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Crystal and Molecular Structure of 9-(Triethylamine)-6-thiadecaborane(11)

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The crystal and molecular structure of 9-(triethylamine)-6-thiadecaborane(11) was determined by single-crystal X-ray diffraction techniques. The crystals form in the monoclinic space group $P2_1/n$ with $a = 12.142$ (2), $b = 9.547$ (2), and $c = 13.553$ (2) Å, with $\beta = 107.71$ (1)°, and with $Z = 4$. The conventional R index converged at 0.049 with full-matrix least-squares refinement including the overall scale factor, all positional parameters and all anisotropic temperature factors for nonhydrogen atoms, and fixed isotropic temperature factors for hydrogen atoms. The triethylamine ligand is attached to the thiaborane cage in an exopolyhedral manner at the B9 position, with a B–N distance of 1.600 (4) Å. This position of addition probably models the initial mode of interaction of alkenes and alkynes during hydroboration by SB_9H_{11}. The sulfur occupies the 6 position and is bound to three borons with an average distance of 1.927 (5) Å. The B–B distances of the cage range from 1.723 (5) to 1.904 (5) Å. The bridging hydrogens on the open face are significantly displaced toward B9 and away from the sulfur.

Introduction

Recent synthetic studies in these laboratories yielded a number of *nido*-platinathiaboranes containing the decaborane-like framework with sulfur and platinum occupying the 6 and 9 positions, respectively.[1] The structures of decaborane[2] and its bis(acetonitrile)[3] and bis(dimethyl sulfide)[4] adducts have been determined, as well as that of $[(Ph_3P)_3Au][SB_9H_{12}]$,[5] containing the arachno 10-vertex $SB_9H_{12}^-$ cage. However, no structural data exist for an unmetalated nido 10-vertex thiaborane. A number of Lewis-base adducts of 6-SB_9H_{11} have been prepared but the position of base attachment was not defined.[6] For instance, the reaction of triethylamine with 6-SB_9H_{11} forms the *arachno*-NEt_3-6-SB_9H_{11}.[6] The single-crystal X-ray examination reported here shows attachment at the 9 position and defines the structural relationships of 9-NEt_3-6-SB_9H_{11} with platinathiaboranes, decaborane (its derivatives), and $SB_9H_{12}^-$.

Experimental Section

The synthesis of 9-NEt_3-6-SB_9H_{11} is described by Hertler, Klanberg, and Muetterties.[6] A large, pale, yellow, single crystal of the triethylamine complex was grown by slow evaporation from a benzene solution under a nitrogen atmosphere. The crystal chosen for data collection was cleaved from the large crystal and mounted into a Lindemann capillary tube which was then flame sealed.

The crystal was then placed on a Syntex $P2_1$ automated four-circle diffractometer. A trial data set indicated the crystal to be monoclinic having space group symmetry of $P2_1/n$ (a nonstandard setting of $P2_1/c$). An accurate determination of the unit-cell parameters was obtained from a least-squares fit of 15 reflections whose angular settings were refined. The unit cell parameters are $a = 12.142$ (2) Å, $b = 9.547$ (2) Å, $c = 13.553$ (2) Å, and $\beta = 107.71$ (1)°. The cell volume is 1496.5 (5) Å^3. The density observed by flotation is 1.10 g cm^{-3} and is in good agreement with the calculated density for four molecules of $C_6H_{26}B_9NS$, fw 241.64, at 1.072 g cm^{-3}.

The intensity data were measured with previously described techniques.[1] One quadrant of reciprical space was measured for $+h$, $+k$, $\pm l$ to a 2θ limit of 55°. Three standard reflections, $(5\bar{1}2)$, $(3\bar{3}\bar{5})$, and $(1\bar{2}\bar{6})$, were monitored every 50 reflections, and their intensities showed no statistical deviation about the mean. Of the 4079 reflections collected, 1666 had intensities greater than 3 times its standard deviation. The value of μ for Mo Kα radiation is 1.82 cm^{-1}, and no corrections for absorption were deemed necessary. A summary of crystal data and intensity information is given in Table I.

Structure Determination and Refinement

The structure was solved by direct methods using MULTAN.[7] The correct E map indicated the positions of all nonhydrogen atoms. The atomic scattering factors for the nonhydrogen atoms were calculated with the analytical expressions of Cromer and Waber.[8] The hydrogen scattering factors were obtained from Stewart, Davidson, and Simpson.[9] Following initial refinement of the scale factor and two cycles of refining positional parameters of all nonhydrogen atoms, the reliability indices were $R_1 = 0.171$ and $R_2 = 0.204$.[10] Two additional cycles of full-matrix refinement including isotropic thermal parameters converged to $R_1 = 0.141$ and $R_2 = 0.181$. Two cycles with anisotropic temperature factors resulted in convergence at $R_1 = 0.106$ and $R_2 = 0.150$. A difference map was calculated to locate hydrogen positions. All hydrogens were found, and refinement of their x, y, and z parameters led to $R_1 = 0.069$ and $R_2 = 0.074$. Three cycles of full-matrix least-squares refinement of the x, y, z parameters of all atoms, the anisotropic temperature factors of the nonhydrogen atoms, and a scale factor resulted in convergence with $R_1 = 0.049$ and $R_2 = 0.053$. During the final three cycles, the isotropic temperature factors for the hydrogen atoms were fixed at a value one unit higher than the isotropic temperature value of the atom to which it was bound. The average shift in all parameters was 0.6 of an esd during the final cycle. The quantity minimized was $\sum w(F_o - F_c)^2$. After the final refinement, the standard deviation of an observation of unit weight was $[\sum w(|F_o| - |F_c|)^2/(m - n)]^{1/2}1.560$, where the number of reflections (m) was 1666 and the number of refined parameters (n) was 232. The maximum density in the final difference Fourier synthesis was 0.28 e Å^{-3}.

Table I. Summary of Crystal Data and Intensity Collection for 9-NEt_3-6-SB_9H_{11}

mol formula	$C_6H_{26}NB_9S$
space group	$P2_1/n$
a, Å	12.142 (2)
b, Å	9.547 (2)
c, Å	13.553 (2)
β, deg	107.71 (1)
V, Å^3	1496.6 (5)
mol wt	241.64
Z	4
d_{obsd}, g/cm^3	1.10
d_{calcd}, g/cm^3	1.072
cryst dimens, mm	0.277 × 0.271 × 0.341
cryst shape	parallelepiped
radiation, Å	λ(Mo Kα) 0.710 69, monochromatized from a graphite cryst
linear abs coeff μ, cm^{-1} (Mo Kα)	1.82
scan speed, deg/min	variable, 2.0–12.0
scan range, 2θ, deg	Mo Kα_1 – 0.8 to Mo Kα_2 + 0.8
ratio of bkgd scan time to peak scan time	0.8
std reflecns	$(5\bar{1}2)$ $(3\bar{3}\bar{5})$ $(1\bar{2}\bar{6})$
dev of stds during data collecn	no statistical dev about the mean
2θ limit, deg	55
reflecns collected	4079
reflecns with $F^2 \geqslant 3\sigma(F^2)$	1666
R factor	0.049
R_w factor	0.053

The positional and thermal parameters along with their associated standard deviations obtained from the final cycle of refinement are given in Tables II and III.

Results and Discussion

The intramolecular distances for 9-NEt_3-6-SB_9H_{11} and their esd's are listed in Table IV. The intramolecular bond angles

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Table II. Final Positional Parameters for $9\text{-}NEt_3\text{-}6\text{-}SB_9H_{11}$

atom	x	y	z	B_{iso}, Å^2
S	0.2640 (1)	0.8739 (1)	0.8410 (1)	
B1	0.0398 (3)	0.7341 (4)	0.8395 (3)	
B2	0.1731 (4)	0.7033 (4)	0.8187 (4)	
B3	0.0516 (4)	0.7208 (4)	0.7097 (3)	
B4	−0.0454 (3)	0.8448 (4)	0.7375 (3)	
B5	0.1541 (4)	0.8378 (4)	0.9149 (3)	
B7	0.1733 (4)	0.8151 (4)	0.7045 (3)	
B8	0.0267 (3)	0.8875 (4)	0.6488 (3)	
B9	−0.0061 (3)	1.0186 (4)	0.7420 (3)	
B10	0.0069 (4)	0.9099 (4)	0.8628 (3)	
N	−0.1035 (2)	1.1371 (2)	0.7070 (2)	
C11	−0.2269 (3)	1.0856 (4)	0.6651 (3)	
C12	−0.2587 (4)	1.0197 (5)	0.5587 (3)	
C21	−0.0718 (3)	1.2283 (4)	0.6277 (3)	
C22	−0.1481 (4)	1.3541 (4)	0.5900 (4)	
C31	−0.1016 (3)	1.2226 (4)	0.8028 (3)	
C32	0.0094 (4)	1.2972 (5)	0.8572 (4)	
H1	−0.0012 (34)	0.6426 (43)	0.8710 (30)	4.7
H2	0.2168 (34)	0.6056 (44)	0.8368 (30)	5.0
H3	0.0184 (34)	0.6272 (44)	0.6629 (31)	4.7
H4	−0.1354 (32)	0.8186 (40)	0.7078 (30)	4.1
H5	0.1895 (35)	0.8246 (42)	0.9940 (32)	5.0
H7	0.2249 (33)	0.7916 (43)	0.6569 (31)	4.6
H8	−0.0154 (33)	0.8929 (41)	0.5660 (30)	4.4
H9	0.0771 (32)	1.0721 (38)	0.7698 (27)	3.5
H10	−0.0530 (32)	0.9227 (38)	0.9109 (29)	4.3
H5,10	0.0961 (34)	0.9612 (44)	0.9015 (30)	4.5
H7,8	0.1207 (33)	0.9370 (42)	0.6669 (29)	4.5
H111	−0.2756 (33)	1.1661 (44)	0.6617 (30)	4.1
H112	−0.2386 (33)	1.0196 (44)	0.7187 (31)	4.1
H121	−0.2611 (39)	1.0864 (49)	0.5069 (35)	5.5
H122	−0.3411 (38)	0.9935 (48)	0.5421 (33)	5.5
H123	−0.2054 (39)	0.9480 (49)	0.5499 (34)	5.5
H211	−0.0690 (36)	1.1642 (45)	0.5690 (33)	4.6
H212	0.0099 (36)	1.2540 (42)	0.6605 (31)	4.6
H221	−0.1422 (40)	1.4226 (50)	0.6382 (37)	5.7
H222	−0.2352 (42)	1.3309 (47)	0.5565 (35)	5.7
H223	−0.1179 (39)	1.3933 (50)	0.5390 (38)	5.7
H311	−0.1220 (34)	1.1535 (42)	0.8508 (31)	4.4
H312	−0.1622 (35)	1.2929 (47)	0.7862 (31)	4.4
H321	0.0733 (40)	1.2304 (52)	0.8858 (33)	5.6
H322	0.0354 (41)	1.3593 (50)	0.8146 (36)	5.6
H323	0.0032 (38)	1.3499 (50)	0.9183 (36)	5.6

Table III. Final Thermal Parameters for $9\text{-}NEt_3\text{-}6\text{-}SB_9H_{11}$[a]

atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
S	3.0 (0)	4.9 (1)	6.2 (1)	0.5 (0)	1.3 (0)	0.3 (0)
B1	4.6 (2)	3.4 (2)	4.3 (2)	0.4 (2)	2.0 (2)	0.9 (2)
B2	4.9 (2)	3.2 (2)	5.2 (2)	1.1 (2)	2.3 (2)	0.5 (2)
B3	4.6 (2)	2.7 (2)	4.6 (2)	0.5 (2)	1.9 (2)	−0.4 (2)
B4	3.1 (2)	2.9 (2)	4.0 (2)	−0.1 (1)	1.5 (1)	0.1 (1)
B5	4.5 (2)	4.1 (2)	3.6 (2)	1.0 (2)	1.1 (2)	0.5 (2)
B7	4.7 (2)	3.5 (2)	4.8 (2)	1.1 (2)	2.7 (2)	0.3 (2)
B8	4.1 (2)	3.1 (2)	3.5 (2)	0.5 (2)	1.7 (2)	−0.1 (2)
B9	2.7 (1)	2.5 (1)	3.1 (2)	0.1 (1)	1.4 (1)	−0.1 (1)
B10	3.8 (2)	3.9 (2)	3.2 (2)	0.6 (2)	1.7 (2)	0.1 (1)
N	2.5 (1)	2.9 (1)	3.3 (1)	0.1 (1)	1.1 (1)	−0.3 (1)
C11	2.5 (1)	3.7 (2)	4.6 (2)	−0.3 (1)	0.8 (1)	−0.6 (1)
C12	4.2 (2)	4.7 (2)	4.5 (2)	−0.3 (2)	0.1 (2)	−0.3 (2)
C21	3.8 (2)	3.3 (2)	4.7 (2)	−0.1 (1)	1.7 (1)	0.9 (1)
C22	5.7 (2)	3.7 (2)	6.6 (3)	0.6 (2)	1.6 (2)	1.3 (2)
C31	3.4 (1)	3.6 (2)	4.5 (2)	0.4 (1)	1.4 (1)	−1.4 (1)
C32	4.8 (2)	4.5 (2)	6.1 (3)	−0.4 (2)	0.7 (2)	−2.5 (2)

[a] The anisotropic thermal parameters have units of Å^2. They enter the expression for the structure factor in the form $\exp[-\frac{1}{4}\cdot(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Table IV. Intramolecular Distances (Å) for $9\text{-}NEt_3\text{-}6\text{-}SB_9H_{11}$

	Nonhydrogen Atoms		
S–B2	1.939 (4)	B4–B8	1.736 (5)
S–B5	1.928 (4)	B4–B9	1.723 (5)
S–B7	1.925 (4)	B4–B10	1.737 (6)
B1–B2	1.750 (6)	B5–B10	1.843 (6)
B1–B3	1.812 (6)	B7–B8	1.844 (6)
B1–B4	1.796 (6)	B8–B9	1.904 (5)
B1–B5	1.757 (6)	B9–B10	1.904 (5)
B1–B10	1.775 (6)	B9–N	1.600 (4)
B2–B3	1.750 (6)	N–C11	1.514 (4)
B2–B5	1.894 (6)	N–C21	1.521 (4)
B2–B7	1.880 (6)	N–C31	1.528 (4)
B3–B4	1.789 (5)	C11–C12	1.512 (5)
B3–B7	1.749 (6)	C21–C22	1.507 (5)
B3–B8	1.776 (5)	C31–C32	1.505 (5)
	Hydrogen Atoms		
B1–H1	1.150 (40)	C11–H111	0.962 (41)
B2–H2	1.064 (40)	C11–H112	1.005 (41)
B3–H3	1.099 (41)	C12–H121	0.942 (47)
B4–H4	1.073 (36)	C12–H122	0.987 (43)
B5–H5	1.035 (40)	C12–H123	0.974 (47)
B7–H7	1.051 (40)	C21–H211	1.012 (43)
B8–H8	1.084 (38)	C21–H212	0.987 (41)
B9–H9	1.091 (36)	C22–H221	0.912 (47)
B10–H10	1.122 (37)	C22–H222	1.039 (47)
B5–H5,10	1.356 (41)	C22–H223	0.953 (49)
B7–H7,8	1.350 (39)	C31–H311	1.009 (42)
B8–H7,8	1.189 (38)	C31–H312	0.971 (44)
B10–H5,10	1.159 (39)	C32–H321	0.988 (49)
		C32–H322	0.946 (47)
		C32–H323	0.991 (48)

Table V. Selected Bond Angles (deg) for $9\text{-}NEt_3\text{-}6\text{-}SB_9H_{11}$

B2–S–B5	58.6 (2)	S–B2–B1	110.1 (3)
B2–S–B7	58.2 (2)	S–B2–B3	110.2 (3)
B5–S–B7	99.3 (2)	S–B5–B1	110.3 (3)
S–B2–B5	60.4 (2)	S–B5–B10	118.5 (3)
S–B2–B7	60.5 (2)	S–B7–B3	110.8 (3)
S–B5–B2	61.0 (2)	S–B7–B8	118.9 (2)
S–B7–B2	61.3 (2)	B2–B1–B4	108.6 (3)
B2–B1–B3	58.8 (2)	B2–B1–B10	117.2 (3)
B2–B1–B5	65.3 (2)	B3–B1–B5	108.8 (3)
B3–B1–B4	59.4 (2)	B4–B1–B5	106.5 (3)
B4–B1–B10	58.2 (2)	B3–B1–B10	109.1 (3)
B5–B1–B10	62.9 (2)	B1–B2–B7	105.6 (3)
B1–B2–B3	62.4 (2)	B3–B2–B5	105.6 (3)
B1–B2–B5	57.5 (2)	B5–B2–B7	102.3 (3)
B3–B2–B7	57.5 (2)	B1–B3–B7	108.6 (3)
B1–B3–B2	58.8 (2)	B1–B3–B8	109.6 (3)
B1–B3–B4	59.9 (2)	B2–B3–B4	109.0 (3)
B2–B3–B7	65.0 (2)	B2–B3–B8	117.3 (3)
B4–B3–B8	58.3 (2)	B4–B3–B7	106.6 (3)
B7–B3–B8	63.1 (2)	B1–B4–B8	112.2 (3)
B1–B4–B3	60.7 (2)	B3–B4–B10	111.9 (3)
B1–B4–B10	60.3 (2)	B8–B4–B10	118.7 (3)
B3–B4–B8	60.5 (2)	B1–B4–B9	116.8 (3)
B8–B4–B9	66.8 (2)	B3–B4–B9	116.9 (3)
B9–B4–B10	66.8 (2)	B2–B5–B10	107.2 (3)
B1–B5–B2	57.2 (2)	B2–B7–B8	107.9 (3)
B1–B5–B10	59.0 (2)	B4–B8–B7	104.8 (3)
B2–B7–B3	57.5 (2)	B3–B8–B9	108.7 (3)
B3–B7–B8	59.2 (2)	B7–B8–B9	110.2 (3)
B3–B8–B4	61.2 (2)	B8–B9–B10	103.4 (2)
B3–B8–B7	57.8 (2)	B1–B10–B9	109.1 (3)
B4–B8–B9	56.2 (2)	B5–B10–B9	110.5 (3)
B4–B9–B8	56.9 (2)	B4–B10–B5	105.4 (3)
B4–B9–B10	57.0 (2)	B1–B10–B4	61.5 (2)
B4–B10–B9	56.2 (2)	B1–B10–B5	58.1 (2)
N–B9–B4	119.9 (2)	B9–N–C11	116.1 (2)
N–B9–B8	123.5 (2)	B9–N–C21	107.0 (2)
N–B9–B10	120.0 (2)	B9–N–C31	107.9 (2)
N–C11–C12	115.4 (3)	C11–N–C21	110.8 (2)
N–C21–C22	115.7 (3)	C11–N–C31	104.4 (2)
N–C31–C32	116.7 (3)	C21–N–C31	110.7 (3)
H9–B9–N	107.1 (19)	H9–B9–B8	100.3 (19)
H9–B9–B10	97.6 (19)	H9–B9–B4	132.9 (19)
B5–H5,10–B10	94.0 (18)	B7–H7,8–B8	92.9 (18)

(with esd's) are given in Table V.

The crystal is composed of discrete molecular entities of $9\text{-}NEt_3\text{-}6\text{-}SB_9H_{11}$ which are separated by normal van der Waals distances. There are no abnormally short intermolecular distances. A view of the molecule with the numbering system is shown in Figure 1. Since the molecules lie in general

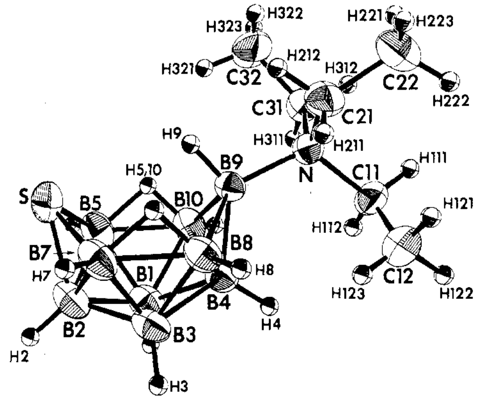

Figure 1. Numbering convention and ORTEP plot of the molecular structure with nonhydrogen ellipsoids at 50% confidence level. Hydrogens are artifically reduced for clarity.

positions with each atom crystallographically independent, a rigorous test of the molecular symmetry may be made. Locally, the thiaborane ligand is nearly symmetrical with a mirror plane through atoms S, B2, B4, and B9. Only the distances B2–B5 and B2–B7 (1.894 (6) and 1.880 (6) Å, respectively) do not lie within one standard deviation of one another.

Not surprisingly, the molecule is similar to $SB_9H_{12}^-$,[5] since in a formal electron-bookkeeping sense, the triethylamine derivative is obtained from the anion by replacement of :H^- with :NEt_3 to yield a neutral product. The triethylamine ligand occupies an exo position analogous to the acetonitrile[3] and dimethyl sulfide[4] adducts of decaborane.

The bond distances in 9-NEt_3-6-SB_9H_{11} compare favorably with other structures. The boron–boron distances range from 1.723 (5) to 1.904 (5) Å. In $SB_9H_{12}^-$ the limits extend from 1.72 (5) to 1.92 (4) Å. The boron–sulfur distances vary from 1.928 (4) to 1.939 (4) Å. In $(Ph_3P)_2PtSB_8H_{10}$ and $(Ph_3P)_2PtSB_8H_9(OEt)$ the B–S distances ranged from 1.91 to 1.98 Å.[1] The reported B–S average in $(Et_3P)_2Pt(H)$-SB_9H_{10}[11] is 2.05 (5) Å. Notably, the sulfur was bound directly to the platinum in the latter structure. Pure thiaboranes having no heteroatoms other than sulfur are 2,2′-$(1\text{-}SB_9H_8)_2$[12] with B–S distances of 1.918 (3)–1.930 (3) Å and $SB_9H_{12}^-$[5] with a range of 1.85 (4)–1.96 (3) Å. The boron–nitrogen distance of 1.600 (4) Å agrees well with that of 1.58 Å found in $NH_3{\cdot}B_3H_7$.[13] The nitrogen–carbon and carbon–carbon bond distances and angles are all typical and require no further comment.

An important consideration in structures of this type is the location of the bridging hydrogens on the open face of the cage. In both $B_{10}H_{12}(NCCH_3)_2$[3] and $B_{10}H_{12}(SMe_2)_2$,[4] the bridging hydrogens are symmetrically bound to atoms B5 and B10 and atoms B7 and B8 with an average distance of 1.22 and 1.28 Å, respectively. In contrast, the bridging hydrogens in 9-NEt_3-6-SB_9H_{11} are significantly displaced toward the B9 position and away from the sulfur. The distances are 1.35 (4) and 1.19 (4) Å for H7,8 and 1.35 (4) and 1.16 (4) Å for H5,10. In $SB_9H_{12}^-$[5] the corresponding distances are 1.47, 1.42 Å for H7,8 and 1.10, 1.38 Å for H5,10, with the hydrogen being displaced toward the sulfur atom. However, the bridged hydrogen distances in $SB_9H_{12}^-$ have large errors associated with them, and the significance of their asymmetrical arrangement is questionable.

The boron–boron distance bridged by a hydrogen is important and requires some comment. In our structure, distances of 1.844 (6) and 1.843 (6) Å are observed for B7–B8 and B5–B10. The corresponding distances are 1.81 (4) and 1.87 (3) Å in $SB_9H_{12}^-$, 1.881 (8) Å in $B_{10}H_{12}(NCCH_3)_2$,[3] 1.91 (2) Å in $B_{10}H_{12}(SMe_2)_2$,[4] 1.84 (2) Å in $(Ph_3P)_2PtSB_8H_{10}$,[1] and 1.86 (3) Å in $(Ph_3P)_2PtSB_8H_9(OEt)$.[1] A notable lengthening is observed if no bridging hydrogens are present. For example, 1.973 (4) Å is seen in $B_{10}H_{14}$,[2] and 2.037 Å is observed in 5-THF-6-$(CO)_3$-6-MnB_9H_{12}.[14]

The facile and clean hydroboration of alkenes and alkynes by 6-SB_9H_{11} to give 9-substituted alkyl- and alkenylthiaboranes has been reported recently.[15] Since the structure reported here shows that donor molecules attack at the 9 position of 6-SB_9H_{11} in an exo rather than endo fashion, it seems reasonable to suggest exo attack by alkenes and alkynes as an initial step of hydroboration. However, the orientation of the π system relative to the B9–H bond is not clear.

In summary, *arachno*-9-NEt_3-6-SB_9H_{11} has been shown to be structurally analogous to *arachno*-$SB_9H_{12}^-$. The amine is exo, and the bridging hydrogens are asymmetrically bound across atoms B5 and B10 and atoms B7 and B8 in the direction of B9 and away from the sulfur. The exo substituent is probably a good model for the initial interaction of alkenes and alkynes during hydroboration by 6-SB_9H_{11}.

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Registry No. 9-NEt_3-6-SB_9H_{10}, 69102-55-2.

Supplementary Material Available: Structure factor tables (7 pages). Ordering information is given on any current masthead page.

References and Notes

(1) T. K. Hilty, D. A. Thompson, W. M. Butler, and R. W. Rudolph, submitted for publication; D. A. Thompson, T. K. Hilty, and R. W. Rudolph, *J. Am. Chem. Soc.*, **99**, 6774 (1977).
(2) T. Hamilton, *Inorg. Chem.*, **8**, 464 (1969).
(3) J. Reddy and W. N. Lipscomb, *J. Chem. Phys.*, **31**, 610 (1959).
(4) A. Zalkin and D. E. Sands, *Acta Crystallogr.*, **15**, 410 (1962).
(5) L. J. Guggenberger, *J. Organomet. Chem.*, **81**, 271 (1974).
(6) W. R. Hertler, F. Klanberg, and E. L. Muetterties, *Inorg. Chem.*, **6**, 1696 (1967).
(7) Computations were carried out on an Amdahl 370 computer. Computer programs used during the structural analysis were SYNCOR (data reduction by W. Shmonsee), MULTAN (Germain, Declero, Main, Woolfson, and Lessinger), FORDAP (Fourier synthesis by A. Zalkin), ORFLS (least squares by Busing, Martin and Levy), ORFEE (distances, angles, and esd's by Busing, Martin, and Levy), and ORTEP (thermal ellipsoid drawings by C. K. Johnson).
(8) D. T. Cromer and J. T. Waber, *Acta Crystallogr.*, **18**, 104, (1965).
(9) R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, **42**, 3175 (1965).
(10) The forms of the reliability indices are $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$ and $R_2 = [\sum w(|F_o| - |F_c|)^2/w|F_o|^2]^{1/2}$.
(11) L. J. Guggenberger, A. R. Kane, and E. L. Muetterties, *J. Am. Chem. Soc.*, **92**, 2571 (1970).
(12) W. R. Pretzer, T. K. Hilty, and R. W. Rudolph, *Inorg. Chem.*, **14**, 2459 (1975).
(13) C. E. Nordman, *Acta Crystallogr.*, **10**, 777 (1957); C. E. Nordman and C. Reimann, *J. Am. Chem. Soc.*, **81**, 3538 (1959).
(14) J. W. Lott and D. F. Gaines, *Inorg. Chem.*, **13**, 2261 (1974).
(15) B. J. Meneghelli and R. W. Rudolph, *J. Am. Chem. Soc.*, **100**, 4626 (1978).