in a 2-l. flask and heated at 110° for 12 hr. and then at 190–200° for 12 hr. The contents of the flask were fractionated through traps at −80 and −190°. A considerable amount of non-condensable gas (H_2 and/or CH_4) was present as well as yellow non-volatile solids. The material trapped at −190° (*ca.* 20 mmoles) consisted largely of unidentifiable compounds as well as traces of 1-methylpentaborane. No detectable amount of methyl chloride was present as determined by infrared. The material trapped at −80° (6 mmoles) was identified as a mixture of 2-methylpentaborane (*ca.* 85%) and 1-methylpentaborane (*ca.* 15%) by infrared[3a,8] and B^{11} n.m.r. spectra (*vide supra*).

2-Ethylpentaborane from Pentaborane.—Using ethylene (15 mmoles) for the alkylating agent instead of methyl chloride, the same reaction conditions, amounts of materials, and work-up procedure were used as described above for the preparation of 2-methylpentaborane from pentaborane. A gas chromatogram of the products trapped at −80° gave 2-ethylpentaborane, $R_v = 3.2$, (1 mmole) with no evidence of 1-ethylpentaborane. The infrared spectrum of 2-ethylpentaborane is very similar to that of 2-methylpentaborane.[8] The B^{11} n.m.r. spectrum consisted of a low field singlet, a low field doublet, and a high field doublet in an estimated area ratio of 1:3:1 (*vide supra*).

1,2-Dimethylpentaborane.—A sealed flask containing 2-methylpentaborane (2 mmoles), methyl chloride (2 mmoles), and freshly sublimed aluminum chloride (*ca.* 0.1 mmole) was heated for 4 hr. at 100°. After fractionating the contents of the flask through −90 and −190° baths the material trapped at −90° was gas chromatographed on the Kel-F column. About 0.4 mmole (20% yield) of 1,2-dimethylpentaborane, m.p. −80° to −78°, and 0.25 mmole of unreacted 2-methylpentaborane were isolated. The infrared spectrum of 1,2-dimethylpentaborane appeared to be nearly a composite of 1-methylpentaborane[3a] and 2-methylpentaborane.[8] The B^{11} n.m.r. spectrum consisted of a low field singlet, low field doublet, and high field singlet in an estimated area ratio of 1:3:1 (*vide supra*).

Acknowledgment.—The authors wish to express their gratitude to the National Science Foundation for financial support of this research. They also thank Tony Byrnes for running many of the B^{11} n.m.r. spectra.

Contribution from the Evans Laboratory of Chemistry,
The Ohio State University, Columbus 10, Ohio

Boron Heterocycles. I. Preparation and Properties of 1,3,2-Dioxaborolane

By S. H. Rose and S. G. Shore

Received June 21, 1962

1,3,2-Dioxaborolane, $(CH_2O)_2BH$, has been prepared through the reaction of $HOCH_2CH_2OH$ with B_2H_6 in $(C_2H_5)_2O$. It is essentially monomeric in the vapor phase. In the liquid state it is a viscous material which is rapidly converted to a non-volatile, glassy solid at room temperature. In the vapor phase disproportionation occurs reversibly.

$$(CH_2O)_2BH(g) \rightleftharpoons \frac{1}{6}B_2H_6(g) + \frac{1}{3}(CH_2O)_2BOCH_2CH_2OB(OCH_2)_2(s)$$

The equilibrium constant at 25° is 25 ± 3. This boron heterocycle is a weak Lewis acid.

Introduction

The general tendency for mixed alkoxyboranes to disproportionate to form more symmetrical species is well known. Mixed trialkoxyboranes, $(RO)_2BOR'$, equilibrate so rapidly with disproportionation products that they cannot be isolated in the pure state in general.[1] Dialkoxyboranes also disproportionate, though less readily. The disproportionation of dimethoxyborane is an example.[2]

$$(CH_3O)_2BH(g) \rightleftharpoons \frac{1}{6}B_2H_6(g) + \frac{2}{3}B(OCH_3)_3(g) \quad (1)$$

Since the heterocyclic analogs of the acyclic alkoxyboranes have been reported to show less tendency to disproportionate,[1] it is of interest to know the properties of the cyclic counterpart of dimethoxyborane, 1,3,2-dioxaborolane, $(CH_2O)_2BH$. The only reference to this compound in the available literature appears as a very brief description given by Schlesinger and Burg,[3] who synthesized it through the reaction of ethylene glycol with diborane.

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[1] J. A. Blau, W. Gerrard, and M. F. Lappert, *J. Chem. Soc.*, 4116 (1957).

[2] A. B. Burg and H. I. Schlesinger, *J. Am. Chem. Soc.*, **55**, 4020 (1933).

[3] H. I. Schlesinger and A. B. Burg, *Chem. Rev.*, **31**, 1 (1942).

In this investigation we have examined the reaction between ethylene glycol and diborane as a route to the synthesis of 1,3,2-dioxaborolane, and have studied properties of the resulting compound.

Experimental

A. **Materials.**—(1) B_2H_6 was prepared and purified according to well known methods.[4] (2) Reagent grade $HOCH_2CH_2OH$ was dried over N,a[5] fractionally distilled under dry N_2, and stored in sealed ampoules. (3) Reagent grade NH_3 was dried over sodium at −78°. (4) $N(CH_3)_3$ was dried over CaH_2 and distilled *in vacuo*. (5) $(C_2H_5)_2O$ and tetrahydrofuran were dried over $LiAlH_4$.

B. **Apparatus.**—(1) Standard high vacuum techniques were used for handling volatile materials. (2) Mass spectra were obtained on a Consolidated Engineering Model 21-620 mass spectrometer. (3) Infrared gas spectra were obtained on a Perkin-Elmer Model 21 spectrometer. The instrument was frequency calibrated with polystyrene.

C. **Synthesis of 1,3,2-Dioxaborolane. 1. Preparation of the $N(CH_3)_3$ Adduct.**—A hypodermic syringe was used to add 50 mmoles of $HOCH_2CH_2OH$ through a serum bottle stopple into a reaction tube containing a dry N_2 atmosphere. The tube then was connected to the vacuum apparatus and evacuated. A 150-ml. quantity of anhydrous $(C_2H_5)_2O$ was distilled into the tube and condensed at −196°. The mixture, which was continuously stirred by an electromagnetically operated spiral stirrer, was allowed to warm to room temperature and then was rapidly cooled to −196° to form a suspension of $HOCH_2CH_2OH$ in $(C_2H_5)_2O$. A 40-mmole quantity of B_2H_6 was distilled into the tube and the tube was warmed to −78°. A reaction commenced, producing H_2, which agitated the suspension. When the rate of H_2 evolution became negligible, the temperature was raised to −40°, increasing the speed of the reaction. Again, when the rate of H_2 production became negligible, the temperature was raised slowly to 20°. After the reaction appeared to have stopped at this temperature, excess $N(CH_3)_3$ was distilled into the reaction tube. The contents of the tube were stirred continuously and maintained at −78° for 2 hr. A white solid formed gradually. To ensure complete reaction, stirring was carried out for an additional 2 hr. at −45°. The bulk of the $(C_2H_5)_2O$ then was distilled from the tube at −63.5°, with the last traces being removed by pumping on the system at −50°. Trimethylamine-1,3,2-dioxaborolane is appreciably more volatile than trimethylamineborane and therefore was removed from the reaction tube through sublimation at room temperature. A typical yield of $(CH_2O)_2BHN(CH_3)_3$ was about 60% based upon the starting amount of $HOCH_2CH_2OH$.

2. **Exchange Reaction with Diborane.**—Ten mmoles of $(CH_2O)_2BHN(CH_3)_3$ was sublimed into a 1-l. pear-shaped flask which terminated in a test tube bottom, suitable for immersion in a dewar flask. Thirty mmoles of B_2H_6 was distilled into the exchange vessel and the temperature of the bath around the test tube bottom was gradually raised to 50°. Crystals of $H_3BN(CH_3)_3$ formed on the sides of the vessel and a gradual increase in pressure was noted as the exchange reaction progressed. After 1.5 hr., the gas in the exchange vessel was passed slowly through a condensation train containing cold baths at −35, −120, and −196°. 1,3,2-Dioxaborolane was trapped in the center trap, and unreacted B_2H_6 and amine adduct were distilled back into the exchange vessel, where the procedure was repeated. After several such exchanges a yield of 7 mmoles of $(CH_2O)_2BH$ had been obtained.

In order to minimize conversion of 1,3,2-dioxaborolane to the non-volatile glassy state it was stored at −78° or lower. When some stored sample was required, it was evaporated by immersion of the storage trap in warm water at about 40°.

Anal. Calcd. for B, 15.0; H (hydridic), 1.40. Found: B, 15.0; H (hydridic), 1.42.

D. **Ethylene Glycol–Diborane Reaction.**—This reaction was carried out in the same manner as described in detail above for the synthesis of 1,3,2-dioxaborolane except on approximately one-tenth scale with respect to solvent and diborane used. The reaction was monitored by measuring H_2 evolution. For a mole ratio of reactants of $2\text{-}HOCH_2CH_2OH/B_2H_6$, H_2 was produced in the ratio of $H_2/\text{-}OH$. After H_2 evolution had ceased, the solvent and volatile solute were distilled from the reaction system, leaving behind a glass residue which was found to be free of hydridic hydrogen and was identified as $(CH_2O)_2BOCH_2CH_2OB(OCH_2)_2$ through its melting point[1] and the determination of its equivalent weight by potentiometric titration with NaOH in the presence of mannitol.

Anal. Calcd. for $(CH_2O)_2BOCH_2CH_2OB(OCH_2)_2$: equiv. wt., 202.0. Found: equiv. wt., 202.

NH_3 was added to the distillate at −78° to form nonvolatile adducts with the solute. Solvent $(C_2H_2)_5O$ then was distilled from the system and the remaining solid was extracted with fresh $(C_2H_5)_2O$. The extract was shown to be H_3NBH_3 from its X-ray powder diffraction pattern,[6] indicating that the ether distillate contained B_2H_6 before the NH_3 was added, thus providing evidence for the decomposition of $(CH_2O)_2BH$ in solution since the original B_2H_6 in the reaction had been expended according to the H_2 evolved. Through continuous extraction of H_3NBH_3, the composition of the ether-insoluble residue approached a composition of 90–95% $(CH_2O)_2BHNH_3$.

E. **Properties of 1,3,2-Dioxaborolane.**—(1) Molecular weights were determined from vapor densities determined at about 40 mm. pressure at room temperature.

Anal. Calcd. for $(CH_2O_2)BH$: mol. wt., 71.9. Found: mol. wt., 73.2, 74.3, 73.3, 74.9, 75.7.

(2) Mass-to-charge ratios in the mass spectrum of $(CH_2O)_2BH$ (Table I) were assigned on the basis of a calibration against the mass spectrum of 2,2-dimethylbutane, which has strong peaks at m/e of 71, 43, 29, and 15. In the mass spectrum of $(CH_2O)_2BH$ there is some uncertainty in the peaks at m/e of 27 and 26, since these are strong peaks in the diborane mass spectrum, and a small amount of diborane impurity is likely to occur through disproportionation of the heterocycle.

The mass spectrum of $(CH_2O)_2BOCH_2CH_2OB(OCH_2)_2$ obtained as the disproportionation product of $(CH_2O)_2BH$ showed a high peak at m/e of about 200. The formula weight for this structure is 202. This peak could not be

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(4) I. Shapiro, H. G. Weiss, M. Schmich, S. Skolnik, and G. B. L. Smith, *J. Am. Chem. Soc.*, **74**, 901 (1952).

(5) K. L. Reinhart and E. G. Perkins, *Org. Syn.*, **37**, 42 (1957).

(6) S. G. Shore and R. W. Parry, *J. Am. Chem. Soc.*, **77**, 6084 (1955).

Table I

Mass Spectrum of 1,3,2-Dioxaborolane

m/e	Relative abundance
72	40.6
71	96.6
70	20.3
45	16.3
44	8.7
43	74.7
42	100.7
41	26.5
40	7.8
29	17.6
28	9.4
27	10.9
26	5.5
15	49.2
14	12 1

attributed to Hg^{+1} since it was appreciably larger than the normal Hg^{+1} background. Peaks also were observed at *m/e* of 100, 93, 90, and 80. This compound is difficultly volatile at room temperature so that only the strongest peaks in the mass spectrum were detectable. Using a Consolidated Engineering micromanometer, its vapor pressure was found to be $(2.7 \pm 0.5) \times 10^{-3}$ mm. at 25°.

(3) Infrared spectra of $(CH_2O)_2BH$ (Table II) and $(CH_2O)_2BHN(CH_3)_3$ were obtained in the vapor phase at room temperature. The weak bands reported in Table II at 1618, 1603, and 1585 cm.$^{-1}$ probably are due to a small amount of diborane impurity, since their intensities increased with increasing time, which is consistent with the disproportionation of the heterocycle to produce diborane. Furthermore these bands are so intense in the diborane spectrum[7] that they could be detected in a 10-cm. cell containing diborane at a pressure of less than 1 mm. The infrared spectrum of the vapor above the solid $(CH_2O)_2BHN(CH_3)_3$ appeared to be a composite of the spectra of pure $(CH_2O)_2BH$ and pure $N(CH_3)_3$.

Table II

Infrared Absorption Peaks of 1,3,2-Dioxaborolane[a]

cm.$^{-1}$	Intensity[b]			cm.$^{-1}$	Intensity[b]		cm.$^{-1}$	Intensity[b]		
2967	ms	sp		1475	m		926	ms		
2915	ms	sp		1404	s		908	ms		
2801	w			1310	mw		893	s	sp	
2591	s	sp		1224	vs	B	877	m		
2463	w			1170	vs		766	mw		B
2128	w			985	ms		758	mw		
1618	w		B	975	ms		751	mw		
1603	w			941	ms		743	mw		
1585	w									

[a] 10-cm. cell at 40 mm. pressure. [b] v, very; s, strong; m, medium; w, weak; b, broad; sp, sharp.

(4) Vapor pressure measurements were made in a way which minimized complications from glass formation and disproportionation of $(CH_2O)_2BH$. A freshly purified sample was used for each datum point, and after each vapor pressure determination possible volatile decomposition products were removed by flaming the apparatus under high vacuum. The vapor pressure apparatus consisted of a U-tube connected to a manometer. Two sets of data were obtained. One set represented data from samples which were allowed to warm from −196° to the temperature of the thermostating bath. The other set represented data from samples which were cooled from room temperature to the temperature of the thermostating bath. In both cases the pressures were read within about 3 min. after placing the thermostating bath under the trap. For vapor pressure determinations above 0° only the warm-up procedure was used as the cool-down procedure presented the possibility of disproportionation in the vapor phase, and the cool-down procedure minimized the possibility of glass-formation. Vapor pressures were obtained over the temperature range 250.2 to 283.6°K. A plot of log p *vs.* $1/T$ was linear in this range, allowing the data to be reduced to the equation: $\log p_{mm} = (-2.40 \times 10^3/T) + 10.01$. The heat of vaporization was found to be 11.0 ± 0.1 kcal./mole.

(5) The stoichiometry of the vapor phase disproportionation of $(CH_2O)_2BH$ was determined through the following procedure. A measured quantity of freshly purified $(CH_2O)_2BH$ was distilled into a tube which was equipped with a break-off tip. The tube was sealed off and the heterocycle was allowed to disproportionate over a period of several weeks at 25°. The tube then was returned to the vacuum system, and its volatile contents were separated by fractionl distillation, measured volumetrically, and positively identified mass spectrometrically. Results are presented in Table III. The data show that $^1/_6$ mole of B_2H_6 was formed for each mole of $(CH_2O)_2BH$ which disproportionated.

Table III

Disproportionation Data

No.	mmoles $(CH_2O)_2BH$ added	mmoles B_2H_6 recovered	$6B_2H_6$ recovered	+ $(CH_2O)_2$-BH recovered	= mmoles $(CH_2O)_2BH$ added
1	0.579	0.067	0.402	0.16	0.56
2	.543	.063	.378	.14	.52

(6) The equilibrium constant for the disproportionation reaction was determined from the disproportionation reaction data. Since the volumes of the tubes had been calibrated, it was possible to calculate the equilibrium partial pressures of B_2H_6 and $(CH_2O)_2BH$ and in turn calculate the equilibrium constant for the reaction. The decomposition tubes were thermostated at 25° and were opened after intervals ranging from 2.5 to 6 weeks. The equilibrium data are given in Table IV below. For the heterogeneous equilibrium

$$K_{eq} = \frac{P_{(B_2H_6)}{}^{1/6}}{P_{(CH_2O)_2BH)}} \qquad P = \text{partial pressure in atm.}$$

Table IV

Equilibrium Data

No.	Time (weeks)	Pressure $(CH_2O)_2BH$ (atm.)	Pressure B_2H_6 (atm.)	$(P_{B_2H_6})^{1/6}$	K_{eq}
1	2.5	0.019	0.0077	0.45	24 ± 2
2	6	.017	.0069	.44	26 ± 2

For the homogeneous equilibrium

$$K_{eq} = \frac{(P_{B_2H_6})^{1/6}(P_{(CH_2O)_2BOCH_2CH_2OB(CH_2O)_2})^{1/3}}{(P_{(CH_2O)_2BH})}$$

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[7] W. C. Price, *J. Chem. Phys.*, **16**, 894 (1948).

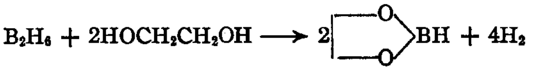
Using a partial pressure of (2.7 ± 0.5) × 10^{-3} mm. for $(CH_2O)_2BOCH_2CH_2OB(CH_2O)_2$, K_{eq} = 0.38 ± 0.07.

F. Lewis Base Adducts of 1,3,2-Dioxaborolane.—(1) Trimethylamine-1,3,2-dioxaborolane was prepared in ether according to the procedure described in section C. The direct reaction between $(CH_2O)_2BH$ and $N(CH_3)_3$ in the liquid state at −78° produced the adduct in about 80% yield, based upon $N(CH_3)_3$ recovered. This compound could not be quantitatively sublimed, owing to the fact that it was essentially completely dissociated in the vapor phase and the heterocycle was slowly undergoing disproportionation. The sublimate always contained small amounts of $H_3BN(CH_3)_3$.

(2) Ammonia-1,3,2-dioxaborolane was prepared, in a typical reaction, by condensing 0.57 mmole of $(CH_2O)_2BH$ onto a broad area of a 25-mm. diameter reaction tube, condensing 2.00 mmoles of ammonia above the heterocycle, and thermostating the system at −78° for about 10 hr. The tube was allowed to warm slowly to room temperature and the excess NH_3 was removed and measured, indicating that 0.56 mmole of ammonia had reacted with the heterocycle. The ammonia adduct is a non-volatile solid which shows no sign of decomposing at room temperature *in vacuo*.

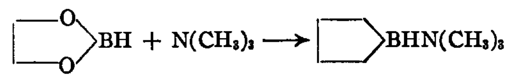
Results

The synthesis and isolation of 1,3,2-dioxaborolane can be summarized through the reaction sequence



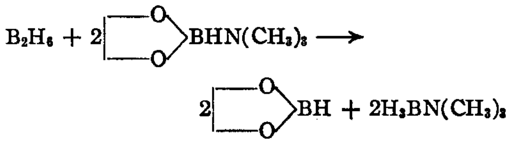
(2)

Separation of the product from solvent ether was achieved through the addition of trimethylamine to the system, followed by distilling away the solvent.



(3)

The free heterocycle was regenerated from the sublimed amine adduct through an exchange reaction which took advantage of the fact that diborane is a stronger Lewis acid than the heterocycle.



(4)

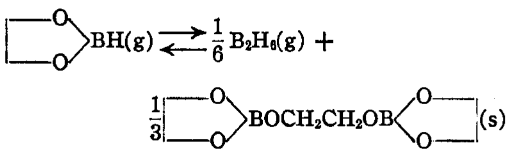
In the ethylene glycol–diborane reaction in diethyl ether, 1 mole of hydrogen was evolved for each OH group. 1,3,2-Dioxaborolane was produced in the range 2/1 to 1/1 glycol-to-diborane ratio. The heterocycle disproportionated in solution to form diborane and 2,2′-(ethylenedioxy)-bis-(1,3-2-dioxaborolane), $(CH_2O)_2BOCH_2CH_2OB(CH_2O)_2$. Tetrahydrofuran as a solvent did not minimize this disproportionation. Noticeable amounts of (ethylenedioxy)-bisborane, $BH_2OCH_2CH_2OBH_2$, were not detected in the ratio 1/1 glycol-to-diborane. The stable chloro analog, $BCl_2OCH_2CH_2OBCl_2$, has been reported,[8] however.

The very brief description of 1,3,2-dioxaborolane given by Schlesinger and Burg[3] is consistent with the results of this investigation.

The heterocycle is essentially monomeric in the vapor phase, showing an average molecular weight of 74.3 ± 1.2 and a parent mass of 72 (Table I) in its mass spectrum. The infrared spectrum shows that the B–H stretching frequency appears as a sharp band at 2591 $cm.^{-1}$, which is a reasonable value for a terminal B–H bond.[9]

At room temperature, $(CH_2O)_2BH$ is a viscous liquid which shows a pronounced tendency to form a stable, hard, colorless, glassy solid which has no measurable vapor pressure, thereby requiring a special procedure for the study of the liquid–vapor equilibrium. The volatile heterocycle can be regenerated by warming this solid; however, heating above 50° causes extensive disproportionation.

In the vapor phase, this reversible disproportionation occurs



(5)

The equilibrium constant for this heterogeneous reaction is 25 ± 3 at 25°, indicating that the standard free energy of disproportionation, ΔF^0_{298}, is −1.9 ± 0.1 kcal./mole. The equilibrium constant for the homogeneous gas phase reaction is K_{eq} = 0.38 ± 0.07, ΔF^0_{298} = 0.6 ± 0.1 kcal./mole.

The initial rate of disproportionation of the heterocycle seems to depend upon the presence of trace impurities. Some samples disproportionated at an initial rate of close to 1%/min. at 25°. Other samples which were carefully purified and handled in a new vacuum system that had not been previously exposed to such compounds disproportionated much more slowly. A typical example of disproportionation in a clean system is given in Table V.

The effect of trace impurities on the rate of dis-

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[8] J. A. Blau, W. Gerrard, and M. F. Lappert, *J. Chem. Soc.*, 667 (1960).

[9] L. J. Bellamy, W. Gerrard, M. F. Lappert, and R. L. Williams, *ibid.*, 2412 (1958).

TABLE V

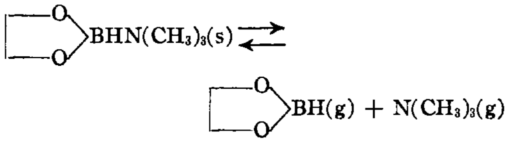
RATE OF DISPROPORTIONATION OF 1,3,2-DIOXABOROLANE[a]

Time, hr.	0	3	17	20	41	51	66	76
Total pressure, mm.	31.6	31.6	29.0	28.6	25.1	24.3	23.0	22.8

[a] Temperature, 25°; volume, 192 ml.

proportionation has been noted for $(CH_3O)_2BH$.[3,10]

1,3,2-Dioxaborolane forms addition compounds with ammonia and trimethylamine which are solids at room temperature. At 25° the ammonia adduct has no detectable vapor pressure, while the trimethylamine adduct has a dissociation pressure of about 8 mm. The vapor above the solid trimethylamine adduct consists essentially of pure components. The B–H stretching frequency observed in this vapor indicates no quaternization of the boron.

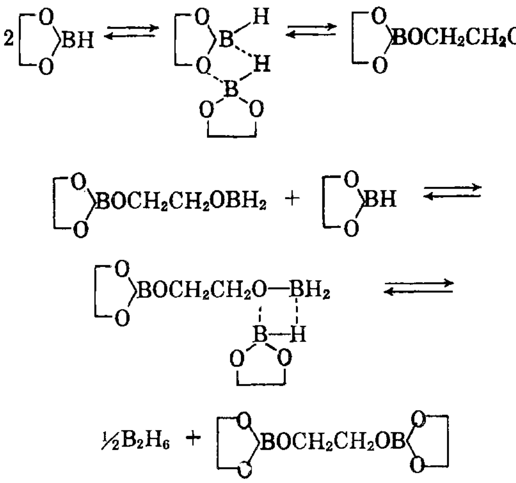


(6)

Discussion

With respect to apparent ease of disproportionation, 1,3,2-dioxaborolane does not seem to be more stable than its acyclic counterpart, dimethoxyborane. $\Delta F_{298}^0 = -1.9 \pm 0.1$ kcal./mole for the disproportionation of the heterocycle (eq. 5), while $\Delta F^0_{298} = -0.41 \pm 0.01$ kcal./mole for the disproportionation of dimethoxyborane[11] (eq. 1). Although these two equilibria represent reactions experimentally observed under similar conditions of temperature and pressure, they are not equivalent in that a heterogeneous, vapor–solid equilibrium is involved in the case of the heterocycle, whereas a homogeneous, vapor equilibrium is involved in the case of the dimethoxyborane. To compare thermodynamic stabilities, the homogeneous gas phase disproportionation of 1,3,2-dioxaborolane must be considered. Since $\Delta F^0_{298} = 0.6 \pm 0.1$ kcal./mole for the homogeneous decomposition, it is seen that the heterocycle is thermodynamically more stable than dimethoxyborane, by about 1 kcal./mole.

A possible mechanism or reaction scheme which has been proposed for the disproportionation of dimethoxyborane[10] and analogous systems[12] could be considered in this case also. It involves the formation of a four-center bridge complex in the transition state, which could lead to rearrangement.



(7)

If such a scheme or sequence is operative, the equilibrium concentration of $(CH_2O)_2BOCH_2CH_2CH_2OBH_2$ is small since this species was not detected among the equilibrium disproportionation products of $(CH_2O)_2BH$. Furthermore, the disproportionation data given in Table III are in good agreement with the stoichiometry given by eq. 5.

The essentially monomeric character of 1,3,2-dioxaborolane in the vapor phase is consistent with the rationale that the electron deficiency of boron is sufficiently relieved through back or π-bonding with oxygen that hydrogen bridge formation does not occur in significant concentration.[13] The weak Lewis acid character of this heterocycle, as demonstrated through the essentially complete vapor phase dissociation of the trimethylamine adduct, also is consistent with this rationale.

Acknowledgment.—We wish to acknowledge, gratefully, the support of this work by the National Science Foundation and the Research Corporation.

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[10] H. S. Uchida, H. B. Kreider, A. Murchison, and J. F. Masi, *J. Phys. Chem.*, **63**, 1414 (1959).

[11] Taken from the value 63 ± 5 for the equilibrium constant at 25° for the equation

$$6(CH_3O)_2BH(g) \longrightarrow B_2H_6(g) + 4B(OCH_3)_3(g)^3$$

[12] A. Finch, J. C. Lockhart, and J. Pearn, *J. Org. Chem.*, **26**, 3250 (1961).

[13] F. G. A. Stone, *Chem. Rev.*, **58**, 101 (1958).