of trimethylamine and 0.33 g. (4.53 mmoles) of trimethylamine borane was obtained from the reaction mixture. The B^{11} n.m.r. of both reaction mixtures consisted solely of a quadruplet.

(B) **Tensiometric Titrations.**—The tensiometric titrations of dimethylamino hydrido aluminum borohydride and dimethylamino aluminum diborohydride with trimethylamine were performed as described previously.[11] A ratio of amine to hydride of 1.04 was found for the former and 1.96 for the latter. Equilibrium was attained in the system shortly after reaching ambient temperature (see Fig. 2).

Attempted Preparation of $Cl_2AlBH_4 \cdot N(CH_3)_3$.—A 5.51-g. (21.8 mmoles) sample of mercuric chloride was added slowly to a solution of 2.18 g. (19.9 mmoles) of I in 10 ml. of benzene at 0°. After the gas evolution had ceased, the mixture was filtered cold and the benzene was removed from the filtrate at 0°. The solid residue was dried for 6 hr. at 0° under vacuum.

Anal. Calcd. for $Cl_2AlBH_4 \cdot N(CH_3)_3$: Al, 15.72; Cl, 41.3; B, 6.30; active H, 2.33. Found: Al, 16.04; Cl, 41.6; B, 5.61; active H, 2.36.

In another run 1.02 g. (10.2 mmoles) of I in 2.61 g. (9.64 mmoles) of mercuric chloride were allowed to react in 50 ml. of toluene at −10° for 1 hr. Then 1.07 g. (48.6 mmoles) of lithium borohydride was added and the mixture was stirred 2 hr. at −10° and 4 hr. at 0°. After filtration, the toluene was removed at 25° under vacuum and a white solid residue was obtained. Sublimation of the residue at 35° afforded 0.76 g. (5.8 mmoles) of III, m.p. 78°.

Analysis.—The active hydrogen content was determined by hydrolysis with a 1:1 methanol–trifluoroacetic acid mixture in sealed bulbs. The bulbs were allowed to stand overnight and then heated for 3 hr. at 50° before being opened. The amount of hydrogen evolved was measured by means of a Toepler pump and a calibrated storage bulb system. The remaining solution was treated as previously described in order to determine the aluminum content.[14] An aliquot was titrated for boric acid by the conventional mannitol titration procedure.

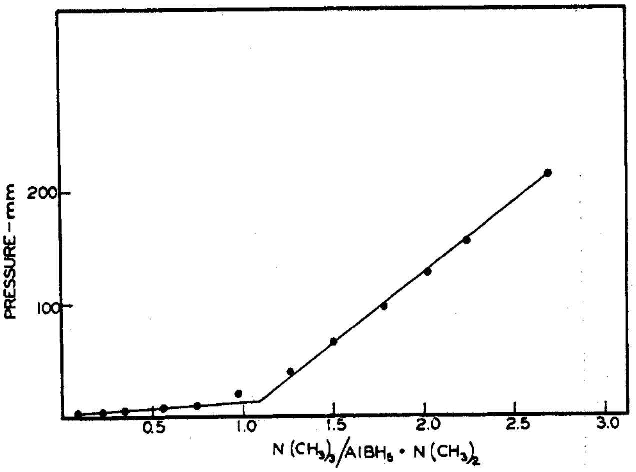

Fig. 2.—Titration of $HAlBH_4 \cdot N(CH_3)_2$ with $N(CH_3)_3$ in decane solution.

Molecular Weight Determination.—The apparent molecular weights of dimethylamino hydrido aluminum borohydride and dimethylamino aluminum borohydride were determined cryoscopically in benzene.[14] Mol. wt. of $HAlBH_4N(CH)_3)_2$: Calcd. 87; Found 245, 250. Mol. wt. of $Al(BH_4)_2N(CH_3)_2$: Calcd. 101; Found 231, 235.

Infrared Spectra.—The infrared spectra of the hydrido aluminum borohydride complexes were obtained on Nujol mulls (for the solid products) or on capillary layers (liquid samples) with a Perkin-Elmer Model 21 spectrophotometer. All samples were prepared and handled in the drybox prior to obtaining the spectra.

N.m.r. Spectra.—The B^{11} n.m.r. spectra were obtained on 10–15% solutions of the complexes in benzene, with a Varian Model V-4300B spectrometer operating at 12.8 Mc. Trimethylborate was used as an external standard. The chemical shifts and coupling constants are presented in Table III.

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[14] J. K. Ruff and M. F. Hawthorne, *J. Am. Chem. Soc.*, **83**, 535 (1961).

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The Synthesis and Spectra of B-Trifluoroborazine

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The reaction of B-trichloroborazine, $Cl_3B_3N_3H_3$, vapor with potassium fluoroborate produces B-trifluoroborazine, $F_3B_3N_3H_3$, together with $F_2ClB_3N_3H_3$ and $FCl_2B_3N_3H_3$. Pure B-trifluoroborazine has been prepared by the reaction of B-trichloroborazine with excess antimony trifluoride. Its mass and infrared spectra are compared with those of B-trichloroborazine and trifluoroboroxine. The tendency for ring cleavage *vs.* breaking of boron–halogen bonds in these isoelectronic heterocyclic ring compounds is discussed.

Introduction

Although numerous B-chloro- and B-bromo-derivatives of borazine have been known for some time there is little information about the fluoroborazines.[2] Wiberg and Horeld[3] have reported that B-trifluoro N-trimethylborazine is formed from dimethylfluoroborane and methylamine. Pearson and Frazer[4] prepared mixtures of fluoroborazines from the reaction of diborane with tetrafluorohydrazine. They obtained mass spectral and infrared evidence for $FH_2B_3N_3H_3$, $F_2HB_3N_3H_3$, and $F_3B_3N_3H_3$ in the mixtures but did not isolate the pure compounds. They suggested that the mixtures might have isomeric molecules present, rendering separation difficult. Reaction of ammonia with boron trifluoride gives the crystalline adduct $H_3N{:}BF_3$, which, when heated, decomposes to NH_4BF_4 and boron nitride with no evidence for the intermediate formation of fluoroborazines.[5] Efforts to prepare

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[1] Alfred P. Sloan Fellow.

[2] J. C. Sheldon and B. C. Smith, *Quart. Rev.* (London), **14**, 200 (1960).

[3] E. Wiberg and G. Horeld, *Z. Naturforsch.*, **6B**, 338 (1951).

[4] R. K. Pearson and J. W. Frazer, *J. Inorg. Nucl. Chem.*, **21**, 188 (1961).

[5] A. W. Laubengayer and G. Condike, *J. Am. Chem. Soc.*, **70**, 2274 (1948).

$F_3B_3N_3H_3$ by the reaction of boron trifluoride and ammonium bifluoride have been negative,[6] although the analogous reaction of boron trichloride with ammonium chloride gives an excellent yield of B-trichloroborazine.

However, considering the relative strengths of B–F and B–Cl bonds, it appeared likely that fluoroborazines could be prepared from B-trichloroborazine by halogen substitution. This paper describes success in applying this method.[7]

Experimental

Apparatus and Materials.—The mass spectrometer, the high temperature oven, and the gas inlet system have been described previously.[8] The inlet system was set up to permit introduction of vapor by direct sublimation from the solid. The B-trichloroborazine[9] was purified by vacuum sublimation at 50° upon a cold finger held at 0°.

The Reaction of $Cl_3B_3N_3H_3$ with BF_3.—Boron trifluoride at 100 mm. pressure was brought together with $Cl_3B_3N_3H_3$ at temperatures between 20 and 85° in an auxiliary vacuum system attached to the mass spectrometer. The excess BF_3 was pumped off and the solids were sublimed into the mass spectrometer. The mass spectrum gave evidence for the formation of very small amounts of $FCl_2B_3N_3H_3$ but none for $F_3B_3N_3H_3$.

The Reaction of $Cl_3B_3N_3H_3$ with KBF_4 in the Mass Spectrometer.—A method was devised to carry out the reaction of $Cl_3B_3N_3H_3$ with KBF_4 directly in the mass spectrometer oven. A graphite cell was packed loosely with large granules of KBF_4 and $Cl_3B_3N_3H_3$ vapor, subliming from the solid at room temperature, was passed into the oven. A molecular beam representative of the gaseous chemical species present above the KBF_4 in the oven entered directly into the ion source for spectral analysis.

When the cell was at room temperature the only species, apart from $Cl_3B_3N_3H_3$, emerging from the oven was a small quantity of $FCl_2B_3N_3H_3$. At temperatures above 200° much more halogen exchange occurred to give a mixture of $Cl_3B_3N_3H_3$, $FCl_2B_3N_3H_3$, $F_2ClB_3N_3H_3$, $F_3B_3N_3H_3$, and BF_3. The mass spectrum of this mixture was complex and no attempt was made to catalog the spectra of the mixed chloro–fluoro species because various fragment ions from different parents would have common masses. Oven temperatures between 300 and 400° were found to be optimum for conversion to $F_3B_3N_3H_3$, this species being estimated to be between 50 and 75 mole % of the total borazine material emerging. At temperatures above 400° the partial pressure of BF_3 formed became too high to allow operation of the spectrometer.

The halogen exchange reaction observed may be summarized by the generalized equation

$$Cl_3B_3N_3H_3 + nKBF_4 \longrightarrow F_nCl_{3-n}B_3N_3H_3 + nKCl + nBF_3$$

the degree of halogen exchange increasing with increased temperature.

The Reaction of $Cl_3B_3N_3H_3$ with KBF_4 in a Copper Reactor.—In order to favor the isolation of a sizable sample of fluoroborazines the vapor from $Cl_3B_3N_3H_3$ held at 45° was passed through a bed of KBF_4 packed loosely in a copper tube fitted inside a glass tube which was part of a vacuum line. The KBF_4 was heated to 200° and the reaction products were condensed in a pair of traps at −78° and −196°, respectively. The whole system was pumped on continuously by pumps beyond the colder trap.

The −196° trap was found to contain only BF_3. A white solid collected in the −78° trap and, when warmed to room temperature, this solid partly melted and partly sublimed to the upper part of the tube to form white crystals. The liquid was distilled out at room temperature and recondensed in a −78° trap. The condensate, when brought to room temperature, again melted partially to give a mixture of liquid with a small quantity of solid. Mass spectral analysis showed this mixture to consist of about equal quantities of $F_2ClB_3N_3H_3$ and $F_3B_3N_3H_3$. The solid left behind in the original −78° trap contained both of these compounds and also $FCl_2B_3N_3H_3$, all in about equal mole proportions. This mixture could not be resolved readily by fractional evaporation and condensation.

Recycle of the mixture of fluoro–chloroborazines through KBF_4 at 200° caused no appreciable increase in the degree of fluorination. The proportion of chloroborazines in the mixture could be reduced to about 10 mole % by stirring with granulated magnesium in dry diethyl ether at room temperature. The species containing chlorine reacted with the metal to form a non-volatile product from which the unreactive $F_3B_3N_3H_3$ could be sublimed, but complete removal of the chloroborazines to give pure $F_3B_3N_3H_3$ was not achieved.

The Reaction of $Cl_3B_3N_3H_3$ with SbF_3.—Pure $F_3B_3N_3H_3$ was finally prepared by the reaction of $Cl_3B_3N_3H_3$ with excess SbF_3.

$$Cl_3B_3N_3H_3 + SbF_3 \longrightarrow F_3B_3N_3H_3 + SbCl_3$$

This reaction took place exothermally when $Cl_3B_3N_3H_3$ was heated with excess SbF_3 under dry nitrogen in a flask to the melting point of $Cl_3B_3N_3H_3$ (83°) or when a solution of $Cl_3B_3N_3H_3$ in toluene was refluxed with excess SbF_3. In either case some decomposition of the $Cl_3B_3N_3H_3$ occurred with evolution of BCl_3, identified by chemical analysis. No reaction occurred in refluxing diethyl ether or petroleum ether.

The $F_3B_3N_3H_3$ was isolated from the solid reaction products by sublimation *in vacuo* or, better, by extraction with petroleum ether or diethyl ether, followed by removal of the solvent by distillation and sublimation of the residue. The sublimate was shown to be pure $F_3B_3N_3H_3$ by mass spectral analysis. The yields were about 20%.

Results and Discussion

Properties of $F_3B_3N_3H_3$.—B-Trifluoroborazine forms colorless needles which sublime readily *in vacuo* and melt at 118° in a sealed capillary tube. It is quite soluble in carbon tetrachloride, carbon disulfide, benzene, and toluene and somewhat less soluble in diethyl ether, cyclohexane, and petroleum ether. It reacts rapidly with moist air and dissolves in water with hydrolysis and evolution of ammonia to form a clear solution. Concentration of this aqueous solution *in vacuo* leaves a clear viscous liquid.

The Mass Spectra of $F_3B_3N_3H_3$, $Cl_3B_3N_3H_3$, and $F_3B_3O_3$.—Table I catalogs the mass spectrum observed for $F_3B_3N_3H_3$, giving the relative intensity of each of the ions observed. Table II compares the monoisotopic mass spectrum of $F_3B_3N_3H_3$ (excluding the low mass range) with those of $Cl_3B_3N_3H_3$ and B-trifluoroboroxine, $F_3B_3O_3$,[10] each ion intensity referring to the total contribution of all the isotopic masses for a given species of ion. The number of boron and chlorine atoms in each species was determined by comparing the isotope struc-

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[6] F. B. Hutto, thesis presented to the Faculty of the Graduate School, Cornell University, in partial fulfillment of the requirements for the M.S. degree, September, 1950.

[7] While this paper was being written a paper presented by K. Niedenzu, J. W. Dawson, and H. Beyer at the September, 1962, National Meeting of the American Chemical Society, Division of Inorganic Chemistry, paper no. 18, described the formation of fluoroborazines by the reaction of a variety of fluorides with $Cl_3B_3N_3H_3$. Their results, together with our findings, establish halogen exchange as a satisfactory route to the fluoroborazines.

[8] D. R. Bidinosti and R. F. Porter, *J. Am. Chem. Soc.*, **83**, 3737 (1961).

[9] Obtained from U. S. Borax Corporation.

[10] R. F. Porter, D. R. Bidinosti, and K. F. Watterson, *J. Chem. Phys.*, **36**, 2104 (1962).

TABLE I

THE MASS SPECTRUM OF B-TRIFLUOROBORAZINE

m/e	Species	Relative intensity
135	$F_3B_3^{11}N_3H_3^+$	100
134	$F_3B_2^{11}B^{10}N_3H_3^+$	73
133	$F_3B^{11}B_2^{10}N_3H_3^+$	21
132	$F_3B_3^{10}N_3H_3^+$	12
119	$F_3B_3N_2H^+$	37
118	$F_3B_2^{11}B^{10}N_2H^+$	29
117	$F_3B^{11}B_2^{10}N_2H^+$	8
116	$F_3B_3^{10}N_2H^+$, $F_2B_3^{11}N_3H_3^+$	5
115	$F_2B_2^{11}B^{10}N_3H_3^+$	4
114	$F_2B^{11}B_2^{10}N_3H_3^+$	2
113	$F_2B_3^{10}N_3H_3^+$	1
99	$F_2B_3^{11}N_2^+$	18
98	$F_2B_2^{11}B^{10}N_2^+$	14
97	$F_2B^{11}B_2^{10}N_2^+$	3
96	$F_2B_3^{10}N_2^+$	1
90	$F_2B_2^{11}N_2H_2^+$	17
89	$F_2B^{11}B^{10}N_2H_2^+$	10
88	$F_2B_2^{10}N_2H_2^+$	1
74	$F_2B_2^{11}N^+$	39
73	$F_2B^{11}B^{10}N^+$	19
72	$F_2B_2^{10}N^+$	3
71	$FB_2^{11}N_2H_2^+$	36.5
70	$FB^{11}B^{10}N_2H_2^+$, $FB_2^{11}N_2H^+$	32
69	$FB_2^{10}N_2H_2^+$, $FB^{11}B^{10}N_2H^+$, $FB_2^{11}N_2^+$	12
68	$FB_2^{10}N_2H^+$, $FB^{11}B^{10}N_2^+$	2
56	$FB_2^{11}NH^+$	3
55	$FB^{11}B^{10}NH^+$, $FB_2^{11}N^+$	10
54	$FB_2^{10}NH^+$, $FB^{11}B^{10}N^+$	4
53	$FB_2^{10}N^+$	<1
51	$B_2^{11}N_2H^+$	2
50	$B^{11}B^{10}N_2H^+$, $B_2^{11}N_2^+$	8
49	$B^{11}B^{10}N_2^+$	3
45	$FB^{11}NH^+$	4
44	$FB^{10}NH^+$, $FB^{11}N^+$	3

TABLE II

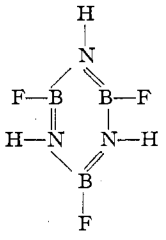
THE MONOISOTOPIC SPECTRA OF $F_3B_3N_3H_3$, $Cl_3B_3N_3H_3$, AND $F_3B_3O_3$

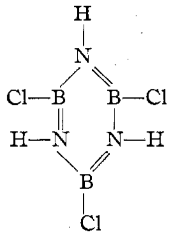
$F_3B_3N_3H_3$		$Cl_3B_3N_3H_3$		$F_3B_3O_3$	
Ion species	Intensities	Ion	Intensities	Ion	Intensities
$F_3B_3N_3H_3^+$	100	$Cl_3B_3N_3H_3^+$	100	$F_3B_3O_3^+$	100
$F_3B_3N_2H^+$	37				
$F_2B_3N_3H_3^+$	5	$Cl_2B_3N_3H_3^+$	79	$F_2B_3O_3^+$	10
$F_2B_3N_3H_2^+$	<0.5				
				$F_3B_2O^+$	60
$F_2B_3N_3H^+$	<0.9				
$F_2B_3N_2^+$	19	$Cl_2B_3N_2^+$	13		
		$ClB_3N_3H_2^+$	13		
$F_2B_2N_2H_2^+$	14			$F_2B_2O_2^+$	31
$F_2B_2N^+$	31	$Cl_2B_2N^+$	8	$FB_2O_2^+$	11
$FB_2N_2H_2^+$	33	$ClB_2N_2H_2^+$	14		
$FB_2N_2H^+$	11	$ClB_2N_2H^+$	6		

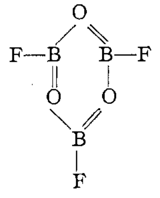
ture of the fragment with that calculated from the normal isotope abundances of B^{10}, B^{11}, Cl^{35}, and Cl^{37}. The presence of very small ion intensities due to background permitted a direct mass count for the $F_3B_3N_3H_3$ fragments below mass 100. Other masses were determined by magnetic field measurements using a rotating coil gaussmeter.

The ion intensities in the spectrum of trifluoroborazine obtained from the reaction of $Cl_3B_3N_3H_3$ with KBF_4 within the oven inside the spectrometer agreed to within 10% and generally to within 5% of the intensities measured on the crystalline compound produced outside the spectrometer by the reaction of $Cl_3B_3N_3H_3$ with SbF_3, except in the case of the ion $B_2F_2N^+$. In this case the value obtained from the crystalline material was taken as being the more reliable.

The coplanar heterocyclic aromatic ring structure of B-trichloroborazine has been well established.[11] The method of synthesis used for making our trifluoroborazine from B-trichloroborazine by halogen exchange under mild conditions should produce B-trifluoroborazine. As will be shown later, the infrared data on our trifluoroborazine substantiates a heterocyclic ring structure with B–F and N–H bonding. Trifluoroboroxine, $F_3B_3O_3$, also has a heterocyclic aromatic ring structure with B–F bonding.[12]


B-Trifluoro-borazine


B-Trichloro-borazine


B-Trifluoro-boroxine

It is particularly interesting to compare these three compounds which have isoelectronic aromatic ring systems.

As indicated in Table II, certain similarities in the mass spectra of the trihalogenoborazines and trifluoroboroxine are evident. The parent ion predominates in each case. This is generally characteristic of aromatic ring systems, whereas for the halogenated derivatives of saturated hydrocarbons the major ion is usually that resulting from the loss of one halogen atom. The major fragment ion from $Cl_3B_3N_3H_3$ results from the loss of one chlorine atom. We also observed this in the mass spectra for $FCl_2B_3N_3H_3$ and $F_2ClB_3N_3H_3$. The presence of fluorine at all of the boron positions causes very different fragmentation patterns. For $F_3B_3N_3H_3$ the major ion fragment is formed by ring cleavage and loss of NH and H, giving $F_3B_3N_2H^+$. With $F_3B_3O_3$ ring opening occurs with loss of one boron and two oxygen atoms, accompanied by fluorine migration to give $F_2B_2O^+$ as the major ion fragment.

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[11] D. L. Coursen and J. L. Hoard, *J. Am. Chem. Soc.*, **74**, 1742 (1952); K. Coffin and S. H. Bauer, *J. Phys. Chem.*, **59**, 193 (1955).

[12] H. D. Fisher, W. J. Lehman, and I. Shapiro, *J. Phys. Chem.*, **65**, 1166 (1961).

The mass spectra of $F_3B_3N_3H_3$ and $Cl_3B_3N_3H_3$ have in common the ion fragments $X_2B_3N_2^+$, $X_2B_2N^+$, $XB_2N_2H_2^+$, and $XB_2N_2H^+$ which result from ring cleavage. The formation of $F_2B_2H_2^+$ and $F_2B_2O_2^+$ again suggests that the ring cleavage is favored over B–F bond breakage. These observations are consistent with the high stability of B–F terminal bonds compared to the B–N and B–O bonds in the rings.

Although these observations cannot be used rigorously to predict properties of the trihalogenated borazines we might expect, for example, that $F_3B_3N_3H_3$ is more likely to undergo ring cleavage to form higher condensed heterocyclic polymers than to split out HF intermolecularly to form diborazinyl compounds. The latter might be the more likely products in the condensation of chlorinated borazines, due to the easy intermolecular elimination of HCl without ring cleavage.

Thermal Decomposition of $F_3B_3N_3H_3$ and $Cl_3B_3N_3H_3$.—A comparison of the thermal stabilities of $F_3B_3N_3H_3$ and $Cl_3B_3N_3H_3$ was made by observing their low pressure pyrolysis reactions at temperatures between 200 and 600°. The procedure was to flow the reactant vapors at pressures of 10^{-5} atmosphere or lower into a heated graphite effusion cell and to observe in the mass spectrometer the gaseous products leaving the cell. At 600° pyrolytic decomposition was extensive. For $F_3B_3N_3H_3$ the products were largely H_2, N_2, and BF_3, but for $Cl_3B_3N_3H_3$, N_2 and HCl were predominant. This again points out the high stability of B–F bonds.

The Infrared Spectra of $F_3B_3N_3H_3$ and $Cl_3B_3N_3H_3$.—The infrared spectra were measured for the vapor state in a cell 10 cm. long with KBr windows on a Perkin-Elmer Model 21 double beam spectrophotometer and are cataloged in Table III. Although the vapor pressure of $F_3B_3N_3H_3$ at room temperature is only about 0.1 mm., strong peaks were obtained. The vapor pressure of $Cl_3B_3N_3H_3$ is so low that only the stronger peaks were visible in the gas cell. The intensity of absorption was increased slightly by condensing solid $Cl_3B_3N_3H_3$ in the cell and then heating to about 60° to take the spectrum. The spectrum of $Cl_3B_3N_3H_3$ also was measured in solution in carbon tetrachloride and in carbon disulfide. The spectrum of the mixture of $F_3B_3N_3H_3$ and $F_2ClB_3N_3H_3$ obtained from the reaction of $Cl_3B_3N_3H_3$ with KBF_4 was measured as vapor.

TABLE III

INFRARED SPECTRA, CM.$^{-1}$

$F_3B_3N_3H_3$ B-Trifluoroborazine	$Cl_3B_3N_3H_3$ B-Trichloroborazine Vapor	CS$_2$ soln.
3509 (m)	3485 (m)	3508
1531 (s)		
1514 (vs)	1475 (sh)	1475
1505 (vs)	1459 (vs)	1461
1401 (m)	(m)	1377
1333 (m)	(m)	1333
	(m)	1215
1135 (s)	(s)	1030
	(sh)	749
923 (m)	(m)	740
735 (s)	717 (w)	724
727 (s)	710 (m)	704
685 (w)		
660 (m)		

Comparison of the infrared spectrum of $F_3B_3N_3H_3$ with the published spectra of borazine,[13] trifluorobenzene,[14] and trifluoroboroxine[15] makes possible a tentative explanation of the main features of the spectrum of $F_3B_3N_3H_3$. A similar comparison also can be made of the spectrum of trichloroborazine with that of trichlorobenzene.

The band near 3500 cm.$^{-1}$ which occurs in all three borazines is attributable to an N–H stretching mode. The absence of a B–H stretching vibration in the region of 2500 cm.$^{-1}$ is apparent. B–N ring stretching vibrations give rise to the band at 1514 cm.$^{-1}$ in $F_3B_3N_3H_3$ and near 1460 cm.$^{-1}$ in $Cl_3B_3N_3H_3$. Fisher, *et al.*,[15] have assigned an absorption band for $F_3B_3O_3$ at 996 cm.$^{-1}$ to a B–F asymmetrical stretching frequency and it seems reasonable to assign the 922 cm.$^{-1}$ band of $F_3B_3N_3H_3$ to a similar mode. The corresponding band for B–Cl in $Cl_3B_3N_3H_3$ is probably at 740 cm.$^{-1}$. Out-of-plane vibrations may then account for the two remaining groups of bands in the spectra of $F_3B_3N_3H_3$ and $Cl_3B_3N_3H_3$.

The spectrum found for $Cl_3B_3N_3H_3$ is in substantial agreement with other reports.[16,17]

The infrared spectrum of the mixture of $F_3B_3N_3H_3$ and $F_2ClB_3N_3H_3$ showed, in addition to bands due to $F_3B_3N_3H_3$, bands at 1507, 1485, 1037 (s), 858 (w), and 823 (s) cm.$^{-1}$. The first four appear close to the bands in $Cl_3B_3N_3H_3$, the fifth is apparently unique to $F_2ClB_3N_3H_3$.

Ultraviolet Study of $F_3B_3N_3H_3$.—An attempt was made to measure the ultraviolet spectrum of $F_3B_3N_3H_3$ vapor by condensing a sample into a 10-cm. silica cell and allowing the cell to come to room temperature to fill the cell with vapor. No absorption could be detected between 3000 and 1900 Å.

The structure of single crystals of $F_3B_3N_3H_3$ is being investigated by means of X-ray analysis and electron diffraction structural analysis is being carried out on the vapor.

Acknowledgments.—We wish to thank the National Science Foundation and the Advanced Research Projects Agency for financial support for this research.

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

(14) J. R. Nielsen, C. Y. Liang, and D. C. Smith, *ibid.*, **9**, 177 (1950).

(15) H. D. Fisher, W. J. Lehmann, and I. Shapiro, *J. Phys. Chem.*, **65** 1166 (1961).

(16) H. Watanabe, M. Narisada, T. Nakagawa, and M. Kubo, *Spectrochim. Acta*, **16**, 78 (1960).

(17) I. M. Butcher, W. Gerrard, E. F. Mooney, R. A. Rothenbury, and H. A. Willis, *ibid.*, **18**, 1487 (1962).