stabilize the boronium ions $C_6H_5BXS_2^+$ and $(C_6H_5)_2$-BS_2^+ to any great extent. In the case of the diphenyl-haloboranes the shape of the conductance curve suggests that all equilibria given in eq 1–4 are involved.

Experimental Section

Preparation and Purification of Reagents.—Boron tribromide (Alfa Inorganics) was purified by repeated vacuum distillation through traps at −45 and −63.5° until the vapor pressure was constant at 18.0 ± 0.2 mm at 0° (lit.[27] 18.5 mm at 0°).

Boron triiodide was prepared according to the procedure described by Klanberg and coworkers.[28] The colorless crystalline boron triiodide readily undergoes photochemically induced dissociation. In order to avoid the difficulty of purifying and storing pure samples of boron triiodide, samples of the boron triiodide–acetonitrile adduct were prepared from slightly impure samples of boron triiodide. The crystalline adduct is easily purified by recrystallization from acetonitrile. Consequently, all conductance studies of the boron triiodide system were carried out using the boron triiodide–acetonitrile adduct.

Dichlorophenylborane was prepared by the method described by Burch, et al.[29] The resulting deep red liquid was fractionally distilled under reduced pressure and the colorless fraction [bp 49–52° (5 mm)] was collected in an ampoule with a breaker side arm. Further purification was obtained by repeated vacuum distillation through traps at −80 and 0°. The final product has a vapor pressure of 78 mm at 23°.

Samples of bromo- and chlorodiphenylboranes were kindly provided by Mr. B. Laube. The slightly discolored chlorodiphenylborane was fractionally distilled under reduced pressure and the center cut boiling at 98–100° (1 mm) [lit. bp 94–96° (0.1 mm),[30] 106–108° (3–4 mm)[31]] was retained. Bromodiphenylborane was fractionally distilled under reduced pressure and the

(27) A. Finch, I. J. Hyams, and D. Steele, *Trans. Faraday Soc.*, **61**, 398 (1965).

(28) F. Klanberg and H. W. Kohlshutter, *Chem. Ber.*, **94**, 786 (1961); *Angew. Chem.*, **69**, 478 (1957).

(29) J. E. Burch, W. Gerrard, M. Howarth, and E. F. Moony, *J. Chem. Soc.*, 4916 (1960).

fraction boiling at 84–85° (0.01 mm) [lit.[30] bp 112–115° (0.05 mm)] was collected.

Boron Halide–Acetonitrile Adducts.—The crystalline 1:1 adducts of acetonitrile with BCl_3, BBr_3, and BI_3 were prepared by standard procedures[1,32] and purified by vacuum sublimation at 60–70°. The adduct of dichlorophenylborane was prepared by the direct addition of acetonitrile to a solution of dichlorophenylborane in carbon tetrachloride and was purified by vacuum sublimation at 70° (1 mm). The white crystalline product melts at 118–121° in a sealed ampoule (lit.[22] mp 118.5–120.5°).

Boron Halide–Pyridine Adducts.—These were prepared by the direct addition of pyridine to a cooled solution (−80°) of the halide in methylene chloride and were purified by vacuum sublimation.

The purified $BCl_3 \cdot py$ adduct melts sharply at 113.0–113.5° (lit.[33] mp 113–114°). *Anal.* Calcd for $C_5H_5BCl_3N$: C, 30.58; H, 2.55; Cl, 54.22; N, 7.14. Found: C, 30.55; H, 2.42; Cl, 54.04; N, 7.27.

Crystalline $BBr_3 \cdot py$ melts sharply at 127.0–127.5° (lit. mp 127–128°,[19] 128–129°[32]). *Anal.* Calcd for $C_5H_5BBr_3N$: C, 18.20; H, 1.50; Br, 72.72; N, 4.25. Found: C, 18.64; H, 1.48; Br, 72.50; N, 4.31.

The preparation of $BI_3 \cdot 2py$ has been described elsewhere.[4]

The apparatus and procedure for conductance measurements were described before in detail.[1,3,4] The ^{11}B nmr spectra were taken with a Varian Model HA-100 high-resolution spectrometer operated at 32.083 Mc using the boron trifluoride–diethyl ether adduct as the external reference. The infrared spectra were recorded on a Beckman Model IR 10 spectrophotometer.

(30) A. Finch, P. J. Gardner, E. J. Pearn, and G. B. Watts, *Trans. Faraday Soc.*, **63**, 1880 (1967).

(31) K. Niedenzu, H. Beyer, and J. W. Dawson, *Inorg. Chem.*, **1**, 738 (1962).

(32) J. M. Miller and M. Onyszchuck, *Can. J. Chem.*, **44**, 899 (1966).

(33) H. C. Brown and R. R. Holmes, *J. Am. Chem. Soc.*, **78**, 2173 (1956)

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Reactivity of Boroxine. Reactions with Azomethane and Azoethane[1]

By ANDREW KALDOR, IRA PINES, AND RICHARD F. PORTER

A product of the reaction of boroxine with azomethane and azoethane has been identified as a 1:1 azoalkane–borine addition compound. Structural information based on infrared and nuclear magnetic resonance data indicate that the product has the unsymmetrical configuration
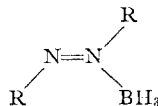
Introduction

Previous investigations of the chemistry of boroxine ($H_3B_3O_3$) have shown this compound to be reactive with a series of simple reagents: O_2,[2] CO,[2] BX_3,[3] and PF_3.[4]

(1) This work was supported by the Army Research Office, Durham, N. C., the Advanced Research Projects Agency, and the Undergraduate Research Participation Program of the National Science Foundation.

(2) G. H. Lee and R. F. Porter, *Inorg. Chem.*, **5**, 1329 (1966).

(3) M. Nadler and R. F. Porter, *ibid.*, **6**, 1192 (1967).

(4) L. Barton, *J. Inorg. Nucl. Chem.*, **30**, 1693 (1968).

In the reactions with CO and PF_3 boroxine behaves as an effective source of BH_3, to form simple addition-type products, while in the reaction with O_2 cyclic $H_2B_2O_3$ is produced. From these observations it was anticipated that azo compounds should react with boroxine either to yield new cyclic derivatives through the N=N linkage or to form addition products. This paper is the result of a series of studies of the reactions of boroxine with azomethane and azoethane.

Experimental Section

The preparation of boroxine by a flash reaction of low pressure $B_2H_6-O_2$ has been described.[5] *As emphasized in the earlier paper specific safety precautions are essential in this preparation.* The experimental procedure involved flashing a 50:50 mixture of B_2H_6 and O_2 at a total pressure of 20 mm in a 3-l. volume and immediately adding azomethane or azoethane in the gas phase from an external reservoir at sufficient pressure to prevent backflow of gases from the vessel. The reaction is rapid and is accompanied by formation of a white precipitate. Under optimum conditions about 3 mm of $H_3B_3O_3$ is produced in the flash reaction. The quantity of azo compound corresponded to about 20 cm of gas in a 100-ml volume. Thus the ratio of azo compound to boroxine exceeded 1:1. Products of the reaction were then pumped through a trap held at $-196°$ and H_2 was removed. The cold trap was found to contain unreacted azoalkane and a new product. The product was separated from the starting material by holding the trap at $-78°$ (in the case of azomethane) and at $-25°$ (in the case of azoethane) and pumping to remove the more volatile azoalkane. A number of isotopically labeled species were also obtained, starting with boroxine-$^{10}B_3$ or boroxine-d_3 and unlabeled azomethane and with boroxine (natural isotope abundances) and azomethane-d_6.

Diborane was prepared from $NaBH_4$ and $SnCl_2$ by the method described by Jeffers.[6] Azomethane was prepared by the reaction of 1,2-dimethylhydrazine hydrochloride and cupric chloride.[7] Azoethane was obtained from Merck Sharp and Dohme.

The product of the azomethane reaction (subsequently referred to as the azomethane–boroxine product) has vapor pressures of 1.5, 3.7, and 9.8 mm at 248, 261, and 273°K, respectively. Vapor density measurements obtained with samples of product weighing from 3 to 5 mg gave a molecular weight of 70 ± 7. The precision in this determination was limited largely by the instability of the compound as will be discussed later. Mass spectra of products were obtained with a 10-in. direction focusing mass spectrometer. Hydrogen nmr spectra were obtained at 60 MHz on a Varian A-60A operating with a temperature controller. Boron-11 nmr spectra of the product from the $H_3B_3O_3-N_2(C_2H_5)_2$ reaction were obtained at 15 MHz on a Varian HA-60 at the University of New York at Buffalo.[8] Infrared spectra were obtained on a Perkin-Elmer 521 spectrophotometer from 4000 to 250 cm^{-1}. Polystyrene film was used for calibration. The position of sharp bands could be located to ±1 cm^{-1}; however in all of the gas-phase spectra only one band exhibited a sharp Q branch. Hence the location of most bands is accurate only to ±5 cm^{-1}. Vapor-phase spectra were obtained in a 10-cm path length cell equipped with CsI windows. At the start of each run, a sample of 15 mm pressure was introduced into the cell, but decomposition was known to occur. The sample lifetime varied from 1 to 3 hr. Solid-film spectra were taken with a liquid helium variable-temperature dewar[9] equipped with a KBr target window and $^1/_{16}$ in. thick KBr external windows. The temperature was not measured directly. Before deposition was attempted the system was allowed to come to equilibrium with liquid He; during deposition no appreciable change in the helium boil-off could be observed. Hence the deposition temperature was assumed to be about 5°K.[10] The gas sample was introduced through a nozzle with a 0.010-in. opening, 3 cm from the target window. The flow rate, controlled by a Nupro stainless steel fine-metering valve with a micrometer handle, was calibrated to reproduce a deposition pressure of 10^{-4} Torr, while the pressure in the dewar was at all times 10^{-8} Torr or better. This rate was sufficiently slow to maintain window temperature dur-

(5) L. Barton, F. A. Grimm, and R. F. Porter, *Inorg. Chem.*, **5**, 2076 (1966).
(6) W. Jeffers, *Chem. Ind.* (London), 431 (1960).
(7) F. P. John, *J. Am. Chem. Soc.*, **59**, 1761 (1937).
(8) The authors wish to thank Professor O. T. Beachley and D. H. Templeman for obtaining the ^{11}B nmr spectra.
(9) Manufactured by the Hofman Division of Minnesota Valley Engineering Inc.
(10) Subsequent tests with a Cu *vs.* 2.1% Co–Au thermocouple imbedded in the target window confirmed this.

ing deposition. The total deposition time was 20 min. Spectra were also obtained on films warmed from ~5 to ~78°K and on films deposited at ~78°K on a KBr window cooled by a specially constructed glass dewar. A spectrum of the $H_3B_3O_3-N_2(CH_3)_2$ product was also obtained in a low-temperature matrix in CCl_4.

Results

Mass Spectra.—The gaseous product of the reaction of $H_3B_3O_3$ and $N_2C_2H_6$ was examined mass spectrometrically in the range m/e 0–300. The products of the reactions of axomethane with $D_3B_3O_3$, $H_3{}^{10}B_3O_3$, and $D_3{}^{10}B_3O_3$ and of boroxine with axomethane-d_6 were also studied. To aid in the analysis of the fragmentation pattern, the spectra of azomethane and azomethane-d_6 were also obtained. The mass spectrum of the product of the $H_3{}^{10}B_3O_3$ and azomethane reaction is given in Table I. The highest mass peak, neglecting the peak

TABLE I
MASS SPECTRUM OF THE BOROXINE–AZOMETHANE
REACTION PRODUCT (^{10}B SUBSTITUTED)^a

m/e	Intens (rel units)	m/e	Intens (rel units)
71	3.0	36	2.5
70	40.5	35	2.5
69	100	30	5.5
68	19.5	29	1.5
67	13.5	28	20.5
66	8.5	27	22.0
65	1.5	26	37.5
59	2.0	25	5.5
58	41.5	24	1.5
57	1.2	15	32.8
56	0.5	14	2.0
55	1.5	13	1.5
54	13.5	12	18.5
53	1.5	11	2.9
52	5.0	10	2.9
51	2.0		
44	.8		
43	65.5		
42	5.7		
41	47.1		
40	16.8		
39	52.0		
38	6.2		
37	8.2		

^{a} The sample was about 96% ^{10}B. All peaks were normalized to m/e 69 = 100. The ionizing electron energy was 75 V.

due to the natural ^{15}N and ^{13}C isotopes, occurs at m/e 71 for the natural isotopic form. The highest mass groupings for four isotopic species are shown in Figure 1. Comparison of those spectra for the compound with normal isotopic distribution with that for the ^{10}B product indicates that the compound has only one boron atom. The mass spectral cutoff for the product of the $H_3B_3O_3-N_2(CD_3)_2$ reaction is six mass units higher than that of the normal compound, indicating that two methyl groups are present. The shift in the pattern for the product of the $D_3B_3O_3-N_2(CH_3)_2$ reaction suggests that only two hydrogen atoms are bound to the boron. However, it should be cautioned that mass spectra for diborane and other boranes[11] do not always indicate the

(11) I. Shapiro, C. O. Wilson, J. F. Ditter, and W. J. Lehmann, Advances in Chemistry Series, No. 32, American Chemical Society, Washington, D. C., 1961, p 127.
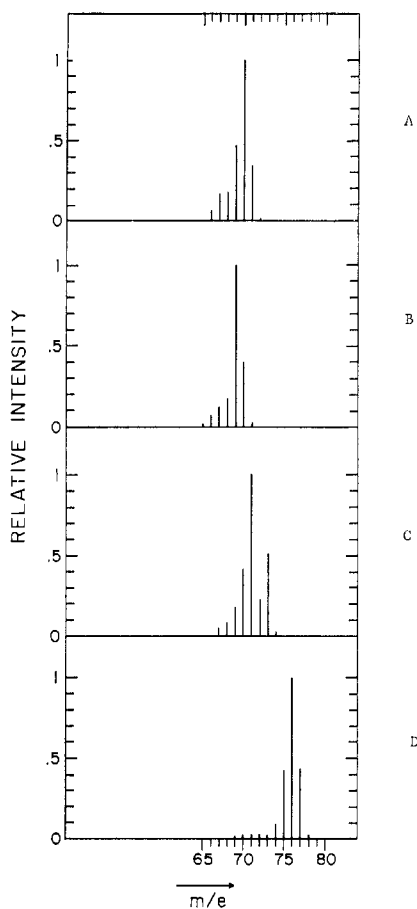
Figure 1.—Portions of the mass spectra (high-mass regions) of isotopically labeled products of the boroxine–azomethane reaction. The reactants are in order: (A) $H_3B_3O_3$ + $(CH_3)_2N_2$; (B) $H_3{}^{10}B_3O_3$ + $(CH_3)_2N_2$; (C) $D_3B_3O_3$ + $(CH_3)_2N_2$; (D) $H_3B_3O_3$ + $(CD_3)_2N_2$.

Figure 2.—Hydrogen nmr spectra of the product of the boroxine–azomethane reaction: A–D, product without solvent at 0, −20, −40, and −60°, respectively; E, product in TMS at −65°.

correct number of hydrogen atoms bound to boron and subsequent evidence is necessary to establish the correct number of bonding hydrogen atoms. Identification of some of the ion fragments was made by consideration of mass shifts from the several isotopic species studied. Several ion species especially those at m/e 58, 43, 28, and 15 may arise partially from azomethane produced from the sample subsequent to its injection in the mass spectrometer. Ion species from the boroxine–azomethane product that can be identified as unique are m/e 55 ($HBN_2CH_3{}^+$) and m/e 27 ($HBCH_3{}^+$). Several of the ion fragments may consist of more than one atomic composition.

The mass spectral cutoff for the boroxine–azoethane product occurred in the group m/e 99, 98, 97, 96, and 95 with intensities in the ratios 6.4:30.7:100:64:18.6.

Nmr Studies.—Hydrogen nmr spectra of the boroxine–azomethane product are shown in Figure 2. For the pure product, a strong broad line (or collapsed group of lines) at about 3.8 ppm downfield from TMS and slightly downfield from azomethane tended to narrow as the temperature was raised from −60 to 0°. In TMS solution this feature appears as a doublet (lower curve in Figure 2). These lines can be associated with the methyl groups bound to nitrogen. Two very broad lines at about 2.75 and 1.1 ppm down-
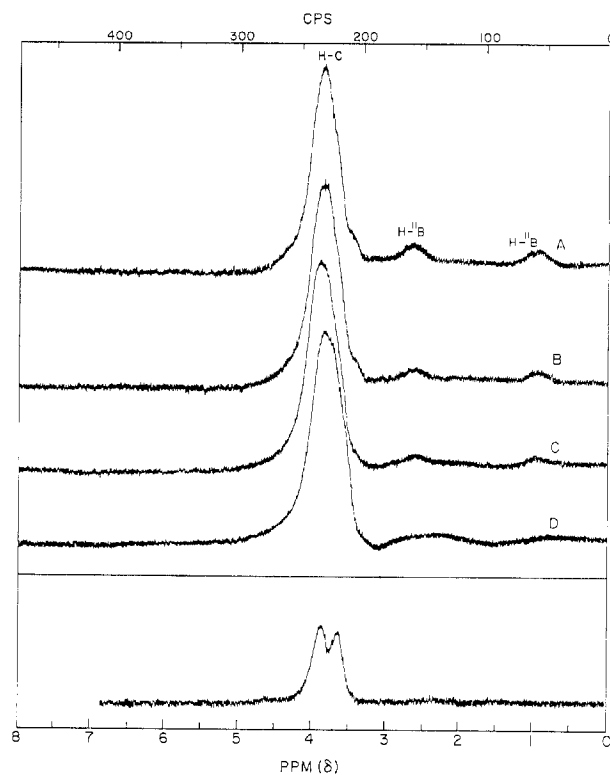
field from TMS narrowed considerably as the sample temperature was increased. These lines are associated with two components of a four-line sequence expected for H atoms bound to ^{11}B (spin of $^3/_2$). One of the other components is apparently masked by the strong methyl protons and the other is to the high field of TMS. The $H-{}^{11}B$ coupling constant is about 100 cps. A sample with deuterated methyl groups on nitrogen and ordinary H atoms on boron also gave an $H-{}^{11}B$ coupling constant close to this value, but the resonances were too broad for accurate measurements. *Experiments with the pure compound were abandoned since on one occasion the compound in the nmr tube exploded when it had reached room temperature.* The product of the boroxine–azoethane reaction appeared to be somewhat more stable and its nmr spectra are more informative. In Figure 3 are shown the hydrogen nmr spectra for both azoethane and the boroxine–azoethane reaction product in TMS solution. It is evident from the figure that the proton resonances in the CH_2 and CH_3 groups are shifted downfield from the analogous protons in azoethane. The low-field resonances in the product appear as a composite of two quartets (H in CH_2 split by CH_3) with centers at about 4.30 and 3.95 ppm relative to TMS. The high-field resonances appear as a partial superposition of two triplets (H in CH_3 split by CH_2) with centers at 1.53 and 1.38 ppm. Peaks at 4.7 and 3.0 ppm are assigned as two components of the $H-{}^{11}B$ resonance. The other two components are masked by the methyl protons in the compound and TMS (not shown in figure). The $H-{}^{11}B$ coupling constant is 102 ± 2 cps. The ^{11}B nmr
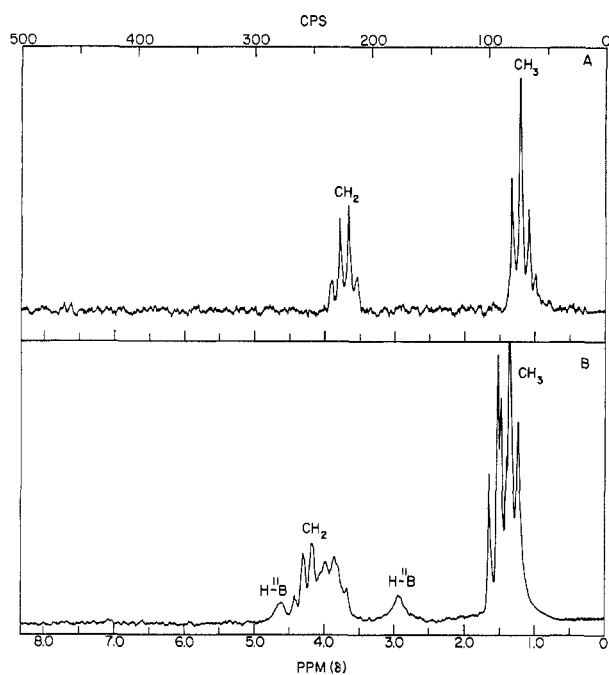
Figure 3.—Hydrogen nmr spectra of azoethane (A) and the boroxine–azoethane product (B) in TMS at 20°.

spectrum of this compound gives a well-defined 1:3:3:1 quartet (Figure 4) upfield from $BF_3 \cdot (C_2H_5)_2O$ by 170 ppm (35.2 ppm from $B(OC_2H_5)_3$). The peak separation gives a $^{11}B-H$ coupling constant of 102 cps in agreement with the hydrogen spectrum.
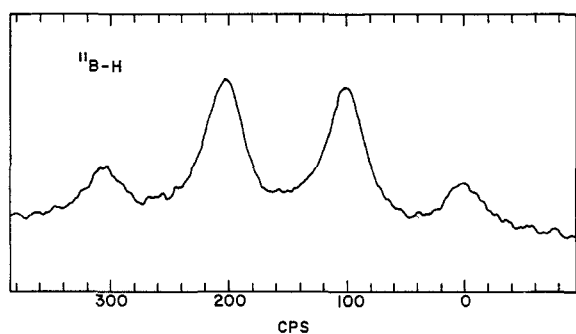

Figure 4.—Boron-11 nmr spectrum of the boroxine–azoethane product in TMS: the $BF_3(OC_2H_5)_3$ reference is not shown.

Infrared Spectra.—Infrared spectra for the boroxine–azomethane product with B–H, C–H; B–D C–H; and B–H, C–D isotopic labeling are illustrated in Figure 5. Low-temperature film spectra and CCl_4 matrix spectrum are shown in Figure 6. Spectra of low-temperature films warmed up and then quenched are shown in Figure 7. Frequency measurements are listed in Table II.

Discussion

The mass spectral data for the boroxine–azomethane and boroxine–azoethane products are somewhat confusing since the highest mass peaks suggest that the molecules contain a BH_2 group. However, the ^{11}B nmr data show that a BH_3 group is present, and we must assume that the molecule loses a proton on ioni-
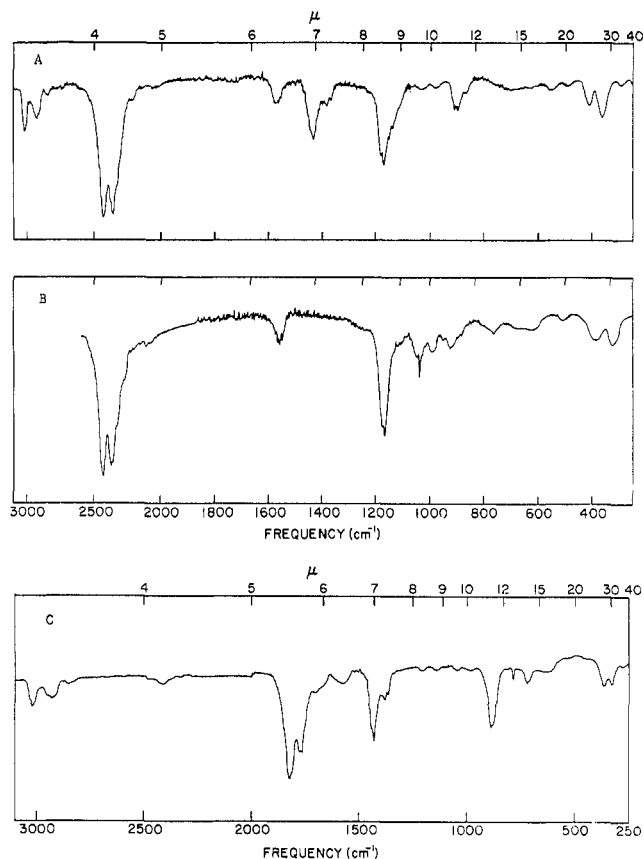

Figure 5.—Infrared spectra of the gaseous product of the reactions: (A) $H_3B_3O_3$ + $(CH_3)_2N_2$; (B) $H_3B_3O_3$ + $(CD_3)_2N_2$; (C) $D_3B_3O_3$ + $(CH_3)_2N_2$.
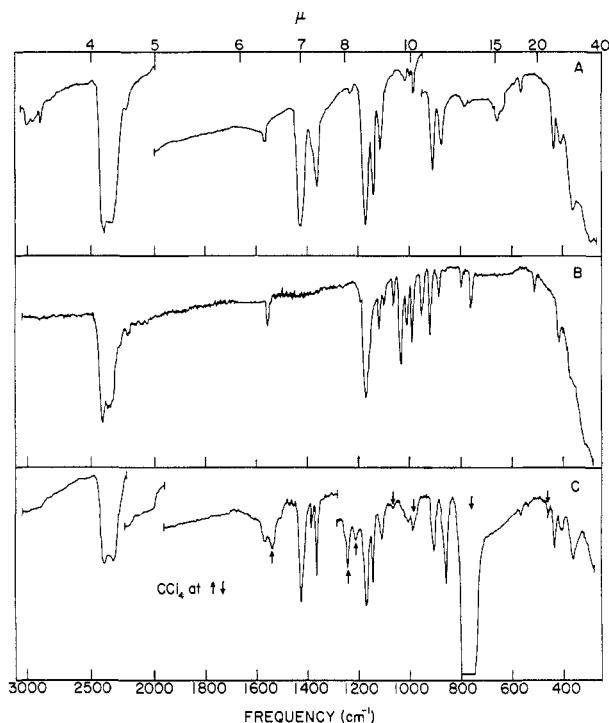
Figure 6.—Infrared spectra of solid films of products for the reactions: (A) $H_3B_3O_3$ + $(CH_3)_2N_2$ at $\sim5°K$; (B) $H_3B_3O_3$ + $(CD_3)_2N_2$ at $\sim5°K$; (C) $H_3B_3O_3$ + $(CH_3)_2N_2$ in CCl_4 matrix at 78°K.
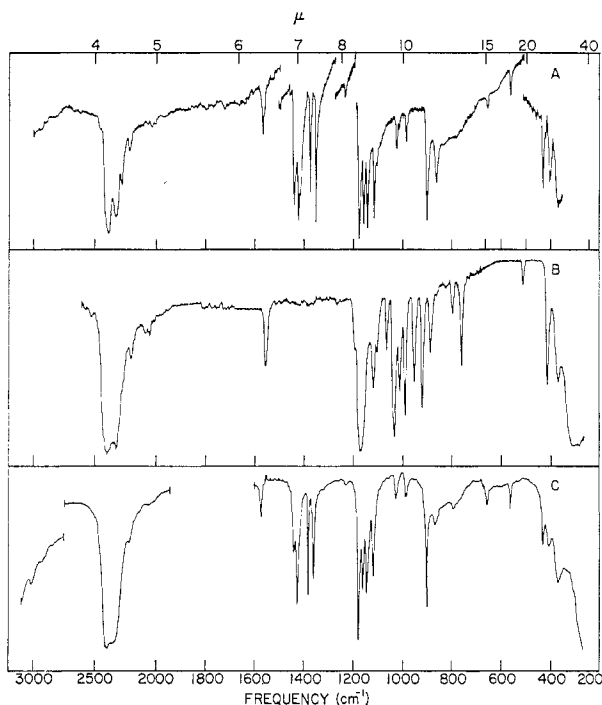
Figure 7.—Infrared spectra of solid films of products for the reactions: (A) $H_3B_3O_3$ + $(CH_3)_2N_2$, at 78°K (warmed from 5°K); (B) $H_3B_3O_3$ + $(CD_3)_2N_2$, at 78°K (warmed from 5°K); (C) $H_3B_3O_3$ + $(CH_3)_2N_2$, at about 230°K, probably liquid.

zation. The mass spectra are additionally complicated since fragmentation by loss of a single methyl group from the parent is not observed. The appearance of ion species like CH_3BH^+ shows that the molecule is undergoing extensive rearrangement on electron impact. From the evidence obtained the empirical formula $R_3N_3BH_3$ is proposed for the reaction products. Hence, the net reaction can be written as

$$H_3B_3O_3 + R_2N_2 \longrightarrow H_3BN_2R_2 + B_2O_3 \qquad (1)$$
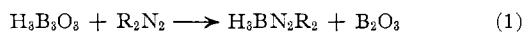
The following structures may be proposed for the products
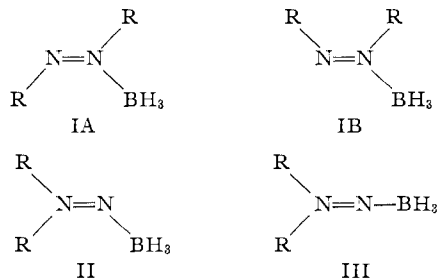
The proton nmr spectrum for the azoethane derivative indicates the C_2H_5 groups to be in nonequivalent sites. The same conclusion with respect to the CH_3 groups can be drawn for the azomethane derivative in TMS solution (Figure 2). The low-temperature broadening of the proton resonance from $H-^{11}B$ in pure $(CH_3)_2N_2BH_2$ may be due to unresolved coupling of the hydrogen nuclei with neighboring BH_3 and/or CH_3 groups. On the basis of the above, highly symmetric structures such

as III can be eliminated. The $H-^{11}B$ coupling constant of about 100 cps is typical of tetrahedrally coordinated boron.[12] The ^{11}B chemical shift (35.2 ppm from B-$(OC_2H_5)_3$) is similar to that observed for the pyridine–borine and dimethylamine–borine addition compounds (31.4 and 32.8-ppm, respectively).[12]

Configurations IA, IB, and II would probably have a plane of symmetry through the heavy atoms resulting in structures with C_s as the highest symmetry group. The $(CH_3)_2N_2BH_3$ molecule would then have 36 fundamental vibrations, 22 A′ and 14 A″, all ir and Raman active. Assignments of the vibration frequencies are given in Table II. These include approximately 26 fundamentals. Broad bands observed in the gas spectra near 1580, 1400, 1180, and 900 cm.$^{-1}$ (Figure 5) are resolved in the low-temperature film spectra (Figure 6). Other lines, very weak in the gas spectra, become readily distinguishable in the solid. Splittings of the absorption lines of the warmed solid film were observed. These are probably due to removal of nearly degenerate BH_3 and CH_3 stretching and deformation modes by the perturbing effect of the annealed film. By comparing the spectrum of methyl deuterated species with that of the normal compound (Figure 7) in the region between 1200 and 1100 cm^{-1} the splittings of the BH_3 deformation and CH_3 rocking modes and the B–N stretching mode were identified. The broad band at 1583 cm^{-1} persists in the isotopically labeled species of the azomethane derivative. By analogy with other $N=N$ bonded systems,[13,14] the band is assigned to an $N=N$ stretching vibration. Most of the bands can be assigned to simple monomeric species and there appears to be no evidence of

bridge coupling in the solid phase, in a CCl_4 matrix, or in the liquid at −45°. Structure II cannot be ruled out by the observations, but this would require an unusual electron distribution within the molecule. Thus it appears from the present evidence that $R_2N_2BH_3$ is best represented by structure I, although the question of *cis vs. trans* configuration remains to be settled.

It is reasonable to expect that in its reactions with azoalkanes boroxine behaves as a source of BH_3 as in reactions with PF_3 and CO. However the reactions with azoalkanes are fast and the reaction mechanism may be different.

At ordinary temperatures the boroxine–azomethane derivative decomposes rapidly. In the gas phase the compound lasts usually less than 1 hr in an infrared cell fitted with Teflon seals. The compound is destroyed more rapidly in vessels containing greased joints or stopcocks. A gaseous product remaining in the infrared cell has been identified as tetramethyl-

(12) W. D. Phillips, H. C. Miller, and E. L. Muetterties, *J. Am. Chem. Soc.*, **81**, 4496 (1959).
(13) M. N. Ackermann, J. E. Ellenson, and D. H. Robinson, *ibid.*, **90**, 7173 (1968).
(14) R. W. Mitchell and J. A. Merritt, *J. Mol. Spectry.*, **27**, 197 (1968).

TABLE II

INFRARED SPECTRA OF DEUTERIUM-LABELED PRODUCTS OF THE BOROXINE–AZOMETHANE
REACTION (HEADINGS INDICATE LABELING SITES) (CM^{-1})

Gas phase			Liq He film		Liq N$_2$ film		
B–H, C–H	B–D, C–H	B–H, C–D	B–H, C–H	B–H, C–D	B–H, C–H	B–H, C–D	Assignment
3027 m	3020 m		3020 vw		3025 vw		CH$_3$ asym str
2934 w	2930 m		2970 vw		2955 vw		CH$_3$ sym str
2851 vw	2855 m		2910 vw		2910 vw		
2441 vs		2441 vs	2410 vs	2415 vs	2415 / 2405 vs / 2390	2415 vs	BH$_3$ asym str
	2412 w, br						
2364 vs		2380 vs	2340 vs	2375 vs / 2354 vs	2335 vs	2385 vs / 2340 vs	BH$_3$ sym str
		2340 s, sh			2330 vs		CD$_3$ asym str
					2306 m	2300 s	CD$_3$ asym str
		2285 m, sh		2275 m	2290 m	2280 s	CD$_3$ sym str
2228 vw		2231 vw	2230 vw		2230 w		
				2210 w		2210 w	
		2110 vw		2110 vw		2110 vw	CD$_3$ str
		2030 vw		2060 vw		2060 vw	CD$_3$ str
	1822 vs						BD$_3$ asym str
	1770 s						BD$_3$ sym str
	1705 vw, br						
1583 m	1575 w	1585 / 1570 m / 1557	1580 w	1560 m	1580 w	1560 s	N=N str
1445 m	1429 s		1435 vs		1451 s / 1435 / 1425 m		CH$_3$ asym def
1394 w, sh	1380 m, sh		1370 m		1384 s / 1360 vs		CH$_3$ sym def
1379 w, sh	1379 w, sh						
	1205 vw		1240 vw	1196 w	1242 w	1195 m	C—N asym str
1184 s (1189 s / 1179 s)		1184 s (1189 s / 1179 s)	1183 s	1173 vs	1184 s / 1170 s	1173 vs / 1158 vs	BH$_3$ def
1160 / 1148 m	1130 vw		1150 s		1152 s		CH$_3$ rock
			1120 m	1122 m	1123 s / 1110	1130 s / 1126 w	B—N str
				1104 w		1106 m	CD$_3$ asym def
						1064 s	
				1066 m / 1036 s		1048 s / 1032 vs	CD$_3$ asym def
1040 vw	1045 vw		1022 w	1013 m	1030 / 1020	1005	CN sym str
988 vw	988 vw		990 w		993 m		CH$_3$ rock
			992 s		1005 s (?) / 989 vs		CD$_3$ sym deform
			954 m		952 s		CD$_3$ rock
		938 w					
916 m / 907 m (913)		900 (buried under 938 sh)	908 m	922 s	908 m	920 s	BH$_3$ def
	885 (890 s / 880 s)						BD$_3$ def
872 w			875 m	802 w	875 m	800 w	CH$_3$ rock
		775 w, br		763 m		761 s	CD$_3$ rock
	722 w						BD$_3$ def
	560 vw		567 vw		565 m		CH$_3$–N def
		512 w		516 m		514 m	CD$_3$–N def
			437 m	417 m	439 m	416 s	BH$_3$–N def (?)
			405 m		410 m		CH$_3$–N def
		390 w		370 s		374 s	CD$_3$–N def
370 w	370 w		362 w		370 w		
		335					CD$_3$ torsion

hydrazine. Diborane reacts with $(CH_3)_2N_2BH_3$ to form nonvolatile solids and a permanent gas. For this reason it is important that an excess of azomethane be used in the reaction with boroxine. Small quanti-

ties of the compound in the liquid phase decompose very rapidly at room temperature to form a yellowish polymeric substance. *It should be cautioned that sizable quantities of the liquid may be spontaneously explosive.*