Contribution from the Rohm and Haas Company,
Redstone Arsenal Research Division, Huntsville, Alabama

The Preparation and Reactions of $B_{10}H_{13}$ (Ligand) Anions

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A series of salts which have the general formula $M^+B_{10}H_{13}X^-$ (where X is an amine or phosphine ligand) have been prepared by the reaction of the $B_{10}H_{13}$ anion with representative amines and phosphines. The reactions of these materials include: (1) quantitative protonation to form an unisolated $B_{10}H_{14}X$ species; (2) conversion to $B_{10}H_{12}X_2$ and $B_{10}H_{12}XX'$ derivatives by reaction with HX or HX', respectively, where HX and HX' are amine hydrochlorides; and (3) aqueous acidolysis to produce $B_9H_{13}X$ derivatives, boric acid, and hydrogen.

It is a well documented fact[1,2] that the decaborane molecule may function as a strong monoprotic Brönsted acid

$$B_{10}H_{14} + H_2O \rightleftharpoons B_{10}H_{13}^- + H_3O^+$$

The acidic character of the four bridge hydrogen atoms of the decaborane molecule has been demonstrated by their rapid exchange with deuterium oxide.[3,4] The recent description of many salts which contain the $B_{10}H_{13}$ anion[2] demonstrates the stability of this species. Lipscomb[5] has proposed a plausible structure for the $B_{10}H_{13}$ anion based upon his topological system for boron hydrides.

We wish to report a study of the reaction of the $B_{10}H_{13}$ anion with neutral Lewis bases, X, to produce $B_{10}H_{13}X$ anions and the utility of these new ions in further synthetic studies.[6]

Results and Discussion

In the recently described study of the various salts of the $B_{10}H_{13}$ anion,[2] a compound $B_{10}H_{14}\cdot 2(C_2H_5)_2NH$ (I) was reported from the reaction of decaborane with excess diethylamine in cyclohexane solution. Conductivity measurements with aqueous solutions of this material proved its ability to conduct an electric current. This same salt was produced in high yield from the slow reaction of diethylammonium decaborane and an additional equivalent of diethylamine. Potentiometric titration of this material with hydrogen chloride in anhydrous dioxane resulted in a single end point from which an average equivalent weight of 264 was obtained (formula weight 268). Hydrogen *was not* evolved under these titration conditions. The products of these titrations were diethylammonium chloride, which was isolated and identified by its infrared spectrum, and an unisolated species, assumed to be $B_{10}H_{14}NH(C_2H_5)_2$. This latter material would be a substituted $B_{10}H_{15}$ anion.[7] Thus, the over-all reaction of decaborane with two equivalents of diethylamine may be described by eq. 1.

$$B_{10}H_{14} + (C_2H_5)_2NH \overset{\text{fast}}{\rightleftharpoons} B_{10}H_{13}^- + (C_2H_5)_2NH_2^+$$
$$B_{10}H_{13}^- + (C_2H_5)_2\overset{+}{N}H_2 + (C_2H_5)_2NH \xrightarrow{\text{slow}} [B_{10}H_{13}NH(C_2H_5)_2]^- + (C_2H_5)_2\overset{+}{N}H_2 \quad (1)$$
$$(C_2H_5)_2\overset{+}{N}H_2 + \overset{-}{Cl} + B_{10}H_{14}NH(C_2H_5)_2 \xleftarrow{\text{HCl}}$$

A more general method for the preparation of $B_{10}H_{13}X$ anions was developed by making use of the readily available sodium salt of decaborane. Previous work[3] has shown that sodium hydride and decaborane in diethyl ether solvent react as shown in (2)

$$NaH + B_{10}H_{14} \xrightarrow{(C_2H_5)_2O} NaB_{10}H_{13} + H_2 \quad (2)$$

The salt, $NaB_{10}H_{13}$, is yellow in color and soluble in diethyl ether. Addition of one equivalent of pyridine, triphenylphosphine, diethylamine, ethylamine, or triethylamine to diethyl ether solutions

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* Department of Chemistry, The University of California, Riverside, Calif.

[1] G. A. Guter and G. W. Schaeffer, *J. Am. Chem. Soc.*, **78**, 3546 (1956).

[2] M. F. Hawthorne, A. R. Pitochelli, R. D. Strahm, and J. J. Miller, *ibid.*, **82**, 1825 (1960).

[3] M. F. Hawthorne and J. J. Miller, *ibid.*, **80**, 754 (1958).

[4] I. Shapiro, M. Lustig, and R. E. Williams, *ibid.*, **81**, 838 (1959).

[5] W. N. Lipscomb, *J. Inorg. Nucl. Chem.*, **11**, 1 (1959), and *Advan. Inorg. Chem. Radiochem.*, **1**, 117 (1959).

[6] W. H. Knoth and E. L. Muetterties, *J. Inorg. Nucl. Chem.*, **20**, 66 (1961) reported the preparation of the $B_{10}H_{13}S(CH_3)_2$ anion as well as similar anions in which the attached ligand was of −1 charge type.

[7] Salts of the $B_{10}H_{15}$ anion recently have been prepared and characterized. Such salts are obtained readily by the addition of one equivalent of strong acid to salts of the $B_{10}H_{14}$ dianion. This latter reaction also is conveniently followed by potentiometric titration techniques. J. A. Dupont and M. F. Hawthorne, *Chem. & Ind.* (London), 405 (1962).

TABLE I

ELEMENTAL ANALYSES OF THE SALTS, $(CH_3)_4\overset{+}{N}\overset{-}{B}_{10}H_{13}X$

X in $(CH_3)_4\overset{+}{N}\overset{-}{B}_{10}H_{13}X$	Calcd. B	Calcd. C	Calcd. H	Calcd. N	Found B	Found C	Found H	Found N
$C_2H_5NH_2$	45.05	29.98	13.32	11.66	44.35	29.48	12.91	9.84
$(C_2H_5)_2NH$	40.34	35.79	13.42	10.44	40.81	34.69	13.58	10.27
$(C_2H_5)_3N$	36.53	40.51	13.50	9.45	37.13	38.89	13.60	6.37
$(C_6H_5)_3P$	23.67	57.74	8.75	3.06	24.06	54.42	8.72	2.54
Pyridine	39.46	39.39	10.94	10.21	36.58	40.90	11.86	10.04
Piperidine	38.48	38.41	13.16	9.96	38.40	38.44	13.75	8.10

of $NaB_{10}H_{13}$ resulted in the slow disappearance of the color and the precipitation of $NaB_{10}H_{13}X$ salts. The relative rates of reaction within this series of ligands is identical to the order in which they are named above with pyridine the most reactive and triethylamine the least reactive. Since the sodium salts prepared in this manner were extremely hygroscopic, they were converted to the corresponding tetramethylammonium salts for characterization purposes. These latter materials were easily purified by recrystallization from ethanol–water. Table I presents these characterization data. Each of the salts indicated in Table I exhibited B–H stretching bands at 4.0, 4.2, and 4.3 μ in the infrared as Nujol mulls.

Structural Considerations.—A poorly resolved B^{11} n.m.r. spectrum of $(C_2H_5)_2NH_2^+$ $[B_{10}H_{13}NH(C_2H_5)_2]^-$ is presented in Fig. 1. Comparison of this spectrum to those recently reported by Pace, Williams, and Williams[8] for a series of $B_{10}H_{12}X_2$ compounds indicates a gross similarity since a sharp high-field doublet is present in both cases.

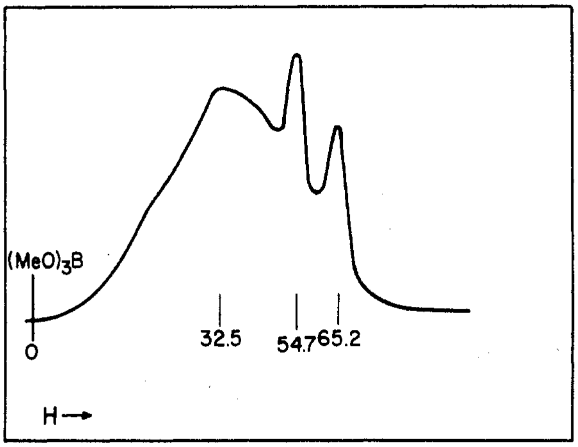

Fig. 1.—B^{11} n.m.r. spectrum of $(C_2H_5)_2NH_2{}^+B_{10}H_{13}NH(C_2H_5)_2{}^-$ in dimethylformamide. Peaks reported as δ with respect to trimethyl borate at 12.8 Mc.

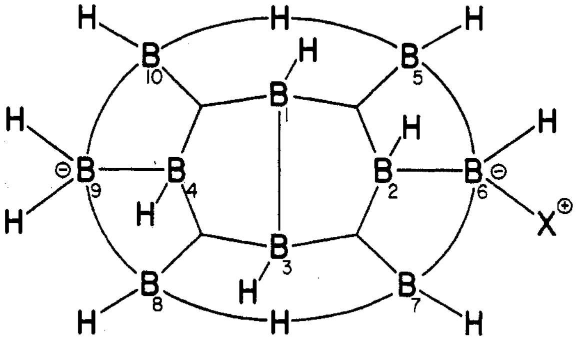

Fig. 2.—Topological representation of the structure of $B_{10}H_{13}X$ ions.

This high-field doublet was identified as the 2- and 4-positions in $B_{10}H_{12}X_2$ compounds. When 1,2,3,4-tetradeuteriodecaborane[9] was converted to $(C_2H_5)_2NH_2{}^+[B_{10}H_9D_4NH(C_2H_5)_2]^-$, this high-field doublet collapsed to a singlet, thus indicating the correctness of this assignment. In view of these data and direct chemical evidence (*vide infra*) it appears that $B_{10}H_{13}X$ anions are structurally identical to $B_{10}H_{12}X_2$ compounds,[10] except that one ligand of the latter is replaced by a hydride ion. This structure is shown in topological form in Fig. 2. Chemical evidence for the presence of a BH_2 group in $B_{10}H_{13}X$ anions is presented below.

Protolysis of $B_{14}H_{13}X$ Anions.—Early in the course of this work it was observed that the thermal decomposition of I in tetrahydrofuran solvent led to the formation of hydrogen and a compound, II, which decomposed at 203°. This same compound was isolated in low yield from the reaction of 6,9-bis-(diethyl sulfide)-decaborane and diethylamine;the principal product being the diethylammonium salt of the $B_{10}H_{10}$ dianion.[11,12] This

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(8) R. J. Pace, J. Williams, and R. L. Williams, *J. Chem. Soc.*, 2196 (1961).

(9) This specifically labeled decaborane was prepared as described by J. A. Dupont and M. F. Hawthorne, *J. Am. Chem. Soc.*, **84**, 1804 (1962).

(10) J. Reddy and W. N. Lipscomb, *J. Chem. Phys.*, **31**, 610 (1959).

(11) M. F. Hawthorne and A. R. Pitochelli, *J. Am. Chem. Soc.*, **81**, 5519 (1959).

(12) W. N. Lipscomb, A. R. Pitochelli, and M. F. Hawthorne, *ibid.*, **81**, 5833 (1959).

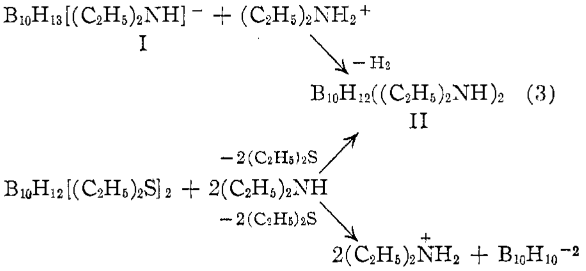
TABLE II

RESULTS OF THE PREPARATION OF $B_{10}H_{12}XX'$ COMPOUNDS

Reactants		Product—$B_{10}H_{12}XX'$									
				Calcd.				Found			
X in $\mathrm{Na^+\overset{-}{B_{10}H_{13}X}}$	Amine hydrochloride (X' in product)	Yield, %	M.p. or dec. pt., °C.	B	C	H	N	B	C	H	N
$(C_2H_5)_2NH$	Pyridine	81	200	39.75	39.68	10.29	10.29	39.00	41.11	11.23	11.07
$(C_2H_5)_2NH$	$(CH_3)_3N$	77	182	42.90	33.31	12.69	11.10	43.06	33.38	12.89	10.74
$(C_2H_5)_2NH$	$C_2H_5NH_2$	35	172	45.42	30.32	12.59	11.75	45.56	28.53	12.31	10.07
$(C_2H_5)_2NH$	$(C_2H_5)_2NH$	42	203	40.95	36.34	12.11	10.60	40.96	36.32	13.91	10.48
Pyridine	Pyridine[a]	65	247	38.89	43.13	7.91	10.06	38.19	44.10	7.96	9.68

[a] Previously reported by B. M. Graybill and M. F. Hawthorne, *J. Am. Chem. Soc.*, **83**, 2673 (1961).

latter mode of synthesis of II and its elemental analysis indicates this material to be 6,9-bis-(diethylamine)-decaborane. Thus the production of this material from I must involve the protolysis of the 6(9)-BH_2 group in I by the diethylammonium ion. These interconversions are shown in (3).



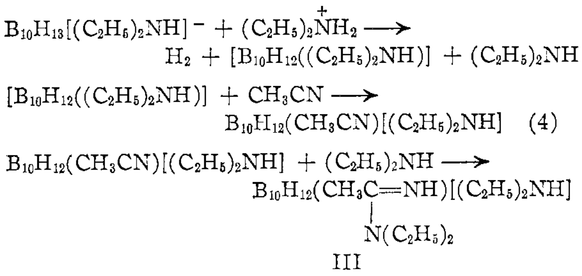
$$\begin{tikzcd} B_{10}H_{13}[(C_2H_5)_2NH]^- + (C_2H_5)_2NH_2^+ \arrow[dr, "-H_2"] & \\ & B_{10}H_{12}((C_2H_5)_2NH)_2 \\ B_{10}H_{12}[(C_2H_5)_2S]_2 + 2(C_2H_5)_2NH \arrow[ur, "-2(C_2H_5)_2S"] \arrow[dr, "-2(C_2H_5)_2S"'] & \\ & 2(C_2H_5)_2\overset{+}{N}H_2 + B_{10}H_{10}^{-2} \end{tikzcd}$$

I

II

(3)

Substitution of acetonitrile for tetrahydrofuran as the reaction medium resulted in another compound, III, whose analysis was in accord with the formulation $B_{10}H_{12}\cdot CH_3CN\cdot 2(C_2H_5)_2NH$. In the infrared, III exhibited NH stretching bands at 2.90 and 3.10 μ and a strong band at 6.20 μ which is characteristic of C=N stretching. A great similarity exists between III and the principal product obtained from the reaction of diethylamine and 6,9-bis-(acetonitrile)-decaborane.[11] On this basis III is formulated as in (4). The assumed intermediate, $B_{10}H_{12}(C_2H_5NH)$, formed by protolysis of I, is topologically equivalent[5] to the well known $B_{10}H_{13}$ anion. The high concentration of acetonitrile present in the reaction mixture apparently allowed this intermediate to capture acetonitrile in preference to the more basic ligand, diethylamine. Subsequent addition of diethylamine to the triple bond of the acetonitrile ligand led to the observed product.



$$B_{10}H_{13}[(C_2H_5)_2NH]^- + (C_2H_5)_2\overset{+}{N}H_2 \longrightarrow H_2 + [B_{10}H_{12}((C_2H_5)_2NH)] + (C_2H_5)_2NH$$

$$[B_{10}H_{12}((C_2H_5)_2NH)] + CH_3CN \longrightarrow B_{10}H_{12}(CH_3CN)[(C_2H_5)_2NH] \quad (4)$$

$$B_{10}H_{12}(CH_3CN)[(C_2H_5)_2NH] + (C_2H_5)_2NH \longrightarrow B_{10}H_{12}(CH_3\underset{\displaystyle N(C_2H_5)_2}{\underset{|}{C}}{=}NH)[(C_2H_5)_2NH]$$

III

A series of reactions analogous to those described above was carried out in tetrahydrofuran solution with the sodium salts of $B_{10}H_{13}X$ anions and amine hydrochlorides. The ligand X was diethylamine or pyridine and the amine hydrochlorides were derived from pyridine, trimethylamine, diethylamine, and ethylamine. The reactions studied took the course shown in (5) and produced *unsymmetrical* members of the $B_{10}H_{12}X_2$ family. Table II presents the yield and analytical data obtained. The velocities of these protolysis reactions were dependent upon the acidity of the amine hydrochloride employed. The qualitative reactivity order observed was: pyridine > trimethylamine > diethylamine > ethylamine.

$$B_{10}H_{13}X^- + R_3NH^+ \longrightarrow H_2 + B_{10}H_{12}(X)(R_3N) \quad (5)$$

Two unsymmetrical $B_{10}H_{12}X_2$ compounds were prepared in which one of the attached ligands was acetonitrile or diethyl sulfide. Thus. when I was treated with dry hydrogen chloride in acetonitrile solvent hydrogen was smoothly evolved and the compound $B_{10}H_{12}[(C_2H_5)_2NH](CH_3CN)$ resulted. A similar reaction carried out in the presence of diethyl sulfide afforded the compound $B_{10}H_{12}[(C_2H_5)_2NH]((C_2H_5)_2S)$. Both of these new materials were characterized by elemental analyses and by their conversion to the triethylammonium salt of the $B_{10}H_{10}^{-2}$ ion on treatment with excess triethylamine.[12] Equation 6 represents this preparative reaction.

$$B_{10}H_{13}[(C_2H_5)_2NH]^- + HCl + \ddot{S} \longrightarrow B_{10}H_{12}[(C_2H_5)_2NH](S) + H_2 + Cl^- \quad (6)$$

S = acetonitrile or diethyl sulfide

Treatment of $B_{10}H_{13}X$ anions with *aqueous* acid solutions resulted in yet another reaction. Under these conditions nonaborane derivatives,[13]

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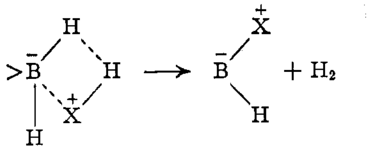
[13] M. F. Hawthorne, B. M. Graybill, and A. R. Pitochelli, Abstracts of Papers, 138th National Meeting of the American Chemical Society, New York, N. Y., p. 45-N.

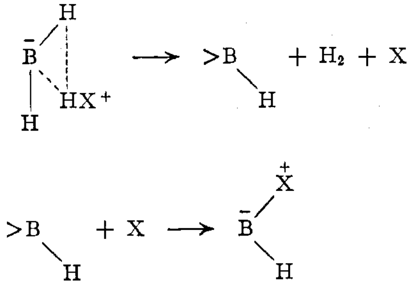
$B_9H_{13}X$, are produced as shown in (7).

$$B_{10}H_{13}X^- + H_3O^+ + 2H_2O \longrightarrow 2H_2 + B(OH)_3 + B_9H_{13}X \quad (7)$$

This particular sequence of reactions will be described in a subsequent paper and in greater detail. However, it is clear that this reaction is closely related to the reactions carried out with anhydrous hydrogen chloride in the presence of acetonitrile or diethyl sulfide. In aqueous media a water molecule apparently plays the role of the attacking ligand. Subsequent degradation of the initial product then leads to the observed products described in (7).

No conclusion can be reached at the present time with regard to the precise mechanism of these protolysis reactions. Two alternatives exist. The conjugate acid of the entering ligand attacks the 6(9)-BH_2 group (which bears a formal charge of -1) in a four-center transition state as in (8) or a three-center transition state[14] is involved as in (9). In the latter situation the discrete intermediate, $B_{10}H_{12}X$, is postulated. It is important to note that Knoth and Muetterties[6] have prepared such a material, $B_{10}H_{12}S(CH_3)_2$.


(8)


(9)

In either event the stereochemistry observed in $B_{10}H_{12}X_2$[15] compounds with respect to ligand attachment results.

Experimental

Materials.—Decaborane was sublimed before use. All amines and amine hydrochlorides were Eastman White Label grade and were used without further purification. Sodium hydride was obtained from the Metal Hydrides Co. Triphenylphosphine was purchased from the Aldrich Chemical Co. Tetrahydrofuran and benzene were distilled from lithium aluminum hydride before use. Acetonitrile was purified by distillation from phosphorus pentoxide. Dioxane was purified by distillation from sodium.

B^{11} N.m.r.—The B^{11} nuclear magnetic resonance spectra were obtained in dimethylformamide solution with a Varian Model V 4300 B high resolution n.m.r. spectrometer at a frequency of 12.8 Mc.

Infrared Spectra.—The infrared spectra of the various compounds were obtained in a Nujol mull on a Model 137 Perkin-Elmer Infracord spectrophotometer.

Preparation of $(C_2H_5)_2NH_2{}^+B_{10}H_{13}(C_2H_5)_2NH^-$ (I).—A solution of 2.0 g. (0.016 mole) of pure decaborane was dissolved in 30 ml. of dry cyclohexane and a chilled solution of 17 ml. of diethylamine in 25 ml. of dry cyclohexane was added. A yellow semisolid precipitated immediately. On standing overnight the precipitated solid crystallized and became white. The solid was recrystallized from water to give 4.0 g. (93% of theory) of white needles, m.p. 204°.

Anal. Calcd. for $B_{10}C_8H_{30}N_2$: B, 40.30; C, 35.82; H, 13.43; N, 10.45. Found: B, 40.16; C, 35.04; H, 13.34; N, 9.91.

Potentiometric Titration of I.—The salt (I) was titrated potentiometrically in the usual manner. A known weight of recrystallized salt was dissolved in purified dioxane. The solutions were titrated, respectively, with standard perchloric acid in anhydrous acetic acid and hydrochloric acid in dioxane. In the latter case diethylammonium chloride precipitated and was identified by its infrared spectrum. An equivalent weight of 264 was obtained (theory 268).

General Preparation of $NaB_{10}H_{13}X$ Salts.—A solution of 2.0 g. (1.6×10^{-2} mole) of decaborane in 20 ml. of diethyl ether was added slowly under nitrogen and with stirring to a suspension of 1.5 g. of sodium hydride in 15 ml. of ether. Hydrogen was evolved and a yellow solution developed. After the addition of decaborane, the solution was stirred for 1 hr. and filtered from the excess sodium hydride.

To the yellow filtrate was added 4×10^{-2} mole of the desired ligand. The solution was stirred under nitrogen for several hours. During this period the product precipitated. The sodium salts were isolated by filtration and converted to the corresponding tetramethylammonium salts in the following fashion: The crude sodium salts were dissolved in a small volume of water and a 10% aqueous solution of tetramethylammonium chloride was added until precipitation ceased. The crude tetramethylammonium salts were purified by recrystallization from ethanol–water. Table I reports the pertinent analytical data. Yields of crude products were essentially quantitative.

Autoprotolysis of I in Tetrahydrofuran.—In a 50-ml. round bottom flask equipped with a magnetic stirrer and reflux condenser was placed 2.0 g. (7.5×10^{-3} mole) of I in 30 ml. of tetrahydrofuran. The solution was heated at the reflux temperature for 4 hr. under nitrogen. Hydrogen was evolved during the reaction. The solution was filtered and the filtrate evaporated to dryness to yield 1.2 g. (60% of theory) of product (II). Recrystallization was accomplished from methylene chloride–pentane. The melting point of the product was 203°.

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[14] M. F. Hawthorne and E. S. Lewis, *J. Am. Chem. Soc.*, **80**, 4296 (1958).

[15] J. van der Maas Reddy and W. N. Lipscomb, *J. Chem. Phys.*, **31**, 610 (1959).

Anal. Calcd. for $B_{10}C_8H_{34}N_2$: B, 40.95; C, 36.34; H, 12.11; N, 10.60. Found: B, 40.96; C, 36.32; H, 13.91; N, 10.48.

Autoprotolysis of I in Acetonitrile.—A solution of 2.0 g. (7.5×10^{-3} mole) of I in 30 ml. of acetonitrile was heated to the reflux temperature and stirred under nitrogen for 4 hr. or until no more hydrogen was evolved. The solution was filtered and the filtrate was evaporated *in vacuo* to yield 1.5 g. (49% of theory) of solid. White crystals, m.p. 175°, were obtained from methylene chloride–pentane. The infrared spectrum contained NH bands at 2.9 μ and 3.1 μ and C═N absorption at 6.2 μ.

Anal. Calcd. for $B_{10}C_{10}H_{37}N_3$: B, 35.22; C, 39.06; H, 12.04; N, 13.67. Found: B, 36.20; C, 38.63; H, 11.90; N, 13.72.

Reaction of Amine Hydrochlorides and $\overset{+}{N}aB_{10}H_{13}X$.—The procedure was essentially the same for all compounds described in Table II.

In a 50-ml. round bottom flask were placed 5×10^{-3} mole of $\overset{+}{N}a\overset{-}{B}_{10}H_{13}X$, 20 ml. of tetrahydrofuran, and 7×10^{-3} mole of the desired amine hydrochloride. The solution was stirred and heated to the reflux temperature for 4 hr. while hydrogen was evolved. After filtering the solution, the filtrate was evaporated to dryness. The solid which was separated by filtration was washed well with water to remove starting material and then combined with the filtrate residue and recrystallized from methylene chloride–pentane. The yields of products are listed in Table II.

Protolysis of I in Acetonitrile Solution.—A 300-ml. round bottom flask containing 3.0 g. (1.1×10^{-2} mole) of I and 50 ml. of acetonitrile was evacuated on a vacuum line. Approximately one atmosphere of dry hydrogen chloride gas was introduced. Hydrogen was evolved as the reaction mixture was stirred. After 20 min., the contents of the flask was evaporated to dryness *in vacuo* and then extracted with methylene chloride. The methylene chloride-insoluble material, after being washed with water, yielded 2.0 g. (76% of theory) of white product, dec. pt. 171°. Recrystallization from acetonitrile gave light yellow crystals of $B_{10}H_{12}[(C_2H_5)_2NH](CH_3CN)$.

Anal. Calcd. for $B_{10}C_6H_{26}N_2$: B, 46.20; C, 30.74; H, 11.10; N, 11.96. Found: B, 45.66; C, 29.90; H, 11.46; N, 12.46.

Protolysis of I in the Presence of Diethyl Sulfide.—A suspension of 2.0 g. (7.5×10^{-3} mole) of I in 30 ml. of diethyl sulfide was placed in a 200-ml. round bottom flask and evacuated on a vacuum line. One atmosphere of hydrogen chloride was admitted and the reaction was allowed to proceed until 7.5×10^{-3} mole of hydrogen was evolved as measured in an attached Toepler system. The solution was evaporated *in vacuo* and the residue washed with water and dried. The yield of crude product was 1.6 g. (77% of theory). White crystals of $B_{10}H_{12}[(C_2H_5)_2NH][(C_2H_5)_2S]$ which melted at 124° were obtained from methylene chloride–pentane.

Anal. Calcd. for $B_{10}C_8H_{33}NS$: B, 38.21; C, 33.90; H, 11.65; N, 4.94. Found: B, 37.44; C, 33.92; H, 11.55; N, 4.88.

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CONTRIBUTION FROM THE ROHM AND HAAS COMPANY, REDSTONE ARSENAL
RESEARCH DIVISION, HUNTSVILLE, ALABAMA

The Preparation and Reactions of B_9H_{13} (Ligand) Compounds[1a]

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Synthetic methods for the preparation of $B_9H_{13}X$ (X = ligand) species are described. Further reactions of $B_9H_{13}X$ compounds include (1) ligand displacement reactions, (2) proton abstraction to form $B_9H_{12}X$ anions, and (3) proton abstraction accompanied by ligand expulsion to produce the B_9H_{12} anion. Spectroscopic examination of a series of $B_9H_{13}(pyZ)$ (Z = substituent group on pyridine) revealed a correlation of excitation energy with the Hammett sigma constant of Z.

The synthesis[2] and structure[3] of an enneaborane-15 have been reported recently. The topological method of Dickerson and Lipscomb[4, 5] allows the determined structure to be described as an icosahedral fragment of 5421 topology.

Previous workers[6, 7] in the area of boron hydride

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(1) (a) Presented in part at the 138th National Meeting of the American Chemical Society, New York, N. Y., September, 1960, Abstracts, p. 45-N; (b) Department of Chemistry, The University of California, Riverside, California.

(2) W. V. Kotlensky and R. Schaeffer, *J. Am. Chem. Soc.*, **80**, 4517 (1958).

(3) R. E. Dickerson, P. J. Wheatley, P. A. Howell, and W. N. Lipscomb, *J. Chem. Phys.*, **27**, 200 (1957).

(4) R. E. Dickerson and W. N. Lipscomb, *ibid.*, **27**, 212 (1957).

(5) W. N. Lipscomb, *Advan. Inorg. Chem. Radiochem.*, **1**, 117 (1959).

(6) R. Schaeffer, private communication, 1956; methanolysis of 6,9-bis-(acetonitrile)-decaborane apparently produced a B_9 derivative.

(7) S. J. Fitch and A. W. Laubengayer, *J. Am. Chem. Soc.*, **80**, 5911 (1958).