nectedness of the polyhedra, number of formal bonds, and extent of d-orbital participation. The evidence presented here, however, is not sufficient to remove these considerations from the purely speculative classification.

Whatever the structure of the aggregate species, the results are in agreement with Wartik's molecular weight observation for dimethylaluminum hydride.[33] The principal species in the gas phase is most probably the dimer. Trimers, however, are possible and exist in the vapor phase and most certainly in solution. The tetramers and hexamers would not be expected with unrearranged dimethylaluminum hydride because there would be insufficient hydrogen bridge bonds. They are observed when the electron impact causes rearrangements producing an average of more than one hydride per aluminum. They should also be observed if methylaluminum dihydride were to be prepared. So far this compound has not been characterized. The results presented not only suggest a possible approach to the synthesis of the dihydride but also suggest that the structural studies of the dihydride may be quite interesting.

An interesting generalization that we have observed is that most of the ion species obey the rule that the number of substituents is $3n - 1$, where n is the number of aluminum atoms in the species. This rule holds true for rearranged species as well as for species which can be formed without rearrangement. Calculations on the basis of this rule are very valuable in identifying the ion species, in particular those of the polymeric species. These identifications confirm the observation that the C–Al bond is more easily broken than the C–H bond of the methyl group attached to the central aluminum atom. This observation is in accord with the results reported for the group IVa metal alkyls.[37-43] The rule is also an indication that ions of the type $Al_nA_xB_y$, where $x + y = 3n - 1$, are ions which have a high probability of being observed in the mass spectra.

The cause for the high hydrocarbon peaks on first introducing the sample is most probably hydrolysis. The hydrocarbons found in studying other organometallic compounds substituted with ethyl and higher alkyls indicate that the hydrocarbon is an alkane rather than an alkene. The former is a hydrolysis product whereas the latter is formed by thermal decompositions. The hydrolysis probably takes place on the walls of the sample introduction system even though the sample system is maintained at a high vacuum. Partially hydrolyzed material might also exist in the sample itself. The sample is carefully fractionated in a vacuum system, but hydrolysis could conceivably take place upon transfer to the sample container.

Acknowledgment.—We wish to acknowledge helpful discussions with Dr. Robert Bohn and the assistance of Mr. Oliver W. Norton in obtaining the mass spectra. Assistance of the University of Connecticut Research Foundation is gratefully acknowledged.

(37) V. H. Dibeler and F. L. Mohler, *J. Res. Natl. Bur. Std.*, **47**, 337 (1951).
(38) V. H. Dibeler, *ibid.*, **49**, 235 (1952).
(39) V. H. Dibeler, *Anal. Chem.*, **27**, 1958 (1955).
(40) R. A. Khmel'nitskii, A. A. Polyakova, A. A. Petrov, F. A. Medvedev, and M. D. Stadnichuk, *Zh. Obshch. Khim.*, **35**, 773 (1965).
(41) E. Heldt, K. Hoppner, and K. H. Krebs, *Z. Anorg. Allgem. Chem.*, **347**, 95 (1966).
(42) G. P. van der Kelen, O. Volders, H. vanOnckelen, and Z. Eeckhaut, *ibid.*, **338**, 106 (1965).
(43) D. B. Chambers, F. Glockling, and M. Weston, *J. Chem. Soc.*, *A*, 1759 (1967).

Contribution from the Evans Chemical Laboratory,
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Preparation of Trialuminum Triboron Heptakis(dimethylamino)pentahydride[1a]

By R. E. Hall[1b] and E. P. Schram[1c]

Received October 18, 1968

Treatment of bis(trimethylamine)-alane, $[(CH_3)_3N]_2AlH_3$, with tetrakis(dimethylamino)diborane(4), $[(CH_3)_2N]_4B_2$, results in the formation of the following known compounds: H_2, $(CH_3)_3N$, $[(CH_3)_2NBH_2]_2$, $[(CH_3)_2N]_2BH$, and $(CH_3)_3NBH_3$. In addition, a new species was prepared, $Al_3B_3[N(CH_3)_2]_7H_5$, which most likely contains three B–Al bonds. Characterization of this new compound is discussed.

Introduction

As part of a continuing effort to prepare metal–metal or metal–metalloid bonds,[2,3] the reduction of bis(trimethylamine)-alane, $[(CH_3)_3N]_2AlH_3$, by tetrakis(dimethylamino)diborane(4), $B_2[N(CH_3)_2]_4$, was investigated.

Experimental Section

Apparatus and Procedures.—All reactions were carried out in a standard vacuum line equipped with mercury float valves and stopcocks lubricated with Apiezon N grease. Experimental equipment was attached to the vacuum line with Viton A O-ring

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[1] (a) R. E. Hall and E. P. Schram, Abstracts, 155th National Meeting of the American Chemical Society, San Francisco, Calif., March 1968, No. M211. (b) Work completed in partial fulfillment of the M.S. degree, 1967. (c) To whom correspondence should be addressed.
(2) E. P. Schram, *Inorg. Chem.*, **5**, 1291 (1966).
(3) G. S. Kyker and E. P. Schram, *J. Am. Chem. Soc.*, **90**, 3672 (1968).

closure assemblies which were previously evacuated to 10^{-6} Torr. Transfer of nonvolatile materials was carried out in a metal drybox continually flushed with General Dynamics "Hi-Pure" nitrogen previously dried by passage over Linde 4A Molecular Sieves and phosphorous pentoxide. Solid samples were filtered, *in vacuo*, employing a medium-porosity sintered-glass frit. The filtration apparatus was equipped with O-ring joint closures and Fisher–Porter Teflon stopcocks. Analyses for aluminum, nitrogen, and boron were by 8-hydroxyquinoline precipitation, and Kjeldahl and standard mannitol titration, respectively. Hydridic hydrogen was determined by conversion to H_2 through treatment with H_2O; collection and measurement was effected by a calibrated-volume Toepler pump assembly and characterized by combustion over CuO to H_2O. D_2 and HD were characterized by mass spectrometry utilizing an Associated Electrical Industries Type 10 spectrometer. Infrared spectra were recorded with a Perkin-Elmer Model 337 grating spectrophotometer. Volatile materials were contained in a 10-cm gas cell equipped with KBr optics. Spectra of nonvolatile materials were obtained as Nujol (dried over Na) and Fluorolube (dried over molecular sieves) mulls between KBr plates. ^1H and ^{11}B nmr spectra were obtained with Varian Associates instruments A-60 (60 Mc) and HA-100 (32.1 Mc), respectively.

Reagents.—**Tetrakis(dimethylamino)diborane(4)**, obtained from U. S. Borax Research Corp., Anaheim, Calif., was distilled, *in vacuo*, utilizing a Nester–Faust spinning-band column. The boiling point of the colorless liquid was found to be 69° (3.5 Torr); lit.[4] value, 69° (3.6 Torr). **Trimethylamine**, obtained from the Matheson Co., Inc., Joliet, Ill., was freed of $HN(CH_3)_2$ by treatment with P_2O_5 at 0° for several days and purified by distillation through a trap maintained at −78° into a −196° trap. Trimethylamine, retained in the −196° trap, exhibited a −78° vapor tension of 6.5 Torr; lit.[5] value, 6.6 Torr. **Hydrogen chloride**, obtained from the Matheson Co., was distilled through a −126° trap to a −196° trap. The HCl collected in the −196° trap exhibited a vapor tension of 119 Torr at −111.6°; lit.[6] value, 120 Torr. **Lithium aluminum hydride**, obtained from Alfa Inorganics Inc., Beverly, Mass., was used without further purification. **Diethyl ether**, analytical reagent, obtained from Mallinckrodt Chemical Works, was distilled from calcium hydride and used without further purification. **Methylcyclohexane**, obtained from Matheson Coleman and Bell, East Rutherford, N. J., was dried over calcium hydride and distilled through a trap maintained at −45° into a −196° trap. The methylcyclohexane, retained at −196°, exhibited a vapor tension of 12 Torr at 0°; lit.[7] value, 12 Torr. **Benzene**, analytical reagent, obtained from Mallinckrodt Chemical Works, was dried over calcium hydride and distilled through a trap maintained at −30.6° into one maintained at −78° until its 0° vapor tension was 25 Torr; lit.[8] value, 25 Torr. **Ethylene**, obtained from the Matheson Co., Inc., Joliet, Ill., was purified by distillation through a trap maintained at −126°. The ethylene, collected at −196°, exhibited a vapor tension of 456 Torr at −111.6°; lit.[9] value, 462 Torr. D_2O, 99.75 atom % purity, obtained from Isotopes, Inc., Westwood, N. J., was used as received. **DCl** was prepared by treating D_2O with excess BCl_3 in the vacuum line. **Bis(trimethylamine)-alane**,[10] **bis(dimethylamino)borane**,[11] and **tris(dimethylamino)aluminum**[12] were prepared by the literature methods. **Dimethylaminodimethylaluminum** was prepared by treating $B_2[N(CH_3)_2]_4$ with $Al(CH_3)_3$.[2] **Dimethylaminodifluoroboron**, $(CH_3)_2NBF_2$, was prepared by treating $[(CH_3)_2N]_2BH$ with BF_3. Characterization was possible from the melting point (159°; lit.[13] 155°) and the ^1H nmr spectrum.

Reaction of Bis(trimethylamine)-Alane with Tetrakis(dimethylamino)diborane(4).—In a typical reaction, tetrakis(dimethylamino)diborane(4), $[(CH_3)_2N]_4B_2$ (12.9 mmol), was syringed into a 15-ml flask and the flask was attached to the vacuum line. First, methylcyclohexane (3 ml) and then bis(trimethylamine)-alane, $[(CH_3)_3N]_2AlH_3$ (*ca.* 10 mmol), were condensed into the reaction flask, and the reaction mixture was magnetically stirred at room temperature for 10 hr. Initially the reaction mixture bubbled vigorously and a white solid (I) precipitated from the solution. The volatile reaction products were separated by trap to trap distillations, characterized by vapor tension measurements, mass spectrometry, and infrared analysis, and found to consist of H_2, $(CH_3)_3N$, $[(CH_3)_2NBH_2]_2$, $[(CH_3)_2N]_2BH$, and $(CH_3)_3NBH_3$.

Purification and Analytical Characterization of I.—Compound I was repeatedly treated with methylcyclohexane in a vacuum filtration apparatus and unreacted $B_2[N(CH_3)_2]_4$ and/or $[(CH_3)_3N]_2AlH_3$ was extracted from I. Next, I was dissolved in benzene, filtered *in vacuo*, and recrystallized from benzene upon addition of methylcyclohexane. This needlelike, white, crystalline material discolors in the temperature range 150–155° and slowly evolves $[(CH_3)_2N]_2BH$ and $[(CH_3)_2NBH_2]_2$. Above 250° H_2 and CH_4 are also produced.

Oxidative hydrolysis was effected by treating I (175.1 mg) with 20% aqueous HCl for 48 hr at 25°. H_2 (3.17 mmol) was evolved; the residue contained Al (32.6 mg), B (13.2 mg), and N (40.3 mg). The molecular weight of I was determined isopiestically by allowing benzene (164.9 mmol) to equilibrate between azobenzene (0.176 mmole) and I (68.0 mg). A molecular weight of 433 was found.

Deuteriolysis of I.—Compound I (73.0 mg) was treated with 2 ml of D_2O–20% DCl, *in vacuo*, for 48 hr at 25°. D_2 (0.477 mmol) and HD (0.865 mmol) were evolved.

Air Oxidation.—Exposure of I to the atmosphere resulted in spontaneous combustion of the sample with an accompanying green flame.

Treatment of I with Ethylene.—A benzene solution of I was exposed to ethylene vapor (637 Torr) for 48 hr at 25° followed by heating the reaction mixture for 8 hr at 75°. Neither of these reaction conditions resulted in the loss of ethylene.

Results and Discussion

Treatment of bis(trimethylamine)-alane, $[(CH_3)_3N]_2AlH_3$, with tetrakis(dimethylamino)diborane(4), $[(CH_3)_2N]_4B_2$, in the reaction ratios 1:3 to 3:1, results in the formation of H_2, $(CH_3)_3N$, $[(CH_3)_2NBH_2]_2$, $[(CH_3)_2N]_2BH$, and $(CH_3)_3NBH_3$. In addition, a nonvolatile white crystalline material is produced with the molecular formulation $Al_3B_3[N(CH_3)_2]_7H_5$. *Anal.* Calcd for $Al_3B_3[N(CH_3)_2]_7H_5$: Al, 19.0; B, 7.6; N, 23.0; mol wt, 426.4. Found: Al, 18.6; B, 7.5; N, 23.1; mol wt, 433. Neutral hydrolysis of $Al_3B_3[N(CH_3)_2]_7H_5$ is incomplete as evidenced by the fact that the residue reduces Ag^+. However, treatment with H_2O–HCl affords a total of 7.9 mmol of H_2; D_2O–DCl affords 5.13 mmol of HD and 2.79 mmol of D_2 ($HD/D_2 = 1.84$). The sources of HD and D_2 are shown by reactions 1 and 2, respectively, where

$$MH + D_2O \longrightarrow MOD + HD \qquad (1)$$

$$\begin{aligned} MM + D_2O &\longrightarrow MOD + MD \\ MD + D_2O &\longrightarrow MOD + D_2 \end{aligned} \qquad (2)$$

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[4] R. Brotherton, A. McCloskey, L. Petterson, and H. Steinberg, *J. Am. Chem. Soc.*, **82**, 6242 (1960).

[5] R. T. Sanderson, "Vacuum Manipulation of Volatile Compounds," John Wiley & Sons, Inc., New York, N. Y., 1948, p 139.

[6] See ref 5, p 143.

[7] See ref 5, p 128.

[8] See ref 5, p 127.

[9] See ref 5, p 126.

[10] G. W. Fraser, N. N. Greenwood, and B. P. Straughan, *J. Chem. Soc.*, 3742 (1963).

[11] H. Noth, W. A. Dorochov, P. Fritz, and F. Pfab, *Z. Anorg. Allgem. Chem.*, **318**, 293 (1962).

[12] J. Ruff, *J. Am. Chem. Soc.*, **83**, 2835 (1961).

[13] A. B. Burg and J. Banus, *ibid.*, **76**, 3903 (1954).

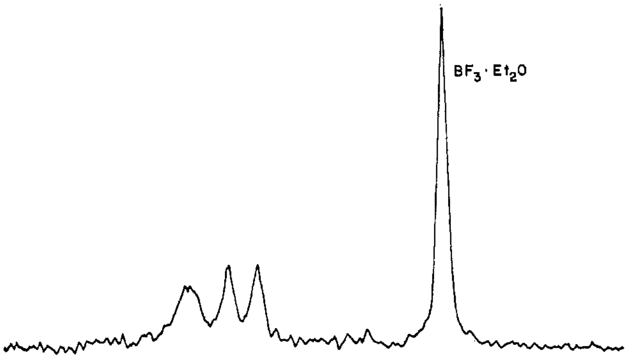
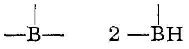


Figure 1.—The ^{11}B nmr spectrum of $Al_3B_3[N(CH_3)_2]_7H_5$.

M = B or Al. Based on the hydrolysis data, the ratios of hydridic hydrogen to number of metal–metal or metalloid–metal bonds are: 4(H–M)/4(M–M) = 1(H–D)/1(D–D), 5(H–M)/3(M–M) = 1.7, or 3(H–M)/5(M–M) = 0.6. The experimental result, (H–M)/(M–M) = 1.84, is in good agreement with a $^5/_3$ ratio. The deuteriolysis reaction is described by

$$Al_3B_3[N(CH_3)_2]_7H_5 + 7D^+ + 18D_2O \longrightarrow 5HD + 3D_2 + 7D_2N(CH_3)_2^+ + 3Al(OD)_3 + 3B(OD)_3$$

^{11}B Nmr Data.—The ^{11}B nmr spectrum of $Al_3B_3[N(CH_3)_2]_7H_5$ is shown in Figure 1 and consists of a broad singlet at −35.25 ppm and a doublet centered at −28.45 ppm (from $BF_3 \cdot O(C_2H_5)_2$) in the relative ratio 1:2; J_{BH} = 133 cps. These results indicate two different boron environments



$$\overset{|}{\text{—B—}} \qquad 2\ \text{—}\overset{|}{\text{B}}\text{H}$$

^1H Nmr Data.—The ^1H nmr spectrum of a fresh solution of $Al_3B_3[N(CH_3)_2]_7H_5$ is depicted in Figure 2 and consists of three absorptions in the relative ratio ~12:2:1 at −2.88, −2.78, and −2.47 ppm, respectively, from tetramethylsilane (TMS). The broad high-field absorption is attributed to H–Al, $I = {}^5/_2$, for Al. The expected 1:1:1:1 quartet for H–B is barely discernible in neat $[(CH_3)_2N]_2BH$ [$\delta(^1HB)$ = −2.72, J_{BH} = 130 cps];[14] therefore lack of ^1H absorptions for B–H in $Al_3B_3[N(CH_3)_2]_7H_5$ is not surprising. Absorptions at −2.78 and −2.88 ppm from TMS indicate two different ^1H environments on the nmr time scale, in a 6:1 ratio. This result is in accord with a total of seven $N(CH_3)_2$ groups per mole as indicated by analyses and molecular weight data.

Infrared Data.—The solid-state infrared spectrum of $Al_3B_3[N(CH_3)_2]_7H_5$ is shown in Figure 3 and is summarized in Table I. Infrared assignments are based on correlated data assignments for related compounds, Table I. The spectra of all of these species are shown in Figure 3 to aid in correlations based on band shapes and relative intensities. Concerning $Al_3B_3[N(CH_3)_2]_7H_5$, the band at 2392 cm^{-1} indicates the presence of a single terminal BH moiety.[15] This interpretation is consistent with a doublet and not a triplet in the ^{11}B nmr spectrum. The strong, relatively sharp band

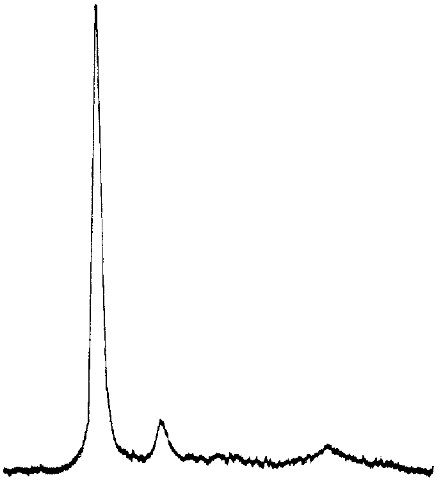


Figure 2.—The ^1H nmr spectrum of $Al_3B_3[N(CH_3)_2]_7H_5$.

TABLE I

INFRARED CORRELATIONS FOR AMINOALANES AND AMINOBORANES

Assignment	$Al_3B_3[N(CH_3)_2]_7H_5$	$\{(CH_3)_2NAl(CH_3)_2\}_2$	$\{[(CH_3)_2N]_3Al\}_2$	$[(CH_3)_2N]_2BH$
ν_{C-H}	2924vs 2762vs 2801vs	2967vs 2857vs 2915vs 2801vs	2941vs 2817vs 2882vs 2763vs	3012m 2890vs 2801s
ν_{B-H}	2481 wsh 2392vs 2343ssh 2273ssh			2457s
ν_{Al-H}	1818vs			
ν_{asy} B-N				1530vs
ν_{sym} B-N				1409vs 1418ssh
δ_{CH_3}	1451vs 1399w 1380w 1277m	1475m 1456m	1473ssh 1376m 1462vs 1441ssh 1279s 1403m	1466ssh 1462s 1362w
ρ_{CH_3}	1234m 1122s 1075w	1258vs 1202vs 1172m 1128s	1235m 1117m 1070m	1239s 1165s
ν_{asy} NC_2 terminal	1164vs		1165vs	1074s
ν_{asy} NC_2 bridging	1046vs	1049s	1043s	
ν_{sym} NC_2 terminal	985vs		980vs	962w
ν_{sym} NC_2 bridging	905vs	966w 918vs	901s	
δ_{B-H}	931m			878m
(?)	813w 625vs	608s	1653w 1608m 813m 608vs	
ν_{asy} Al-C		772s		
terminal $N(CH_3)_2$ wag (?)	769s		722m	
terminal CH_3 wag		714s		
ν_{sym} Al-C		576s		
bridge $N(CH_3)_2$ rock (?)	513vs	509m	524m	
bridge stretch (?)	714vs 467m	476w 453w 463w	670s 428w	
ν_{Al-N} terminal	531vs		546	

s = strong; v = very; m = medium; w = weak; sh = shoulder

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(14) This work.

(15) J. Dawson, P. Fritz, and K. Niedenzu, *J. Organometal Chem.* (Amsterdam), **5**, 13 (1966).

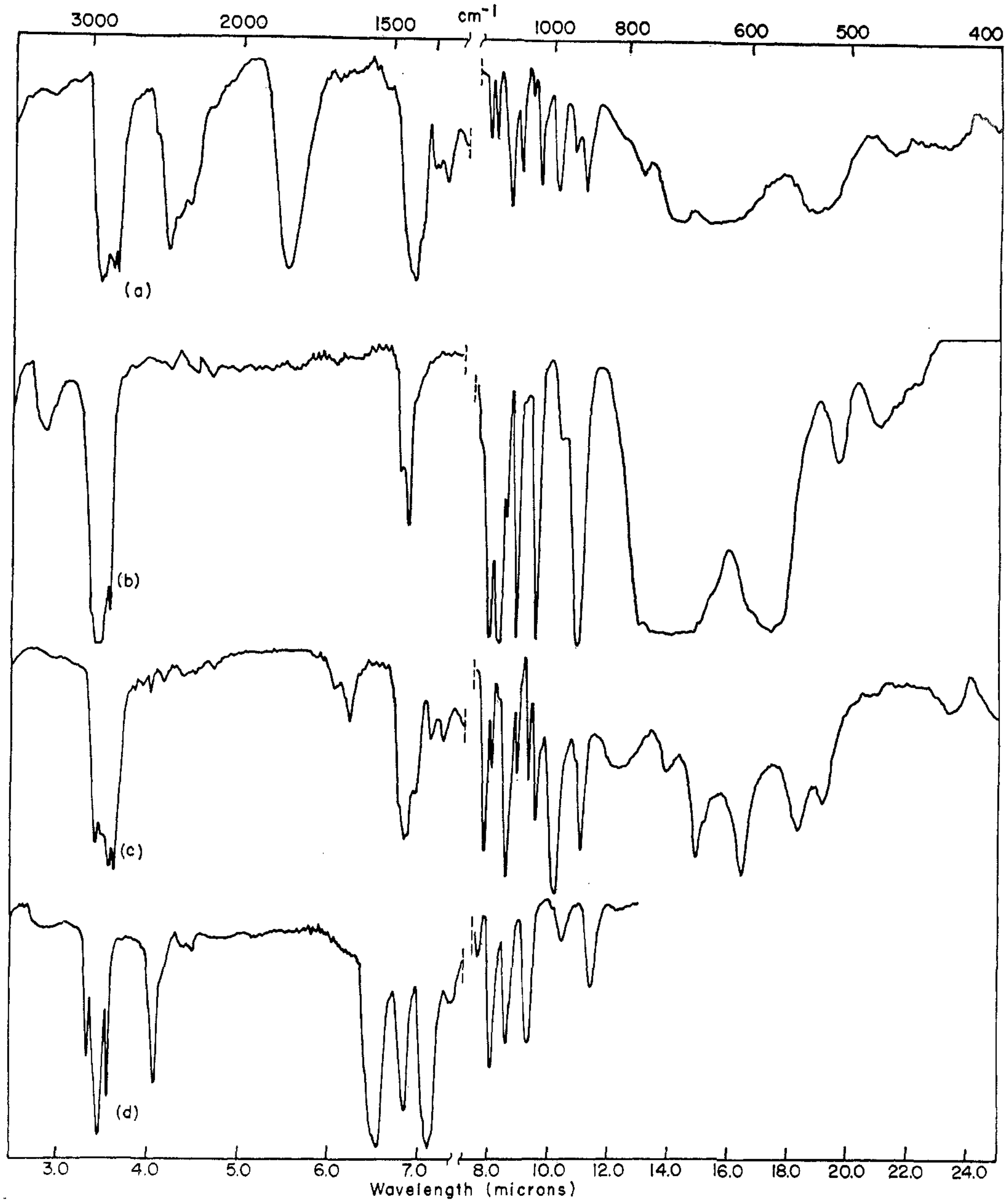

Figure 3.—Infrared spectrum of (a) $Al_3B_3[N(CH_3)_2]_7H_5$, (b) $[(CH_3)_2NAl(CH_3)_2]_2$, (c) $[[(CH_3)_2N]_3Al]_2$, and (d) $[(CH_3)_2N]_2BH$.

centered at 1818 cm^{-1} establishes the presence of terminal AlH moieties.[16] The band centered at 1451 cm^{-1} is found in all of the amino derivatives in Table I and is due to δ_{asym} and δ_{sym} CH_3 modes. The absorptions at 1399 and 1380 cm^{-1} are also observed in the spectrum of dimeric $[(CH_3)_2N]_3Al$, 1403 and 1376 cm^{-1}, and are assigned to $\delta(CH_3)$ modes of terminal $N(CH_3)_2$ moieties. In a similar fashion the absorption found at 1277 cm^{-1} is assigned to $\delta(CH_3)$ associated with the terminal $N(CH_3)_2$ moiety. This deformation is located at 1279 cm^{-1} in dimeric $[(CH_3)_2N]_3Al$. The strong band at 1234 cm^{-1} is assigned to $\rho(CH_3)$ modes associated with $(CH_3)_2NB$. In $[(CH_3)_2N]_2BH$ this absorption is found at 1239 cm^{-1} and in $(CH_3)_2NBF_2$ at 1250 cm^{-1}. The band at 1164 cm^{-1} arises from overlap of $\nu_{asym}(NC_2$ terminal), found at 1165 cm^{-1} in dimeric $[(CH_3)_2N]_3Al$, and $\rho(CH_3)$, found at 1165 cm^{-1} in $[(CH_3)_2N]_2BH$. The strong absorption at 1122 cm^{-1} is assigned to $\rho(CH_3)$ associated with bridging $(CH_3)_2NAl$ which occurs at 1128 cm^{-1} in the $(CH_3)_2NAl(CH_3)_2$ dimer. The band at 1046 cm^{-1} is observed at 1043 cm^{-1} in $[(CH_3)_2N]_3Al$ and at 1049 cm^{-1} in $(CH_3)_2NAl(CH_3)_2$ and is assigned to $\nu_{asym}(NC_2$ bridge). In a similar fashion, the band at 985 cm^{-1} is associated with $\nu_{sym}(NC_2$ terminal) which occurs at 980 cm^{-1} in $[(CH_3)_2N]_3Al$. The absorptions at 931 and 905 cm^{-1} are most likely due to $\delta(BH)$ and ν_{sym} $(NC_2$ bridge), respectively. Bands from 770 to 531 cm^{-1} are assigned to $\nu(AlN)$, both bridge and terminal. Similar bands are found in the spectrum of $[(CH_3)_2$-

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[16] E. G. Hoffman and G. Schomburg, *Z. Elektrochem.*, **61**, 1101 (1957).

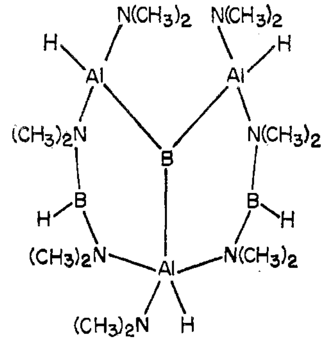


Figure 4.—Suggested structure for $Al_3B_3[N(CH_3)_2]_7H_5$.

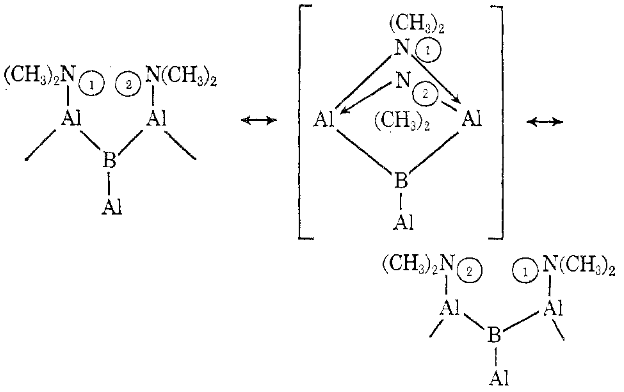
N]$_3$Al. The band at 531 cm^{-1} appears to be due to ν(AlN terminal), found at 546 cm^{-1} in $\{[(CH_3)_2N]_3Al\}_2$, whereas the absorption in dimeric $[(CH_3)_2N]_2Al(CH_3)_2$ is found at 509 cm^{-1}, ν(AlN bridge).

Concerning the structure of $Al_3B_3[N(CH_3)_2]_7H_5$, the following conclusions are based on the infrared data: (1) presence of bridging and terminal $N(CH_3)_2$ moieties bonded to aluminum; (2) presence of $N(CH_3)_2$ groups bonded to boron [the lack of strong ν_{asym} and ν_{sym} BN_2 modes is consistent with coordinated $N(CH_3)_2$ on boron; similar results are found when comparing the spectra of $Cl_2BN(CH_3)_2$ with $[Cl_2BN(CH_3)_2]_2$ and the spectrum of $[H_2BN(CH_3)_2]_2$];[10,17] (3) presence of terminal AlH bonds; (4) presence of terminal BH bonds which is collaborated by the ^{11}B data; (5) absence of AlHB bridging based on the absence of an absorption at *ca.* 2031 cm^{-1} as found in $Al(BH_4)_3$;[18] (6) absence of BHB bridges based on the absence of an absorption at *ca.* 1600 cm^{-1}.[19,20]

Molecular Formulation of $Al_3B_3[N(CH_3)_2]_7H_5$.—The suggested structure for $Al_3B_3[N(CH_3)_2]_7H_5$ is shown in Figure 4.

The oxidative hydrolysis data indicate three metal–metal or metal–metalloid bonds, *i.e.*, three B–Al bonds. This model is consistent with the ^{11}B nmr data; there are two different boron environments in a 2:1 ratio with the more intense signal split into a doublet with J_{BH} very close to that found for $[(CH_3)_2N]_2BH$ (J_{BH} = 126 cps, δ = 28.6 ppm).[21] The $\delta(^{11}B)$ data indicate both types of boron are three-coordinate and these results are consistent with the structure shown in Figure 4. A Catalin[22] molecular model of $Al_3B_3[N(CH_3)_2]_7H_5$ clearly reveals steric interactions by $N(CH_3)_2$ moieties preventing nucleophilic attack, on the boron bonded to aluminum, by other aminoboranes and $N(CH_3)_3$ which are present during the original synthesis.

Infrared data, $\rho(CH_3)$ assignments, indicate the presence of $(CH_3)_2N$ moieties bonded to B. These assignments coupled with the absence of normal ν(BN) absorptions result in the conclusion that the $(CH_3)_2N$ moieties on B are coordinated. Furthermore, infrared data indicate both bridging and terminal $N(CH_3)_2$ groups on Al. The ^{1}H nmr data indicate two different $N(CH_3)_2$ environments in a 6:1 ratio. The less intense of these two absorptions is considered to be associated with the five-coordinate aluminum, Figure 4. The two terminal $N(CH_3)_2$ moieties, associated with the formally four-coordinate Al, are considered equivalent to the bridging $N(CH_3)_2$ groups. This equivalence is envisaged *via* exchange of the type



This exchange results in a 6:1 ratio of bridging *vs.* terminal $N(CH_3)_2$, the unique $N(CH_3)_2$ moiety being associated with the five-coordinate Al.

Proof of the presence of coordinated $[(CH_3)_2N]_2BH$ in $Al_3B_3[N(CH_3)_2]_7H_5$ is most difficult. However, its presence during the reduction of alane is assured because it was isolated as a by-product. Also, heating $Al_3B_3[N(CH_3)_2]_7H_5$ affords $[(CH_3)_2N]_2BH$. In addition, the $\delta(^{11}B)$ and J_{BH} data for $Al_3B_3[N(CH_3)_2]_7H_5$ are consistent with the presence of $[(CH_3)_2N]_2BH$. One might expect $\delta(^{11}B)$ of $[(CH_3)_2N]_2BH$ to change when comparing the noncomplexed species to the bridged material. To this end, we treated $[(CH_3)_2N]_2BH$ with BF_3, but $(CH_3)_2NBF_2$ was formed rather than $[(CH_3)_2N]_2BH \cdot 2BF_3$. Thus far, we have not been able to displace $[(CH_3)_2N]_2BH$ from $Al_3B_3[N(CH_3)_2]_7H_5$.

We are investigating the chemistry of $Al_3B_3[N(CH_3)_2]_7H_5$ and will report the results shortly.

Acknowledgment.—Support of this work by the National Science Foundation and the Petroleum Research Fund, administered by the American Chemical Society, is most appreciated.

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[17] W. C. Price, R. D. B. Fraser, T. S. Robinson, and H. C. Longuet-Higgins, *Discussions Faraday Soc.*, **9**, 131 (1950).

[18] W. C. Price, *J. Chem. Phys.*, **17**, 1044 (1949).

[19] R. C. Lord and E. Nielsen, *ibid.*, **19**, 1 (1951).

[20] D. E. Mann, *ibid.*, **22**, 70 (1954).

[21] H. Noth and H. Vahrenkamp, *Ber.*, **99**, 1049 (1966).

[22] Manufactured by Catalin Limited, Waltham Abbey, Essex, England.