

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Preparation and Properties of 6-Ethoxy-, 6-Phenyl-, and 6-Trimethylsiloxydecaborane(14)[1]

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Although many terminally substituted decaboranes(14) have been prepared, only two types of decaboranyl ethers, alkoxydecaboranes[2,3] and 6,6'-$B_{10}H_{13}OB_{10}H_{13}$,[4] are known. Recently, during our studies of the syntheses of alkylsila-, alkylgerma-, and alkylstannaundecaboranes[5,6] and our attempts to extend these syntheses to arylstannaundecaboranes, we have found reactions which result in two new decaboranyl ethers, 6-$(CH_3)_3SiOB_{10}H_{13}$ and 6-$C_2H_5OB_{10}H_{13}$. The 6-$(CH_3)_3SiOB_{10}H_{13}$ forms in reactions of $NaB_{10}H_{13}$ or $Na_2B_{10}H_{12}$ with $(CH_3)_3SiX$ (X = Cl, Br) in ethers. 6-$C_2H_5OB_{10}H_{13}$ results from the reaction of $NaB_{10}H_{13}$ or $Na_2B_{10}H_{12}$ with $SnCl_4$ in diethyl ether. Also, pyrolysis of $(C_6H_5)_2SnB_{10}H_{12}$ results in 6-$C_6H_5B_{10}H_{13}$, the latter of which although reported previously[7,8] had not been characterized completely.

Experimental Section

Infrared spectra were obtained with a Beckman IR-12 spectrometer. Proton NMR spectra at 60.0 and 100.0 MHz were recorded using Varian A-60 and JEOL-INM-PFT-100 spectrometers, respectively. Chemical shifts were measured relative to internal $(CH_3)_4Si$. Boron-11 NMR spectra were obtained with a Varian HA-100 equipped with standard 32.1-MHz probe and radiofrequency unit accessories. Chemical shifts were measured relative to external BBr_3 and are given relative to $BF_3 \cdot O(C_2H_5)_2$. Mass spectra were obtained at 70 eV with Varian MAT CH-7 and CH-5 spectrometers.

Diphenyltin dichloride was prepared and purified as described by Gilman.[9] $NaB_{10}H_{13}$[10] and $Na_2B_{10}H_{12}$[11] salts were prepared from reactions of NaH with $B_{10}H_{14}$ in diethyl ether. Bulk $(C_2H_5)_2O$ was removed from the decaboronate salts before their use. The resulting compounds, presumed to be etherates $Na_2B_{10}H_{12} \cdot x(C_2H_5)_2O$ and $NaB_{10}H_{13} \cdot x(C_2H_5)_2O$, were used in all syntheses described below. Trimethylsilyl chloride, $(CH_3)_3SiBr$, and $SnCl_4$ (Alfa Inorganics) were distilled before use. Diethyl ether was distilled from $LiAlH_4$ prior to use.

6-$(CH_3)_3SiOB_{10}H_{13}$. $(CH_3)_3SiCl$ (20 mmol) was added under N_2 to 2.0 mmol of $Na_2B_{10}H_{12}$ or $NaB_{10}H_{13}$. After 6 h at 50°, reaction materials were sublimed (65°) through a U-trap at 0°. Pure $(CH_3)_3SiOB_{10}H_{13}$ was collected in the 0° trap after two sublimations; mp 36–37°. Yields from $Na_2B_{10}H_{12}$ and $NaB_{10}H_{13}$ reactions were ca. 20% and 5%, respectively. Anal.[12] Calcd for $C_3H_{22}B_{10}SiO$: C, 17.11; H, 10.54; B, 51.39. Found: C, 17.41; H, 10.52; B, 51.15. Mass spectral data (70 eV): most intense envelope centered at *m/e* 73 ($(CH_3)_3Si^+$ ions), parent envelope at *m/e* 208 ($(CH_3)_3SiOB_{10}H_x{}^+$ ions), and spectral cutoff at *m/e* 214 ($(^{12}CH_3)_3{}^{30}SiO^{11}B_{10}H_{13}{}^+$). The methyl ($CH_3$) resonance occurs at τ 9.75 ppm in the ^1H NMR spectrum. Infrared spectral absorptions (in benzene in matched solution cells) occur at 2958 (m), 2569 (vs), 2092 (w), 1545 (w), 1486 (m), 1411 (w), 1371 (vs), 1345 (w), 1310 (w), 1279 (s), 1247 (vs), 1114 (m), 1061 (w), 1032 (w), 988 (m), 951 (m), 926 (w), 892 (s), 854 (vs), 805 (w), 751 (m), 713 (w), 678 (w), 526 (w) cm^{-1}. The ^{11}B NMR spectrum exhibits the following resonances (area; *J*, Hz): doublets at δ −2.0 (5; 146), 16.9 (2; 157), 36.4 (1; 157), and 48.8 ppm (1; 157); an area 1 singlet at δ −23.8 ppm.

In separate experiments in a closed reaction tube, 2.0 mmol of $NaB_{10}H_{13}$ in ca. 0.5 ml of $(C_2H_5)_2O$ was allowed to react with 6.5 mmol of $(CH_3)_3SiCl$ at 58° for 16 h. Volatile reaction materials were separated by fractional condensation and low-temperature distillation techniques[13] and found to consist of 0.62 mmol of hydrogen (confirmed by vapor density), 1.1 mmol of C_2H_6 (confirmed by vapor tension[13] and ir spectrum[14]), 4.1 mmol of unreacted $(CH_3)_3SiCl$ (confirmed by ir spectrum), 1.0 mmol of $[(CH_3)_3Si]_2O$ (confirmed by ir[15] and ^1H NMR spectra[16]), diethyl ether solvent containing what may have been a small quantity of C_2H_5Cl (confirmed by ir spectrum[17]), 0.66 mmol of $B_{10}H_{14}$ (confirmed by ^{11}B NMR spectrum), and 0.04 mmol of $(CH_3)_3SiOB_{10}H_{13}$. Heating the residue to 150° produced additional $B_{10}H_{14}$. No products could be characterized or isolated from the intractable material that remained after volatile products were removed. Similar products were observed from $Na_2B_{10}H_{12}$–$(C_2H_5)_2O$–$(CH_3)_3SiCl$ reactions and in reactions where $(CH_3)_3SiCl$ was replaced by $(CH_3)_3SiBr$.

Solutions of $Na_2B_{10}H_{12}$ (2.5 mmol) and $NaB_{10}H_{13}$ (2.0 mmol) in neat $[(CH_3)_3Si]_2O$ were heated separately for 12 h at 58°. The ^{11}B NMR spectra of the benzene extract of the reaction materials showed traces of $B_{10}H_{14}$. No $(CH_3)_3SiOB_{10}H_{13}$ formed in either reaction.

A benzene solution of $NaB_{10}H_{13}$ (3.0 mmol) from which bulk diethyl ether had been removed and to which 5.0 mmol of $(CH_3)_3SiCl$ was added was allowed to react while the ^1H NMR spectrum was monitored periodically. After the addition of $(CH_3)_3SiCl$, a singlet resonance at τ 9.92 ppm appeared and grew slowly at the expense of the $(CH_3)_3SiCl$ resonance at τ 9.72 ppm. After 2 h, a small singlet at τ 9.75 ppm ($(CH_3)_3SiOB_{10}H_{13}$) could be seen. After 8 h, no further reaction was apparent. $(CH_3)_4Si$ and $[(CH_3)_3Si]_2O$ were added as second internal calibrants. The $[(CH_3)_3Si]_2O$ resonance occurred at τ 9.92 ppm, superimposed on the τ 9.92 ppm resonance from the reaction. The ^{11}B NMR spectrum after reaction showed resonances due only to $B_{10}H_{14}$ and $(CH_3)_3SiOB_{10}H_{13}$.

An 85:15 6-$C_2H_5OB_{10}H_{13}$–5-$C_2H_5OB_{10}H_{13}$ mixture (3.0 mmol) was heated at 55° for 8 h with 3 ml of $(CH_3)_3SiCl$. Some decomposition of $C_2H_5OB_{10}H_{13}$ occurred; however, the isomer ratio of recovered $C_2H_5OB_{10}H_{13}$ was unchanged and no other boron-containing products were formed.

6-$C_2H_5OB_{10}H_{13}$. Stannic chloride (4.3 mmol) in toluene was added dropwise under N_2 to $NaB_{10}H_{13}$ (5.0 mmol) in ca. 2 ml of diethyl ether at −112°. After 30 min the reaction was warmed to room temperature, high-volatility materials were removed in vacuo, and the remaining solid was sublimed (60°) through a U-tube at 0°. The $C_2H_5OB_{10}H_{13}$ along with some $B_{10}H_{14}$ condensed at 0°. Repeated sublimation resulted in the isolation of pure $C_2H_5OB_{10}H_{13}$, which from ^1H and ^{11}B NMR was found to be an 85:15 mixture of 6-$C_2H_5OB_{10}H_{13}$–5-$C_2H_5OB_{10}H_{13}$.[2] Mass spectral analyses of the reaction solids which were involatile at 60° showed evidence for traces of chlorodecaborane products and a series of peaks due to chlorotin species.

Chromatography under N_2 of the $C_2H_5OB_{10}H_{13}$ mixture in benzene on a silica gel column[18] resulted in removal of 5-$C_2H_5OB_{10}H_{13}$. Some 6-$C_2H_5OB_{10}H_{13}$ was also lost on the column. The 6-$C_2H_5OB_{10}H_{13}$ obtained contained less than 5% of the 5 isomer, as adjudged by ^1H NMR spectral analysis. The 6-$C_2H_5OB_{10}H_{13}$ solidified to a glass, so no melting point was obtained. Anal. Calcd for $C_2H_{18}B_{10}O$: C, 14.43; H, 10.90. Found: C, 14.20; H, 10.71. Mass spectral data (70 eV): highest intensity parent envelope centered at *m/e* 164 ($C_2H_5OB_{10}H_x{}^+$ ions), spectral cutoff at *m/e* 168 ($^{12}C_2H_5O^{11}B_{10}H_{13}{}^+$). ^1H NMR spectral resonances occur at τ 8.91 ppm (triplet, CH_3 protons, *J* = 7.0 Hz). Infrared spectral absorptions (neat liquid film): 3322 (w), 2824 (s), 2915 (m), 2583 (vs), 2353 (w), 2079 (m), 1904 (m), 1553 (m), 1490 (s), 1447 (w), 1408 (m), 1379 (vs), 1317 (vs), 1258 (vs), 1162 (w), 1117 (m), 1105 (m), 1047 (s), 1025 (s), 1015 (vs), 966 (s), 939 (m), 917 (w), 897 (w), 866 (s), 844 (m), 810 (m), 764 (w), 722 (s), 707 (s), 687 (s), 641 (m), 578 (m), 548 (m), 499 (m) cm^{-1}. The ^{11}B NMR spectrum exhibits the following resonances (area; *J*, Hz): doublets at δ −2.9 (5; 145), 17.3 (2; 155), 33.3 (1; 155), and 45.1 ppm (1; 155); an area 1 singlet at δ −25.9 ppm.

In separate experiments, $SnCl_4$ was allowed to react with $NaB_{10}H_{13}$–$(C_2H_5)_2O$ solutions as above except that the reaction mixtures were maintained at −78, −30, and 25° during the initial 30 min reaction period. From these reactions the 6-$C_2H_5OB_{10}H_{13}$:5-$C_2H_5OB_{10}H_{13}$ product ratios were found to be 85:15 (−78°), 75:25 (−30°), and 70:30 (25°), respectively.

A sample consisting of 85:15 6-$C_2H_5OB_{10}H_{13}$–5-$C_2H_5OB_{10}H_{13}$ was heated at 100° for 8 h, during which time the ^{11}B and ^1H NMR spectra were monitored periodically. Only slight decomposition and no measurable change in the isomer ratio were detected.

$(C_6H_5)_2SnB_{10}H_{12}$. Typically, 5.0 mmol of $(C_6H_5)_2SnCl_2$ in 8 ml of benzene was added dropwise under N_2 to 5.0 mmol of $Na_2B_{10}H_{12}$. The solution turned red immediately. After 1 h at 25° reaction materials were centrifuged. The supernatant solution was evaporated in vacuo leaving a red solid which contained $(C_6H_5)_2SnB_{10}H_{12}$ (yield 30%) and a trace of $(C_6H_5)_3SnCl$ (identified by mass spectrum).

The $(C_6H_5)_2SnB_{10}H_{12}$ decomposes slowly in vacuo at room temperature. Attempts to purify the $(C_6H_5)_2SnB_{10}H_{12}$ by sublimation or column chromatography on silica gel were unsuccessful. The ^{11}B NMR spectrum at 32° in benzene shows a poorly resolved low-field group of resonances (area 8) and a doublet (area 2) at δ 25.7 ppm (*J* = 155 Hz). Cooling caused further spectral broadening; however, at temperatures above 32° resolution of the low-field region of the spectrum improved and the resulting spectrum agreed closely with the spectra of the dialkylstannaundecaboranes.[5,6] At 90°C distinct maxima at δ −13.8, −9.0, −6.9, and −3.0 ppm and a doublet at δ 4.6 ppm (area 2; *J* = 146 Hz) could be seen in addition to the δ 25.7 ppm doublet. Decomposition occurred at this temperature causing slow spectral changes. Mass spectral data (70 eV): most intense envelope centered at *m/e* 312 ($C_6H_5SnB_{10}H_x{}^+$ ions), highest mass envelope centered at *m/e* 388 ($(C_6H_5)_2SnB_{10}H_x{}^+$ ions). Because $(C_6H_5)_2SnB_{10}H_{12}$ samples free of $(C_6H_5)_3SnCl$ could not be obtained, infrared spectral data were not collected.

6-$C_6H_5B_{10}H_{13}$. The solid $(C_6H_5)_2SnB_{10}H_{12}$ was heated for 8 h at 95° in a continuously evacuated vessel attached to a U-tube at 0°. Repeated passage of the 0° condensate into a 0° trap resulted in the isolation of pure $C_6H_5B_{10}H_{13}$ (ca. 10% yield). Anal. Calcd for $C_6H_{18}B_{10}$: C, 36.31; H, 9.14. Found: C, 36.22; H, 9.21. The $C_6H_5B_{10}H_{13}$ froze to a glass at ca. −15° without apparent crystallization. Phenyl (C_6H_5) resonances occur at τ 2.56 ppm in the ^1H NMR spectrum. Mass spectral data (70 eV): most intense envelope centered at *m/e* 192 ($C_6H_5B_{10}H_x{}^+$ ions), spectral cutoff at *m/e* 200 ($^{12}C_6H_5{}^{11}B_{10}H_{13}{}^+$). Infrared spectral absorptions (neat liquid film): 3040 (m), 2910 (m), 2840 (m), 2550 (vs), 1980 (m), 1955 (m), 1905 (m), 1815 (m), 1770 (m), 1690 (w), 1660 (m), 1640 (m), 1595 (s), 1552 (s), 1511 (vs), 1500 (vs), 1480 (vs), 1430 (vs), 1340 (s), 1300 (m), 1250 (s), 1230 (vs), 1190 (m), 1160 (m), 1110 (m), 1075 (s), 1040 (m), 1025 (s), 1000 (vs), 975 (m), 958 (s), 938 (s), 925 (s), 867 (s), 835 (s), 817 (s), 789 (m), 744 (m), 756 (vs), 725 (vs), 692 (vs), 643 (m), 624 (s), 566 (m), 558 (m), 536 (s), 422 (s), 401 (m) cm^{-1}. ^{11}B NMR resonances agree with those reported earlier for 6-$C_6H_5B_{10}H_{13}$.[8]

Results and Discussion

Characterization of 6-$(CH_3)_3SiOB_{10}H_{13}$ and 6-$C_2H_5OB_{10}H_{13}$ is based on ^1H NMR, ^{11}B NMR, infrared, and mass spectral data and elemental analyses data. The presence of a low-field area 1 singlet and the splitting of the B(2,4) resonance[19] into two area 1 doublets in the ^{11}B NMR spectra confirm the assignment of 6-substitution. The area 2 doublets at δ 16.9 and 17.3 ppm for the trimethylsiloxy and ethoxy derivatives, respectively, can be assigned tentatively to B(8,10) borons, since they are in the region where these boron resonances occur in other 6-substituted decaboranes(14) and $B_{10}H_{14}$.[19] Our data also complete the characterization of 6-$C_6H_5B_{10}H_{13}$.[8] Our infrared data are difficult to compare with the published data of Hanousek et al.;[7] thus we cannot conclude whether or not the $C_6H_5B_{10}H_{13}$ they report is 6-substituted as is the isomer reported herein and that reported by Siedle et al.[8]

The new 6-substituted compounds are formed in low to moderate yields in reactions which appear complex and which are not yet fully understood. The 6-$(CH_3)_3SiOB_{10}H_{13}$ forms in reactions of $(CH_3)_3SiCl$ or $(CH_3)_3SiBr$ with $NaB_{10}H_{13}$ or $Na_2B_{10}H_{12}$ in dialkyl ethers. Other products of the reactions are H_2, C_2H_6, $[(CH_3)_3Si]_2O$, $B_{10}H_{14}$, and possibly C_2H_5Cl. In an experiment carried out so that NMR spectra could be monitored periodically, the reaction of $NaB_{10}H_{13}$ with $(CH_3)_3SiCl$ in diethyl ether showed that $[(CH_3)_3Si]_2O$ forms early in the reaction sequence, before appreciable quantities of 6-$(CH_3)_3SiOB_{10}H_{13}$ are present. However, reactions of $[(CH_3)_3Si]_2O$ with $B_{10}H_{13}^-$ or $B_{10}H_{12}^{2-}$ do not yield $(CH_3)_3SiOB_{10}H_{13}$ as a product. Also the absence of $(CH_3)_3SiOB_{10}H_{13}$ as a product in reactions of $(CH_3)_3SiCl$ with $C_2H_5OB_{10}H_{13}$ precludes direct reaction pathways. It is possible that the reaction involves initially the exchange of $(CH_3)_3Si$ for C_2H_5 groups of etherated $B_{10}H_{13}^-$ (or $B_{10}H_{12}^{2-}$), i.e., $B_{10}H_{13}O(C_2H_5)_2^-$, to form species such as $B_{10}H_{13}O$-$Si(CH_3)_3(C_2H_5)^-$ and $B_{10}H_{13}O[Si(CH_3)_3]_2^-$. Although it has been shown that the ^{11}B NMR spectrum of the $B_{10}H_{13}^-$ ion is not affected significantly by coordinating solvents,[20] solvation of $NaB_{10}H_{13}$ by ethers is well established.[21] Oxidative elimination of an ethyl group from $B_{10}H_{13}OSi(CH_3)_3(C_2H_5)^-$ could yield 6-$(CH_3)_3SiOB_{10}H_{13}$ and C_2H_6. Substitution of $(C_2H_5)_2O$ for the less basic $[(CH_3)_3Si]_2O$ on $B_{10}H_{13}$-$O[Si(CH_3)_3]_2^-$ would account for the presence of $[(CH_3)_3Si]_2O$ as a reaction product. Radical processes leading to the observed products can be considered; however, products such as C_4H_{10}, $(CH_3)_3SiH$, and $[(CH_3)_3Si]_2$, which might be expected if $C_2H_5\cdot$ or $(CH_3)_3Si\cdot$ radicals were present, were not detected.

Since redistribution of alkyl groups on silicon is slow,[22] redistribution of $(CH_3)_3SiX$ to form $(CH_3)_2SiX_2$ which could then react with the decaboronate anions to form $(CH_3)_2Si$-$B_{10}H_{12}$, as occurs with alkyltin compounds, does not occur.[5,6] Also, no evidence of compounds such as $(CH_3)_3SiB_{10}H_{13}$, reported earlier by Amberger and Liedl,[23] or $[(CH_3)_3Si]_2$-$B_{10}H_{12}$ was obtained.

The 6-$C_2H_5OB_{10}H_{13}$ forms in reactions of $SnCl_4$ with $NaB_{10}H_{13}$ (or $Na_2B_{10}H_{12}$) in diethyl ether along with a lesser amount of 5-$C_2H_5OB_{10}H_{13}$.[2,3,24] This reaction may involve $SnCl_4$ as a two-electron oxidizing agent, in a role analogous to that of I_2 in $NaB_{10}H_{13}^-$–ether reactions suggested by Hawthorne and Miller.[2]

The 6-$C_2H_5OB_{10}H_{13}$:5-$C_2H_5OB_{10}H_{13}$ isomer distribution depends on the reactant combination and to a greater extent on temperature. In $NaB_{10}H_{13}$–$(C_2H_5)_2O$–$SnCl_4$ reactions, the isomer ratio varied from 85:15 to 70:30 at reaction temperatures from −112 to +25°, respectively. Previously, we showed that the product of the reaction of I_2 with $NaB_{10}H_{13}$–$(C_2H_5)_2O$ is mainly 5-$C_2H_5OB_{10}H_{13}$;[24] although from our ^{11}B NMR spectral data the presence of small amounts (5–10%) of 6-$C_2H_5OB_{10}H_{13}$ is evident. The presence of both isomeric forms in the $SnCl_4$ and I_2 reactions, along with the observation that the 6:5 ratio is temperature dependent in $SnCl_4$ reactions, suggests that the reactions may involve 5- and 6-ether-coordinated decaboronate intermediate species, though possibly not the same intermediate species since the product isomer ratios in the I_2 and $SnCl_4$ reactions are quite different. The 6-$C_2H_5OB_{10}H_{13}$–5-$C_2H_5OB_{10}H_{13}$ isomer mixture does not form as a result of the equilibration of an initially formed 5- or 6-$C_2H_5OB_{10}H_{13}$, since heating either the 5 or 6 isomer for prolonged periods of time produces no detectable isomerization.

The $C_6H_5B_{10}H_{13}$ forms during the thermal decomposition of $(C_6H_5)_2SnB_{10}H_{12}$, along with substantial amounts of $B_{10}H_{14}$. No other products have been isolated. Although $(C_6H_5)_2SnB_{10}H_{12}$ is too unstable to allow isolation from small amounts of coformed phenyltin chloride products, its spectral properties allow it to be adequately characterized as an aryl analogue of the previously reported dialkylstannaundecaboranes[5,6] and to be confirmed as the source of 6-$C_6H_5B_{10}H_{13}$. The thermal instability of $(C_6H_5)_2SnB_{10}H_{12}$ is in striking contrast to that of the dialkyl compounds, where heating at 60° for sustained periods causes only slight decomposition. It is interesting to note that no aryltin–decaborane products were isolated from reactions of $(C_6H_5)_2SnCl_2$ with $[(C_6H_5)_4As]_2B_{10}H_{12}$.[25]

Registry No. $(CH_3)_3SiCl$, 75-77-4; $Na_2B_{10}H_{12}$, 12046-70-7; $NaB_{10}H_{13}$, 12008-65-0; 6-$(CH_3)_3SiOB_{10}H_{13}$, 57527-18-1; $SnCl_4$, 7646-78-8; $(C_2H_5)_2O$, 60-29-7; 6-$C_2H_5OB_{10}H_{13}$, 57527-19-2; $(C_6H_5)_2SnCl_2$, 1135-99-5; $(C_6H_5)_2SnB_{10}H_{12}$, 57527-53-4; 6-$C_6H_5B_{10}H_{13}$, 38998-69-5.

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Investigation of the Structure of Barium Iridate ($BaIrO_3$) by High-Resolution Electron Microscopy

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Donohue, Katz, and Ward[2] suggested that the structure of barium iridate, $BaIrO_3$, was based on a nine-layer stacking of BaO_3 layers in the sequence $(chh)_3$ similar to the rhombohedral phase of $BaRuO_3$ (a = 5.75 Å, c = 21.6 Å). The transition metal ions are in strings of three face-shared MO_6 octahedra which are corner linked.[3,4] However, in order to