of 2,3-$C_2B_4H_8$; 1,7 positions of 2,4-$C_2B_5H_7$;[17] B_5H_9[18] > 1 position of 2,3-$C_2B_4H_8$. The relative unreactivity of the bridge positions in 2,3-dicarbahexaborane(8) toward an exchange of this nature is analogous to the previously reported observations for the pentaborane–deuteriodiborane exchange.[18] However, unlike pentaborane, the basal positions of 2,3-dicarbahexaborane(8) exchange much more readily than the apex.

Acknowledgment.—The authors wish to express their gratitude to the Office of Naval Research for financial support of this research.

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[18] W. S. Koski, J. J. Kaufman, and P. C. Lauterbur, *J. Am. Chem. Soc.*, **79**, 2382 (1957).

CONTRIBUTION FROM SHIONOGI RESEARCH LABORATORY, SHIONOGI & CO., LTD.,
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Borazines Stable to Hydrolysis

BY KOICHIRO NAGASAWA

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In order to evaluate the steric effect of increasing the stability of the borazine ring, B-tri-2,6-xylyl- and B-trimesityl-N-trimethylborazine were prepared and subjected to Friedel–Crafts acetylation, free-radical bromination, and hydrolysis experiments. The corresponding acetyl and bromo derivatives were obtained in good yields, and no hydrolysis was observed even in acidic and basic solution, indicating the remarkable enhancement of the ring stability by the *o*-methyl groups on the aryl substituents.

The lack of hydrolytic stability of covalent boron–nitrogen bonds has been an obstacle in the investigation of various reactions and application of boron–nitrogen compounds and has been referred to in many reports to show the authors' concern.[1,2] It has previously been shown in this laboratory[3] that the resonance effect in ethynylborazine derivatives can deter the hydrolysis to some extent but is not yet sufficient for thermodynamical stabilization. Attempts to stabilize the borazine ring toward hydrolysis by introduction of various substituents in aryl groups were not successful[4] until bulky substituents were placed in the *ortho* positions of B-aryl groups. The present work was undertaken to prepare borazine derivatives highly stabilized by the steric effects of substituents.

Brown, *et al.*,[5] have shown that triarylboranes are far less reactive toward oxygen and water when the vacant p orbital of the central boron atom is completely shielded by bulky substituents. Hawkins, *et al.*,[6] could enhance the hydrolytic stabilities of the arylboron compounds by introducing bulky substituents onto the *ortho* positions of the aryl groups. Such studies led us to infer that the use of the 2,6-xylyl group as a substituent would stabilize the borazine ring to hydrolysis.

B-Tri-2,6-xylyl-N-trimethylborazine (I) and B-trimesityl-N-trimethylborazine (II), shown in Figure 1, were prepared by treating B-trichloro-N-trimethylborazine with 2,6-xylyl- and mesitylmagnesium bromide, respectively, in the same way as described in the literature.[7] The yields and analytical data are summarized in Table I. In an attempt to prepare N-tri-2,6-xylylborazines, the position isomers of I, the reaction of *m*-xylidine with boron trichloride was carried out in dry toluene. The reaction mixture evolved hydrogen chloride on refluxing giving, contrary to expectation, the corresponding aminoborane instead of the borazine derivative.

Examination of molecular models of I and II suggests that the three aromatic groups on the boron atoms are forced to be perpendicular to the plane of the borazine ring by the steric effect. This was, in fact, supported by the ultraviolet and proton magnetic resonance spectra (Table II). Becher, *et al.*,[8] have shown from the ultraviolet spectra of some arylborazines that the conjugation between the phenyl groups and the borazine ring is negligible for B-triphenyl-N-trimethylborazine; *i.e.*, the phenyl groups are oriented at a right angle to the borazine ring. Compounds I and II displayed a $\pi-\pi^*$ transition band at almost the same wavelengths as did B-triphenyl-N-trimethylborazine; this indicates that the aryl groups are perpendicular to the borazine ring. The proton chemical shift data afford still further support to this structure.

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[1] W. Gerrard, H. R. Hudson, and E. F. Mooney, *J. Chem. Soc.*, 113 (1962); M. J. Bradley, G. E. Ryschkewitsch, and H. H. Sisler, *J. Am. Chem. Soc.*, **81**, 2635(1959); J. H. Smalley and S. F. Stafiej, *ibid.*, **81**, 582 (1959); G. E. Ryschkewitsch, *ibid.*, **82**, 3290 (1960); H. C. Kelley, F. R. Marchelli, and M. B. Giusto, *Inorg. Chem.*, **3**, 431 (1964).

[2] R. J. Brotherton and A. L. McCloskey, "Boron–Nitrogen Chemistry," Advances in Chemistry Series, No. 42, American Chemical Society, Washington, D. C., 1964, p 131.

[3] H. Watanabe, T. Totani, and T. Yoshizaki, *Inorg. Chem.*, **4**, 657 (1965).

[4] H. Watanabe, T. Totani, K. Nagasawa, T. Yoshizaki, and T. Nakagawa, unpublished work.

[5] H. C. Brown and V. H. Dodson, *J. Am. Chem. Soc.*, **79**, 2302 (1957).

[6] R. T. Hawkins, W. J. Lennarz, and H. R. Snyder, *ibid.*, **82**, 3053 (1960).

[7] G. E. Ryschkewitsch, J. J. Harris, and H. H. Sisler, *ibid.*, **80**, 4515 (1958); S. J. Groszos and S. F. Stafiej, *ibid.*, **80**, 1357 (1958).

[8] H. J. Becher and S. Frick, *Z. Physik. Chem.* (Frankfurt), **12**, 241 (1957).

TABLE I

ANALYTICAL DATA FOR B-ARYLBORAZINES

Compd. no.[a]	Yield, %	Mp, °C	% C		% H		% N		% Br or acetyl		Mol wt	
			Found	Calcd	Found	Calcd	Found	Calcd	Found	Calcd	Found	Calcd
I	50.2	282.5–283.0	74.69	74.53	8.61	8.34	9.71	9.66	...	...	461	435.1
II	38.2	274.0–276.0	75.90	75.51	9.21	8.87	8.78	8.81	...	...	486	477.1
III	48.0	196.0–198.0	70.98	70.62	8.04	7.54	7.68	7.48	22.8	23.0	557	561.2
IVa	20.0	207.0–209.0	57.65	56.74	6.72	6.35	6.71	6.65	26.20	25.17	673	634.9
IVb	50.0	196.0–197.0	51.01	50.47	5.95	5.50	6.10	5.89	33.46	33.58	760	713.9
V	37.2	256.0 dec	47.98	48.27	4.94	4.95	6.10	6.25	36.10	35.69	699	671.8

[a] I, $[C_6H_3(CH_3)_2]_3B_3N_3(CH_3)_3$; II, $[C_6H_2(CH_3)_3]_3B_3N_3(CH_3)_3$; III, $[C_6H_2(CH_3CO)(CH_3)_2]_3B_3N_3(CH_3)_3$; IVa, $[C_6H_2(CH_3)_3][C_6H_2(CH_3)_2(CH_2Br)]_2B_3N_3(CH_3)_3$; IVb, $[C_6H_2(CH_3)_2(CH_2Br)]_3B_3N_3(CH_3)_3$; V, $[C_6H_3(CH_3)(CH_2Br)]_3B_3N_3(CH_3)_3$.

TABLE II

CHEMICAL SHIFTS AND RELATIVE INTENSITIES[a]

Compd no.[b]	Chemical shift τ value				
	Ring proton	Methylene proton	N-Methyl proton	Phenylmethyl proton	
				Position	
I	3.04 3.05 (8.9)	...	7.59 (9.3)	2-, 6-	7.77 (17.8)
II	3.27 (5.5)	...	7.59 (9.4)	4- 2-, 6-	7.77 (9.1) 7.82 (18.1)
III	2.47[c] (2.9) 2.97 (2.7)	...	7.59 (8.9)		7.52 (9.2) 7.62 (8.9) 7.75 (9.5)
IVa	2.79–3.27 (5.6)	5.69 (4.1)	7.57 (9.6)		7.73 (20.8) 7.78
IVb	2.75–3.25 (6.0)	5.63 (6.3)	7.56 (9.7)	4- 6-	7.72 (7.8) 7.76 (9.3)
V	2.65–3.02 (8.8)	5.57 (7.2)	7.51 (9.0)	6-	7.70 (8.1)

[a] The values in parentheses represent relative intensities. [b] For compound identification see Table I, footnote *a*. [c] AB-type quartet; J_{AB} = 7.65 cps.

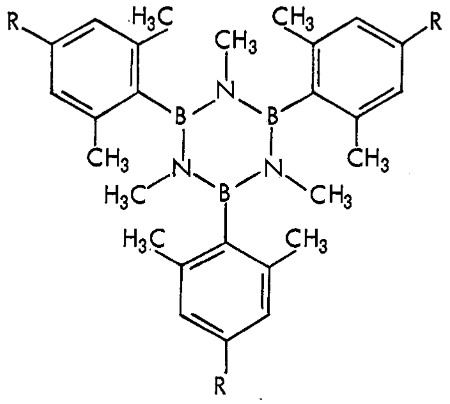

Figure 1.—Structures of compounds I and II: I, R = H; II, R = CH_3.

The proton magnetic resonance spectrum of B-triphenyl-N-trimethylborazine in carbon disulfide solution showed a single peak due to the N-methyl protons at τ 7.51, higher by about 0.7 ppm than the position of the peak due to the protons in B-trihalogeno-N-trimethylborazines.[9] Mooney has explained this high-field shift as an effect of diamagnetic anisotropy of phenyl rings which are perpendicular to the borazine ring, following the explanation given for the high-field shift of the B-methyl signal of B-trimethyl-N-triphenylborazine.[10] In the present case, the N-methyl signals of both I and II occur at τ 7.59, which is even higher than the τ 7.51 of B-triphenyl-N-trimethylborazine. This yields additional evidence for the orientation of the aryl rings; it is now apparent that the *o*-methyl groups on the aromatic rings are located at such a position that they will cover the vacant 2p orbitals of the three boron atoms.

We have previously prepared various arylborazines and examined the hydrolytic stability of the borazine ring.[4] These borazine derivatives, which are identified by elemental analyses and infrared spectra, are listed in Table III with some of their properties and infrared characteristic bands. When the compounds were subjected to hydrolysis in water or water–acetone mixtures, the precipitates recovered from the solution did not exhibit an infrared absorption in the boron–nitrogen stretching region, suggesting the decomposition of the borazine ring. However, compounds I and II display remarkable stability to hydrolysis in a mixed solvent of water (5%) and dioxane (95%) and can be recovered quantitatively after the solutions are allowed to stand for 1 month. B-Trimethyl-N-triphenylborazine is completely decomposed under the same conditions. Although the hydrolysis rate of the borazines is known to increase greatly by the addition of a base or an acid,[2,11] such addidion did not give rise to appreciable decomposition of the borazine ring in the case of compounds I and II. Compounds I and II, when subjected to Friedel–Crafts acetylation and free-radical bromination, exhibited an extraordinary stability which suggests that the *ortho* substituents in

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(9) E. F. Mooney, *Spectrochim. Acta*, **18**, 1355 (1962).

(10) K. Ito, H. Watanabe, and M. Kubo, *J. Chem. Phys.*, **34**, 1043 (1961).

(11) T. Yoshizaki, to be published.

Table III

Characteristics of Some Arylborazines

Compd	Mp, °C	Solubility,[c] g/100 ml of H_2O	ν_{B-N},[e] cm^{-1}
$(C_2H_5)_3B_3N_3(p\text{-}C_6H_4Cl)_3$[a]	260–261	. . .	1388
$(C_2H_5)_3B_3N_3(p\text{-}C_6H_4Br)_3$[a]	320	. . .	1388
$(C_2H_5)_3B_3N_3(p\text{-}C_6H_4COOC_2H_5)_3$[a]	203–204	. . .	1389
$[p\text{-}C_6H_4N(CH_3)_2]_3B_3N_3(C_2H_5)_3$[a]	283–284	. . .	1422
$(C_2H_5)_3B_3N_3[p\text{-}C_6H_4N(CH_3)_2]_3$[a]	297–299	. . .	1390
$[p\text{-}C_6H_4N(CH_3)_2]_3B_3N_3(C_6H_5)_3$[a]	322[d]	. . .	1380
$(C_6H_5)_3B_3N_3[p\text{-}C_6H_4N(CH_3)_2]_3$[a]	320[d]	. . .	1392
$(C_2H_5)_3B_3N_3[p\text{-}C_6H_4N(CH_3)_2C_2H_5Br]_3$[b]	243[d]	5.0	1393
$(C_2H_5)_3B_3N_3[p\text{-}C_6H_4N(CH_3)_3I]_3$[b]	307[d]	2.5	1391
$[p\text{-}C_6H_4N(CH_3)_3I]_3B_3N_3(C_2H_5)_3$[b]	280[d]	5.2	1424
$(C_6H_5)_3B_3N_3[p\text{-}C_6H_4N(CH_3)_3I]_3$[b]	300[d]	50	1384
$[p\text{-}C_6H_4N(CH_3)_3I]_3B_3N_3(C_6H_5)_3$[b]	>320	2.6	1387

[a] These compounds have been prepared by the action of the appropriate Grignard or lithium reagents on the B-trichloro-N-trialkyl(aryl)borazines. [b] The quaternary products have been obtained by treating an excess of trialkyl halides with the parent borazines in chloroform. [c] Solubility at room temperature. [d] With decomposition. [e] Nujol mull.

the B-aryl groups may exert a steric hindrance to the attack of reagents on the borazine ring. Although the acetylation was attempted on B-triphenyl-N-trimethylborazine under the same conditions, compounds containing boron–nitrogen bonds were not isolated.

The proton magnetic resonance spectra should now be referred to because they have been employed to clarify the position of substituents in the molecules. The location of the acetyl groups in B-tris(3-acetyl-2,6-xylyl)-N-trimethylborazine (III) was determined by the proton magnetic resonance spectrum, which showed four singlet signals of the same intensity due to N-methyl, acetyl, and two kinds of phenylmethyl groups. This feature could occur when and only when an acetyl group is replaced for a *m*-hydrogen on each aryl ring. The ring protons afford two doublets centered at τ 2.47 and 2.97, the coupling constant between them being 7.65 cps. The coupling constant can lend further support for the existence of two adjacent hydrogens, *i.e.*, the replacement of an acetyl group at the *meta* position, because the coupling constant between two adjacent hydrogens usually falls in the range of 7 to 10 cps, while that between the hydrogens *meta* to each other is 2 to 3 cps.[12] B-Mesityl-B′,B′′-bis(2-bromomethyl-4,6-xylyl)-N-trimethylborazine (IVa) and B-tris(2-bromomethyl-4,6-xylyl)-N-trimethylborazine (IVb) gave a singlet signal attributable to the methylene protons at τ 5.69 and 5.63, respectively, with proper integrated intensity. In going from compound II to the bromo derivatives IVa and IVb, the simple methyl signals of the former change to a composite group of lines with decreased intensity; this indicates that the *o*-methyl groups on the aryl rings are brominated. Similarly, bromination of compound I gives B-tris(2-bromomethyl-6-methylphenyl)-N-trimethylborazine (V) showing that the *o*-methyl groups are brominated in this case too.

It is to be noted that the thermal degradation of compound I was not observed by heating at 450°, for 3 hr, *in vacuo*.

Experimental Section

B-Tri-2,6-xylyl-N-trimethylborazine.—This compound was prepared in a manner similar to that of Ryschkewitsch, *et al.*[7] A tetrahydrofuran solution of 2,6-xylylmagnesium bromide,[6] prepared from 6.3 g (0.259 mole) of magnesium turnings and 45.7 g (0.247 mole) of 2-bromo-*m*-xylene,[13] was added dropwise to a stirred solution of 15.0 g (0.067 mole) of B-trichloro-N-trimethylborazine in 130 ml of dry benzene. After addition was completed, the reaction mixture was refluxed for 7 hr, and the solvent was replaced by ether. Then, 100 ml of saturated ammonium chloride solution was added to the reaction mixture to decompose the excess Grignard reagent, and the organic layer was concentrated to about 50 ml. The product was separated from solution in the form of needles by addition of methanol. Recrystallization from ether–methanol solvent pair gave 14.5 g of B-tri-2,6-xylyl-N-trimethylborazine (I). Ultraviolet: λ_{max} 200.2 mμ, ϵ_{max} 174,000; λ_{max} 220.0 mμ, ϵ_{max} 36,200; λ_{max} 265.0 mμ, ϵ_{max} 800. Infrared: 3071, 3001, 2925, 2906, 1596, 1453, 1446, ν_{BN} 1400, 771 cm^{-1}.

B-Trimesityl-N-trimethylborazine.—The same molar ratios and the same technique as cited above were employed in the reaction between mesitylmagnesium bromide[6] and B-trichloro-N-trimethylborazine. The resulting product was obtained as colorless needles. Ultraviolet: λ_{max} 204.9 mμ, ϵ_{max} 167,700; λ_{max} 223.8 mμ, ϵ_{max} 38,500; λ_{max} 268.0 mμ, ϵ_{max} 810. Infrared: 2972, 2822, 2747, 1628, 1443, ν_{BN} 1402, 847 cm^{-1}.

Friedel–Crafts Acetylation of B-Tri-2,6-xylyl-N-trimethylborazine.—To a solution of 1.8 g (0.004 mole) of I in 100 ml of carbon disulfide were added 2.0 g (0.015 mole) of anhydrous aluminum chloride and a solution of 1.2 g (0.015 mole) of acetyl chloride in 25 ml of carbon disulfide with vigorous stirring at room temperature. The reaction mixture was agitated for about 8 hr and allowed to stand overnight at room temperature. Then, the reaction mixture was poured into water and extracted with 200 ml of methylene chloride. The organic layer was dried over sodium sulfate. Removal of the solvent under reduced pressure followed by recrystallization from methanol gave 1.1 g of B-tris(3-acetyl-2,6-xylyl)-N-trimethylborazine (III). Compound III was identified by analytical data, infrared, and proton magnetic resonance spectra. Here, it should be mentioned that the acetyl group could not be determined by the Kuhn–Roth method,[14] and therefore infrared spectrophotometry was employed. The absorption intensity of carbonyl stretching vibration at 1686 cm^{-1} indicated that three acetyl groups had been introduced into the molecule. Infrared: 3057, 3009, 2922, 2847, ν_{CO} 1686, 1584, 1447, ν_{BN} 1383, 814 cm^{-1}. Compound III is soluble in methanol (solubility, 1.6 g/100 ml at room temperature).

Bromination of B-Trimesityl-N-trimethylborazine.—To a solution of 2.39 g (0.005 mole) of compound II in 30 ml of carbon tetrachloride were added 2.70 g (0.015 mole) of N-bromosuccinimide and 15 mg of benzoyl peroxide. The resulting mixture was refluxed for 3 hr. The succinimide isolated by filtration weighed 1.32 g (88.6%). The filtrate was evaporated to dryness to leave 2.60 g of the residue. The residue was dissolved in *n*-hexane, placed on a column of alumina (50 g of Merck neutral alumina), and eluted first with *n*-hexane and then with ether to give two separate fractions. The dibromo derivative $C_{30}H_{40}B_3N_3Br_2$ (IVa) in the yield of 20% was obtained from the *n*-hexane eluent and the tribromo derivative $C_{30}H_{39}B_3N_3Br_3$ (IVb) in the yield of 50% from the ether eluent. These were purified by recrystallization from methanol–ether mixed solvent. Although it has not been rigorously demonstrated that these are

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(12) R. M. Silverstein and G. C. Bassler, "Spectrometric Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1963, p 88.

(13) J. L. Hartwell, "Organic Syntheses," Coll. Vol. III, John Wiley and Sons, Inc., New York, N. Y., 1955, p 185.

(14) R. Kuhn and H. Roth, *Ber.*, **66**, 1274 (1933).

the exclusive products of the reaction, the latter appears to be at least the predominant product. Infrared: IVa: 1613, 1442, ν_{BN} 1402 cm^{-1}; IVb: 1611, 1445, ν_{BN} 1403 cm^{-1}.

Bromination of B-Tri-2,6-xylyl-N-trimethylborazine.—The same molar ratios and the same technique as described above were employed in the bromination of compound I; the tribromo derivative $C_{27}H_{33}B_3N_3Br_3$ (V) was isolated from the reaction mixture but not the dibromo derivative. Infrared: 1595, 1448, ν_{BN} 1402 cm^{-1}.

Stability to Hydrolysis.—A weighed sample of I was dissolved in the dioxane (95%)–water (5%) mixed solvent. After 1 month the solvent was gradually replaced by water. The crystal formed was collected by filtration on a sintered-glass funnel, washed with a small amount of methanol, dried under vacuum, and weighed (recovery: 100%; mp 282.5–283.5°; *Anal.* C, 74.68; H, 8.59; N, 9.74; mol wt, 439). The same technique was also applied to the hydrolysis experiments in the dioxane (95%)–1 *N* NaOH (5%) and the dioxane (95%)–1 *N* HCl (5%) solution after 150 hr (each recovery: 100%). All recovered crystals were identical with starting borazine as shown by melting point measurement, infrared, proton nuclear magnetic resonance spectra, and elemental analyses.

Physicochemical Determination.—Infrared spectra were recorded in carbon tetrachloride and carbon disulfide solution on a DS-201B spectrophotometer from Japan Spectroscopic Co., Ltd., Tokyo. Proton magnetic resonance spectra were taken with a Varian A-60 high-resolution nmr spectrometer operating at 60 Mc. In these experiments, carbon tetrachloride solutions were used with tetramethylsilane as an internal reference. Ultraviolet spectra were recorded in *n*-heptane solution using a Beckman DK-2A ratio recording spectrophotometer from Beckman Instruments Inc. Molecular weights were determined by a vapor pressure osmometer, Model 301A, from Mechrolab Inc.

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A Nuclear Magnetic Resonance Study of Hydrogen Bonding in Tris(2-N-methylaminoethyl) Borate and Similar Compounds[1]

BY DEVON W. MEEK AND CHARLES S. SPRINGER, JR.

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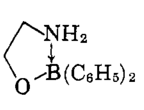
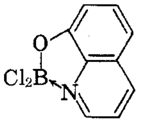
Tris(2-aminoethyl) borate, $B(OCH_2CH_2NH_2)_3$, and the analogous N-methylamino and N,N-dimethylamino compounds have been prepared by the transesterification of methyl borate with the appropriate 2-aminoethanol. The nuclear magnetic resonance spectra show that extensive association of the terminal amino groups occurs in pure tris(N-methylaminoethyl) borate and that this association can be broken apart by heating to 160° or by dissolution in polar organic solvents such as triethylamine or acetonitrile. In order to determine whether the association results from hydrogen bonding or internal B–N coordination, several model systems have been investigated. The spectra of $NH_2C_2H_5 \cdot BF_3$, piperidine·BF_3, and $(C_6H_5)_2BOCH_2CH_2NH_2$ in acetonitrile contain very complicated NCH_2 peaks and broad NH peaks which appear at low applied magnetic field (τ 5.26–5.56). The broadening of the NH peak in the boron–nitrogen adducts is attributed to the effect of the N^{14} quadrupole, whereas the complex splitting of the NCH_2 multiplet is attributed to coupling with B^{11} in the dative bond with nitrogen and possibly with the nitrogen protons. The spectra of $B(OCH_2CH_2NH_2)_3$ and $B(OCH_2CH_2NHCH_3)_3$, on the other hand, show sharp NH peaks at somewhat higher applied magnetic fields (τ 7.84, 6.78) and contain two sharp triplets attributed to the two sets of methylene protons in the $-O-CH_2-CH_2-N<$ units (τ_{OCH_2} 6.26–6.55; τ_{NCH_2} 7.23–7.57). The N–H peak of $B(OCH_2CH_2NHCH_3)_3$ appears as a sharp singlet in acetonitrile solutions owing to rapid exchange of the amine proton. Evidence is presented that the exchange is catalyzed by a trace (<8.7 ppm) of water in the hygroscopic solvent, acetonitrile, even after rigorous drying. All of the data indicate that intermolecular hydrogen bonding strongly predominates over N→B dative bonding in the association of $B(OCH_2CH_2NHCH_3)_3$.

Introduction

The B–N dative bond is one of the most common in nonmetallic coordination compounds. Several types of boron compounds which contain an aminoethyl or similar donor linkage have been reported to form internal[2] B–N dative bonds. Typical examples are 2-aminoethyl diphenylboronite (I)[3,4] and 8-oxyquinoline dichloroboronite (II).[5]



I



II

As a result of transannular interaction from the confined nitrogen atom, the boron derivatives of diethanolamine[4,6,7] and triethanolamine[7,8–10] have been reported to form B–N bonds in structures III and IV. However,

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[1] The authors gratefully acknowledge the support by Grant DA-ARO-D-31-124-G500 from the U. S. Army Research Office (Durham).

[2] Internal coordination, as used in this paper, refers to formation of a chelate within the molecule itself.

[3] R. L. Letsinger and I. Skoog, *J. Am. Chem. Soc.*, **77**, 2491 (1955).

[4] M. F. Lappert, *Chem. Rev.*, **56**, 959 (1956).

[5] H. K. Soha, *J. Inorg. Nucl. Chem.*, **26**, 1617 (1964).

[6] O. C. Musgrave and T. O. Park, *Chem. Ind.* (London), 1552 (1955).

[7] P. M. Maithis, *Chem. Rev.*, **62**, 223 (1962).

[8] H. C. Brown and E. A. Fletcher, *J. Am. Chem. Soc.*, **73**, 2808 (1951).

[9] H. Steinberg and D. L. Hunter, *ibid.*, **82**, 853 (1960).

[10] P. Castle, R. Stoesser, and E. F. Westrum, Jr., *J. Phys. Chem.*, **68**, 47 (1964).