the opposite conclusion. The solid straight-line correlations which neglect d orbitals are fairly good. Small errors in the parameterization of silicon and germanium in both the CNDO/2 and CHELEQ methods may cause the low empirical values of k and c. Perhaps relaxation effects can account for at least part of the deviations from the dashed lines, especially in the cases of $M(CH_3)_4$, MBr_4, and MCl_4. Inasmuch as the silicon and germanium correlations (without consideration of d orbitals) are almost as good as the carbon correlations, it seems unlikely that any treatment including d-orbital bonding could significantly improve the silicon and germanium correlations relative to the carbon correlation. All in all, the data offer little support for the participation of d orbitals in the bonding of silicon and germanium compounds.

Chlorine and Bromine Chemical Shifts. Core binding energies for the halogen, oxygen, and methyl carbon atoms in the compounds which we have discussed and also for molecular chlorine, bromine, hydrogen chloride, and hydrogen bromide were measured and are given in Table VI. No correlations were made for the oxygen or fluorine binding energies because there were insufficient data for these elements. The EHT, CNDO/2, and CHELEQ correlation data for the chlorine binding energies are listed in Table VII. The data from the bromine correlations, listed in Table VIII, closely parallel the chlorine data. All of these correlations have considerable scatter, as indicated by the correlation coefficients. The low standard deviations are a consequence of the small range of binding energies involved. Some of the experimental shifts (which may be obtained from the data in Table VI) deserve comment. The chemical shift $E_B(HX) - E_B(X_2)$ is much smaller for X = Cl, Br than was observed by other workers for X = F.[48] The halogen binding energies for corresponding silicon and carbon compounds are quite close, whereas those for the corresponding germanium compounds are shifted to lower energy. One might have expected the halogens on corresponding silicon and germanium compounds to have nearly the same energies. More satisfactory correlation methods, probably including relaxation effects, seem to be necessary to understand these halogen chemical shifts.

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Registry No. CH_4, 74-82-8; CH_3CH_3, 74-84-0; $C(CH_3)_4$, 463-82-1; $(CH_3)_2O$, 115-10-6; CF_4, 75-73-0; CH_3Cl, 74-87-3; CCl_4, 56-23-5; CH_3Br, 74-83-9; CBr_4, 558-13-4; SiH_4, 7803-62-5; SiH_3CH_3, 992-94-9; $Si(CH_3)_4$, 75-76-3; $(SiH_3)_2O$, 13597-74-3; SiF_4, 7783-61-1; SiH_3Cl, 13465-78-6; $SiCl_4$, 10026-04-7; SiH_3Br, 13465-73-1; $SiBr_4$, 7789-66-4; GeH_4, 7782-65-2; GeH_3CH_3, 1449-65-6; $Ge(CH_3)_4$, 865-52-1; GeF_4, 7783-58-6; GeH_3Cl, 13637-65-5; $GeCl_4$, 10038-98-9; GeH_3Br, 13569-43-2; $GeBr_4$, 13450-92-5; Cl_2, 7782-50-5; HCl, 7647-01-0; Br_2, 7726-95-6; HBr, 10035-10-6; C, 7440-44-0; Si, 7440-21-3; Ge, 7440-56-4; O_2, 7782-44-7; F_2, 7782-41-4.

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Boron Insertion Reactions. III. Synthesis of $2\text{-}CH_3B_6H_9$ and $1\text{-}[(CH_3)_3M^{IV}]B_6H_9$ (M^{IV} = Si, Ge)

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Reaction of $(CH_3)_3B$ with B_5H_9 (assisted by $(CH_3)_3Ga$) produces $2\text{-}CH_3B_6H_9$ *via* a pathway that can be considered formally analogous to a carbene insertion reaction. Boron insertion reactions using $H_2BCl\cdot OR_2$ with $(CH_3)_3M^{IV}B_5H_7^-$ anions (M^{IV} = Si, Ge) give rise to the corresponding $1\text{-}(CH_3)_3M^{IV}B_6H_9$ products, which constitute the first examples of apically substituted hexaborane(10) derivatives.

Until recently no derivatives of hexaborane(10), B_6H_{10}, were known and even now the list is not extensive. Typical compounds prepared to date include $B_6H_{10}\cdot L$,[1] $B_6H_{10}L_2$,[2] $B_6H_{10}\cdot BCl_3$,[3] $\mu\text{-}Fe(CO)_4B_6H_{10}$,[4] $\mu\text{-}Cl_2Pt(B_6H_{10})_2$,[5] $2,3\text{-}(CH_3)_2B_6H_8$,[6] $2\text{-}CH_3B_6H_9$,[7] $2\text{-}BrB_6H_9$,[8] and the salts of the protonated and deprotonated hexaborane(10) species $B_6H_{11}^+$, $2\text{-}CH_3B_6H_{10}^+$,[9] and $B_6H_9^-$.[10]

We have been studying boron insertion reactions for some time, with emphasis on the synthesis of B_6H_{10} derivatives from B_5H_9 derivatives. We report here the synthesis of $2\text{-}CH_3B_6H_9$ by an unprecedented boron insertion reaction and the syntheses of the first two examples of apically substituted B_6H_{10} derivatives, $1\text{-}[(CH_3)_3M^{IV}]B_6H_9$ (M^{IV} = Si, Ge).

Results and Discussion

$2\text{-}CH_3B_6H_9$. Gas-phase thermolysis of equimolar mixtures of B_5H_9 and $(CH_3)_3B$ in sealed Pyrex vessels produces $2\text{-}CH_3B_6H_9$ at 200° or above. At these temperatures, however, decomposition of $2\text{-}CH_3B_6H_9$ to $2\text{-}CH_3B_5H_8$ occurs at a

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Table I. Representative Data for Gas-Phase Reactions of B_5H_9 with $B(CH_3)_3$

Amt used, mmol			Reaction temp, °C	Reaction time, hr	Amt recovered, mmol		Amt produced, mmol		% yield of $2\text{-}CH_3B_6H_9$[b]
B_5H_9	$B(CH_3)_3$	$Ga(CH_3)_3$[a]			B_5H_9	$B(CH_3)_3$	$2\text{-}CH_3B_5H_8$	$2\text{-}CH_3B_6H_9$	
10.12	10.12	0.00	200	139	1.14	1.20	0.543	0.235	2.62
10.12	10.12	1.01	200	18	9.56	10.66	0.039	0.306	53.97
10.12	10.12	1.01	200	18	4.62	5.93	0.099	0.773	14.03
12.90	12.90	2.81	175	5	11.62	15.54	0.00	0.453	35.50
12.90	12.90	2.81	175	14	11.52	15.34	0.00	0.472	34.11

[a] All the $Ga(CH_3)_3$ was consumed in these reactions. [b] Per cent yield is based on B_5H_9 consumed in the reaction.

significant rate.[11] In addition, the presence of the $2\text{-}CH_3B_5H_8$ impurity in the reaction mixture seriously interferes with the purification of $2\text{-}CH_3B_6H_9$.

It has subsequently been found that addition of other trialkyl group III compounds to the B_5H_9-$(CH_3)_3B$ reaction mixture assists the synthesis of $2\text{-}CH_3B_6H_9$. The best assisting reagent to date is $(CH_3)_3Ga$, which, when added in amounts of 5-10 mol % to the B_5H_9-$(CH_3)_3B$ mixture, allows the formation of much better yields of $2\text{-}CH_3B_6H_9$ (based on B_5H_9 consumed) at a temperature of only 175°. Under these less severe conditions no $2\text{-}CH_3B_5H_8$ is formed. Representative reaction data are presented in Table I. Additional experiments indicate that the yields of $2\text{-}CH_3B_6H_9$ are not significantly affected by changes in reagent concentrations or surface area over rather broad limits. Table I also shows that apparently identical experiments on occasion produce results that vary by as much as a factor of 3. When $(CH_3)_3Al$ is substituted for $(CH_3)_3Ga$, the desired reaction occurs at 175°, though the yield of $2\text{-}CH_3B_6H_9$ is somewhat reduced. Though the absolute yield of $2\text{-}CH_3B_6H_9$ never exceeds 10%, the yield is relatively high based on the B_5H_9 consumed in the reaction. The selective formation of $2\text{-}CH_3B_6H_9$ under the optimum conditions indicates that an unusually facile reaction pathway is available. The generally low percentage of the starting reagents consumed, even under the most favorable conditions, suggests that conditions that favor conversion of B_5H_9 are also likely to favor decomposition of B_5H_9 and $2\text{-}CH_3B_6H_9$. The overall reaction can be considered an example of a formal insertion process in which a CH_3B moiety, which is formally analogous to a carbene, is inserted into a neutral borane framework. This reaction appears to be the only example of its type.

$1\text{-}(CH_3)_3SiB_6H_9$. The synthesis of 1-trimethylsilylhexaborane(10), $1\text{-}(CH_3)_3SiB_6H_9$, is achieved by reaction of H_2BCl with either the 1- or $2\text{-}(CH_3)_3SiB_5H_7^-$ anion according to the idealized equation

$$Li^+(CH_3)_3SiB_5H_7^- + {}^1/_3B_2H_6 + {}^1/_3BCl_3 \rightarrow 1\text{-}(CH_3)_3SiB_6H_9 + LiCl$$

This reaction is analogous to that of $(CH_3)_2BCl$ with LiB_5H_8 to produce μ-$[(CH_3)_2B]B_5H_8$, a moderately stable B_5H_9 derivative that is formally an insertion reaction intermediate in that it rearranges to 2,3-dimethylhexaborane(10), $2,3\text{-}(CH_3)_2B_6H_8$, in ethereal solvents at room temperature.[6] In the present case, it appears that the intermediate μ-H_2B-$(CH_3)_3SiB_5H_7$ may rearrange to $1\text{-}(CH_3)_3SiB_6H_9$ and also decompose to the $(CH_3)_3SiB_5H_8$ starting material. Furthermore, the apically substituted derivative is the only isomer obtained whether the precursor is 2- or $1\text{-}(CH_3)_3SiB_5H_7^-$, and the yield is low. These data suggest that $1\text{-}(CH_3)_3SiB_6H_9$ is much more thermodynamically stable than is $2\text{-}(CH_3)_3SiB_6H_9$ and that a facile rearrangement pathway is available. Studies of B_5H_9 derivative isomers have shown that $2\text{-}CH_3B_5H_8$ is much more thermodynamically stable than $1\text{-}CH_3B_5H_8$. When the substituent is H_3Si or $(CH_3)_3Si$, however, the thermodynamic stabilities are reversed and the barriers to internal rearrangements are lower. Our results, along with prior results,[7,8] suggest that similar trends obtain for B_6H_{10} derivatives.

The molecular structure of $1\text{-}(CH_3)_3SiB_6H_9$ was deduced primarily from its ^{11}B nmr spectrum, Figure 1, which is consistent with spectra of other B_6H_{10} derivatives, as indicated in Table II, and very different from the spectra of the B_5H_9 derivatives.[12] The ^{11}B chemical shifts of resonances arising from boron atoms in the base of the B_6H_{10} pentagonal pyramid are shifted to a very low-field region compared to the shifts of boron atoms in other neutral boranes. As can be seen in Table II, the B(1) resonance in $1\text{-}(CH_3)_3SiB_6H_9$ is shifted upfield by 4 ppm from the B(1) resonance in B_6H_{10}. Similar upfield shifts were observed for B(1) in $1\text{-}(CH_3)_3SiB_5H_8$ and $1\text{-}H_3SiB_5H_8$ compared to B_5H_9.[13]

The ^1H nmr spectrum of $1\text{-}(CH_3)_3SiB_6H_9$, obtained at 100 MHz, shows a quartet centered at τ 5.87 ($J = 147$ Hz) due to the five basal terminal hydrogen atoms. This shift value is in the same range as found for B_6H_{10} and its methyl derivatives. A large, broad resonance assigned to the four bridge protons occurs at τ 11.47. The ^1H spectra of B_5H_9 and its derivatives show τ values between 7 and 8 for the basal terminal hydrogens. In addition the structural assignments of B_6H_{10} derivatives are supported by the occurrence of a characteristic B-H-B band at 1900-1950 cm^{-1} in their infrared spectra.

$1\text{-}(CH_3)_3GeB_6H_9$. The method of synthesis of 1-trimethylgermylhexaborane(10), $1\text{-}(CH_3)_3GeB_6H_9$, was as described for $1\text{-}(CH_3)_3SiB_6H_9$, with the $2\text{-}(CH_3)_3GeB_5H_7^-$ anion in place of $(CH_3)_3SiB_5H_7^-$. However, no $[2\text{-}(CH_3)_3Ge](\mu\text{-}H_2B)B_5H_7$ was isolated.

The ^{11}B and ^1H nmr and infrared spectra of $1\text{-}(CH_3)_3GeB_6H_9$ are very similar to those of $1\text{-}(CH_3)_3SiB_6H_9$. ^{11}B nmr data are given in Table II.

Experimental Section

Methods and Materials. Standard high-vacuum techniques were employed in the handling of these volatile air-sensitive materials.[14] The ^{11}B and ^1H nmr spectra were obtained on a Varian XL-100 spectrometer operating at 32.1 and 100 MHz, respectively. Mass spectra were obtained using an AEI MS-9 spectrometer. The B_5H_9, B_2H_6, and BCl_3 were obtained from laboratory supplies. The trimethylborane was prepared *via* the reaction

$$BCl_3 + 3(CH_3)_4Sn \rightarrow B(CH_3)_3 + 3(CH_3)_3SnCl$$

Trimethylgallium was prepared by the method of Gaines, Borlin, and Fody.[15] 1- and $2\text{-}(CH_3)_3SiB_5H_8$ and $2\text{-}(CH_3)_3GeB_5H_8$ were prepared as previously described.[13,16]

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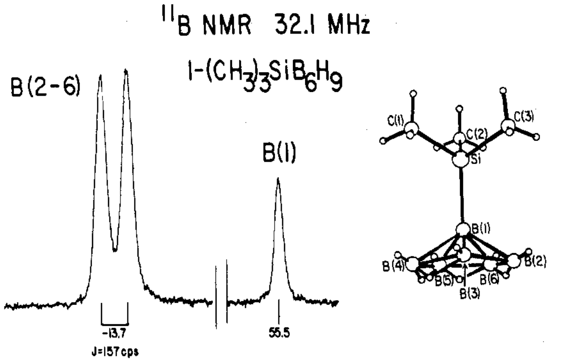

Figure 1. The ^{11}B nmr spectrum and structure of 1-$(CH_3)_3SiB_6H_9$.

Table II. Boron-11 Nmr Data for Selected Hexaborane(10) Derivatives (Ambient Conditions)

Compd	Assignment	δ	*J*, Hz
B_6H_{10}[a]	B(1)	+51.2	182 ± 5
	B(2–6)	−15.0	160 ± 5
1-$(CH_3)_3SiB_6H_9$	B(1)	+55.5	
	B(2–6)	−13.7	157
1-$(CH_3)_3GeB_6H_9$	B(1)	+53.3	
	B(2–6)	−13.7	156
2-$CH_3B_6H_9$	B(1)	+49.9 (49.4)[b]	152
	B(3, 6)	−6.8 (−6.5)	150
	B(4, 5)	−18.0 (−17.6)	157
	B(2)	−30.4 (−29.4)	
2,3-$(CH_3)_2B_6H_8$	B(1)	+48.4	152
	B(4, 6)	−13.1	152
	B(5)	−17.8	160
	B(2, 3)	−20.2	

[a] T. P. Onak, H. Landesman, R. E. Williams, and I. Shapiro, *J. Phys. Chem.*, 63, 1533 (1959). [b] Values from ref 8.

Preparation of 2-$CH_3B_6H_9$. In a typical reaction 12.90 mmol of B_5H_9, 12.90 mmol of $(CH_3)_3B$, and 2.81 mmol of $(CH_3)_3Ga$ were condensed into a 1-l. Pyrex reaction flask equipped with a seal-off constriction and a break-tip. The flask was then sealed and heated at 175° for 5 hr. Deposition of an opaque gray film occurred on the walls of the flask shortly after heating was begun. The film was uneven in that parts were dull gray and other parts were metallic gray (the metallic patches were formed mostly at the bottom of the reaction vessel). After reaction 11.62 mmol of B_5H_9 (90.1%) and 15.54 mmol of $(CH_3)_3B$ (120.5%) were recovered (the recovery of more than 100% of $(CH_3)_3B$ is due to the reaction between $(CH_3)_3Ga$ and B_5H_9 to form trimethylborane). No $(CH_3)_3Ga$ was recovered. The 2-$CH_3B_6H_9$ was isolated by condensation in a trap at −78°. The yield of 2-$CH_3B_6H_9$ was 0.453 mmol (0.0402 g), or 35.5% based on B_5H_9 consumed in the reaction. Other representative reactions are summarized in Table I. The characterization of the 2-$CH_3B_6H_9$ was by means of infrared, ^{1}H nmr, and ^{11}B nmr spectra, all of which were in agreement with those reported by Johnson, Brice, and Shore[7,8] (see Table II). The mass spectrum exhibited a strong parent ion. As a check the exact *m/e* was determined for the $^{12}C^1H_3{}^{10}B^{11}B_5{}^1H_9{}^+$ ion: calcd, 89.1534; found, 89.1537. Reagents that did not assist the synthesis of 2-$CH_3B_6H_9$ include gallium, aluminum, and copper, $Mn_2(CO)_{10}$, and $GaCl_3$; ultraviolet irradiation and a large increase in surface area also did not help.

Preparation of 1-$(CH_3)_3SiB_6H_9$. In a typical preparation, 2-$(CH_3)_3SiB_5H_7{}^-$ was prepared by condensing 9.0 mmol of 2-$(CH_3)_3SiB_5H_8$ into a diethyl ether solution of 9.0 mmol of *n*-butyllithium. The solution was warmed from −78 to −30° over a 90-min period. A mixture of 3.0 mmol of B_2H_6 and 3.0 mmol of BCl_3 was then allowed to expand into this stirred solution of 2-$(CH_3)_3SiB_5H_7{}^-$ at −78°. The solution took on a slight green color at this point. The reactants were stirred 2 hr while the temperature gradually rose to −30°. Near the end of the 2 hr, the reaction mixture appeared as a dark mustard-colored suspension. When the stirring was stopped, a white solid settled and the solution above was clear, dark red-orange. The 1-$(CH_3)_3SiB_6H_9$ was isolated and purified by high-vacuum fractional distillation through a trap at −22° and by condensation in a −36° trap. The yield of 1-$(CH_3)_3SiB_6H_9$ was less than 5%, and much of the original $(CH_3)_3SiB_5H_8$ was recovered. Another product, formed in higher yield, had a volatility in the same range as 1-$(CH_3)_3SiB_6H_9$, so that the two were inseparable by trap-to-trap distillation. A ^{11}B nmr spectrum of this mixture indicated that this second product was most likely $[2\text{-}(CH_3)_3Si](\mu\text{-}H_2B)B_5H_7$, formed by insertion of a BH_2 group into a bridging position on 2-$(CH_3)_3SiB_5H_7{}^-$. The mixture of this compound and 1-$(CH_3)_3SiB_6H_9$ was stirred with diethyl ether at room temperature in order to form more 1-$(CH_3)_3SiB_6H_9$. Though some decomposition to 2-$(CH_3)_3SiB_5H_8$ was noted, this procedure proved to be an effective means of obtaining pure 1-$(CH_3)_3SiB_6H_9$, since it is easily separated from the 2-$(CH_3)_3SiB_5H_8$. Similar yields of 1-$(CH_3)_3SiB_6H_9$ were obtained when the B_5 anion was 1-$(CH_3)_3SiB_5H_7{}^-$.

Several attempts were made to develop a higher yield synthesis of 1-$(CH_3)_3SiB_6H_9$. These included changing from the diethyl ether solvent to isopropyl ether, preforming the H_2BCl before adding it to the $(CH_3)_3SiB_5H_7{}^-$ anion solution, and treating the final reaction mixture with anhydrous HCl to recover any $(CH_3)_3SiB_6H_8{}^-$ that might have formed. In no case was the yield of 1-$(CH_3)_3SiB_6H_9$ significantly improved.

The 1-$(CH_3)_3SiB_6H_9$ is a colorless liquid that melts just below room temperature and has a vapor pressure of about 1 mm at 25°. Its infrared spectrum, obtained as a thin film at −196°, showed the following absorptions: 2960 (m) and 2905 (w) (C-H stretch), 2605 (sh) and 2580 (s) (B-H stretch), 1935 (w) (B-H-B), 1480 (m), 1255 (s), 1110 (w), 1030 (w), 965 (w), 880 (mw), 845 (sh), 835 (s), 690 (sh), and 675 (s) cm^{-1}. The mass spectrum exhibited a strong parent ion group with a cutoff at *m/e* 148. The exact *m/e* was determined for the $(^{12}C^1H_3)_3{}^{28}Si^{10}B^{11}B_5{}^1H_9{}^+$ ion: calcd, 147.1773; found, 147.1780.

Preparation of 1-$(CH_3)_3GeB_6H_9$. An ethereal solution of 15 mmol of 2-$(CH_3)_3GeB_5H_7{}^-$ was prepared by warming a diethyl ether solution of 15 mmol of 2-$(CH_3)_3GeB_5H_8$ and 15 mmol of *n*-butyllithium from −78 to −40° over a 2-hr period. A mixture of 5 mmol of BCl_3 and 5 mmol of B_2H_6 was expanded into this solution while it was being stirred at −78°. This temperature was maintained for 1 hr and then was raised gradually over a 2-hr period to −30°. The appearance of the reaction mixture at both start and finish was as described in the 1-$(CH_3)_3SiB_6H_9$ synthesis. The 1-$(CH_3)_3GeB_6H_9$ product was purified by high-vacuum distillation through a trap at −22° and by condensation in a −30° trap. The yield was approximately 3%.

The 1-$(CH_3)_3GeB_6H_9$ is a colorless liquid with a vapor pressure of less than 1 mm at 25°. A sharp cutoff at *m/e* 194 was observed in the mass spectrum. The parent group, however, was of low intensity compared to that corresponding to the loss of a methyl group. Exact *m/e* determinations were therefore obtained for **1**, $(^{12}C^1H_3)_3{}^{74}Ge^{11}B_6H_9{}^+$ [calcd, 194.1186; found, 194.1199], and for **2**, $(^{12}C^1H_3)_2{}^{74}Ge^{11}B_6H_9{}^+$ [calcd, 179.0951; found, 179.0953].

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Registry No. 2-$CH_3B_6H_9$, 50860-40-7; 1-$(CH_3)_3SiB_6H_9$, 50860-41-8; 1-$(CH_3)_3GeB_6H_9$, 50860-42-9; $(CH_3)_3B$, 593-90-8; B_5H_9, 19624-22-7; $(CH_3)_3Ga$, 1445-79-0; 1-$(CH_3)_3SiB_5H_8$, 28323-19-5; 2-$(CH_3)_3SiB_5H_8$, 22142-53-6; B_2H_6, 19287-45-7; BCl_3, 10294-34-5; 2-$(CH_3)_3GeB_5H_8$, 22142-56-9.