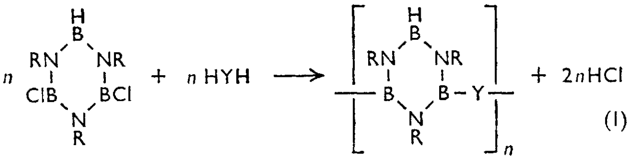
Synthesis and Properties of Linear Polyborazoles. Part II.[1] Condensation of *B*-Dichloro-*N*-tri-(2,6-dimethylphenyl)borazole and Aromatic and Heterocyclic Diamines

By J. M. Turner

The linear polymerisation of *B*-dichloro-*N*-tri-(2,6-dimethylphenyl)borazole through the aromatic diamines, *m*- and *p*-phenylenediamine, *p,p'*-diaminodiphenylmethane, and benzidine, and the heterocyclic diamines, piperazine and ammeline, has been investigated and the range and degree of plasticity, stability in air, and hydrolytic and thermal stability of the linear polyborazoles formed have been compared and related to their structure and degree of polymerisation.

THIS work continues that in Part I[1] and attempts to synthesise linear polyborazoles of high hydrolytic, thermal, and oxidative stabilities.

The synthesis of the linear polyborazoles in solvent tetrahydronaphthalene was based on equation (1),



(1)

where R = 2,6-dimethylphenyl here and throughout this Paper and HYH represents a diamine.

The reaction of *B*-dichloro-*N*-tri-(2,6-dimethylphenyl)-borazole with *m*-phenylenediamine was straightforward. Apart from hydrogen chloride and the linear polyborazole (I; 94·1% yield), the only other substance formed was a little (5·9%) insoluble borazole material which contained no chlorine. Such materials of no or low chlorine content were also formed with *p*-phenylenediamine. Four experiments were carried out using this reagent, and the amount of insoluble borazole material increased with the time of refluxing, and there was a corresponding decrease in the amount of linear polyborazole (Table 1). These observations imply that the

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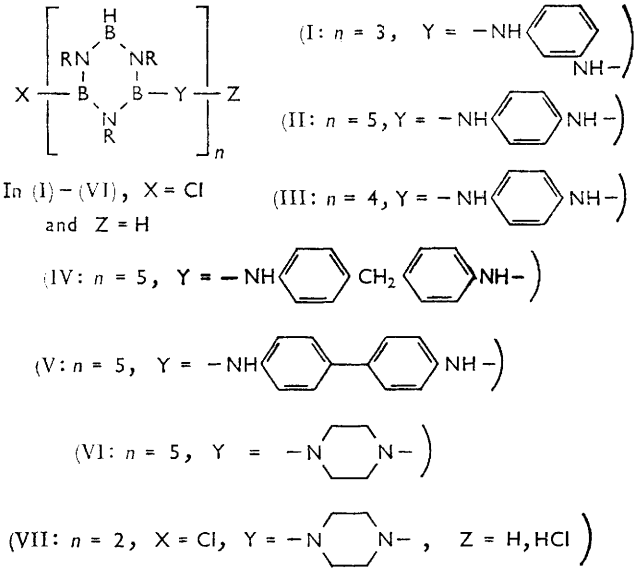
[1] Part I, J. M. Turner, preceding Paper.

insoluble borazole materials were formed either through the gradual increase in the degree of cross-linking or chain-branching, or through the increase in the molecular weight of the linear polyborazoles.

TABLE 1

No.	Time of refluxing (hr.)	Linear polyborazole (%)	Insoluble borazole (%)
2	3	(II) 92·6	7·4
3	5	(III) 67·5	32·5
4	5½	(III) 36·1	63·9
5	7	(II) 18·6	81·4

Cross-linking or chain-branching was also indicated by the infrared spectra of the insoluble borazole materials from experiments 3 and 5 (groups of two adjacent free ring-hydrogen atoms were present). That from experiment 5 also had cationic amino-groups or intermolecularly hydrogen-bonded amino-groups. Both compounds also showed the absorptions expected of the borazole units present except that of the boron–hydrogen bond which, if present, failed to show; primary amino-groups were present, too. The hydrolysate of the material from experiment 5 showed additional infrared absorptions; besides those caused by hydroxyl groups, that of the boron–hydrogen group was present. This latter absorption did, therefore, fail to show in the corresponding insoluble borazole material itself.



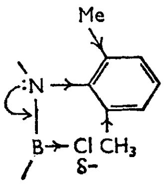
Deficient total analyses, typical of cross-linked polyborazoles,[2] were found for the insoluble borazole material of experiment 5. The *X*-ray patterns of the insoluble borazoles from experiments 1—5 were similar to those of the linear polyborazoles, but were darker and more diffuse, indicating more cross-linking or chain-branching or, perhaps, a higher degree of polymerisation.

In experiments 2, 3, and 5 there was a second insoluble by-product. *X*-Rays confirmed the presence of *p*-phenylenediamine dihydrochloride in all these, but the chlorine contents showed that only that from experiment 2 was pure dihydrochloride and that from experiment 5 was the monohydrochloride. The latter was, more likely, a mixture of the free amine and its dihydrochloride, as probably was the material from experiment 3 because its chlorine content was lower than that for the monohydrochloride. Hydrogen chloride was also evolved in experiments 2—5, as in every experiment. With *p,p'*-diaminodiphenylmethane and benzidine, insoluble borazole compounds (8·6 and 12·3%, respectively) were formed but no amine hydrochlorides, in accordance with the weak basicity of these amines (first and second dissociation constants for benzidine in aqueous solution are $9{\cdot}3 \times 10^{-10}$ and $5{\cdot}6 \times 10^{-11}$ at 30°, respectively). The insoluble borazole compounds contained chlorine and their *X*-ray patterns were similar to those of the corresponding linear polyborazoles (IV and V), which were isolated in good yields.

With piperazine, the insoluble borazole material (29·2%) had an *X*-ray pattern which was not similar to that of the linear polyborazole (VI). Its chlorine content of 7·1% together with its insolubility suggested that it had structure (VII), which contains 6·95% chlorine. The other insoluble by-product was, from its *X*-ray pattern and chlorine content, piperazine dihydrochloride.

Ammeline was unreactive, and was recovered unchanged, though *B*-dichloro-*N*-tri-(2,6-dimethylphenyl)-borazole lost some hydrogen chloride after being refluxed in tetrahydronaphthalene for 5 hr.

All these condensations of *B*-dichloro-*N*-tri-(2,6-dimethylphenyl)borazole with aliphatic,[1] aromatic, and heterocyclic diamines are probably dependent on the inductive and mesomeric effects in this borazole (VIII).



(VIII)

The electronegative chlorine atoms will combine with protons from a diamine with the elimination of hydrogen chloride and the formation of a diamine link. For aromatic diamines containing unconjugated phenyl groups (*e.g.*, *p,p'*-diaminodiphenylmethane electrons will be withdrawn from the nitrogen atoms, thus making the loss of a proton easier.

The structures of the linear polyborazoles were not strictly those designated by the formulæ; the molecular weights indicated that other polymers were present. Slightly higher degrees of polymerisation were obtained with these linear polyborazoles than with those described in Part I,[1] which contained aliphatic diamine links. The presence of a certain degree of cross-linking or chain-branching was indicated from the infrared spectra of all the linear polyborazoles because groups containing two adjacent free ring-hydrogen atoms were present.

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[2] C. F. Blakely, Ministry of Aviation Symposium, " Fundamental Research on New Macromolecules," Sept. 1961.

(I), (II), (III) (from experiments 3 and 4) and (V) contained amine hydrochloride or intermolecularly hydrogen-bonded amino-groups also. The absorptions attributed to the borazole rings present were all revealed except that the boron–hydrogen groups failed to show in (I), (III), and (IV). The hydrolysate of (III) did absorb at 2520 cm.$^{-1}$, however, so it seems that boron–hydrogen groups did not show rather than their being absent. Primary and secondary amino-groups (from the terminal links) were present in every case except (VI) in which (as expected from a consideration of its terminal link which is based on piperazine) secondary amino-groups only were present. Hence, as with the linear polyborazoles containing aliphatic diamine links,[1] the structures deduced from the elemental analysis and molecular weights are to some extent supported by the infrared spectra but there is the complication of the presence of some cross-linking or chain-branching and either some cationic amino-groups or intermolecularly hydrogen-bonded amino-groups in many cases.

All the linear polyborazoles except (VI) gave dark diffuse *X*-ray patterns which contained two broad halos. (VI) had a good pattern which had many resemblances to that of *B*-dichloro-*N*-tri-(2,6-dimethylphenyl)borazole, which may be present in this material, therefore.

The ranges of plasticity in nitrogen of the linear polyborazoles are compared in Table 2. Although a " range of plasticity " (from the point where softening commences to the point of decomposition) is referred to for (I), (II), and (III), these linear polyborazoles softened only slightly before decomposing. This was expected because of their short rigid bridging groups (*m*- and *p*-phenylenediamine links). With the longer aromatic diamine bridging groups, which also have central points allowing free rotation, the linear polyborazoles (IV) and (V) softened at a lower temperature and became " like putty " eventually. They never became " very mobile," however, in contrast to those which contained long aliphatic diamine links.[1] Rotation about the carbon–carbon bond in benzidine is not so free as that about the methylene group in p,p′-diaminodiphenylmethane so the initial sotening temperature of (V) was higher (by 20°) than that of (IV). The fact that (VI), containing piperazine links, softened at the lowest temperature (85°) and finally became a mobile liquid, was unexpected because the piperazine link is shorter than that of *m*- and *p*-phenylenediamine so might reasonably have been expected to give the highest softening temperature of all even though there is more flexibility in a piperazine ring (between *cis*- and *trans*-forms). None of these materials has as high a range of plasticity as the linear polyborazoles containing ethylenediamine links.[1]

TABLE 2

Linear polyborazole	(I)	(II)	(III)	(IV)	(V)	(VI)
Range of plasticity	190—250°	190—260°	190—260°	155—260°	175—220°	85—215°
Degree of polymerisation	3	5	4	5	5	5

All the linear polyborazoles were stable in air. Appreciable weight losses occurred on refluxing in water. From the molecular weights of the hydrolysates from (I) and (III) (experiment 3), it was concluded that even after 8 hours' refluxing in water dimers remained. These polymers with *m*- and *p*-phenylenediamine links were the most resistant to hydrolysis; stereochemical factors are, no doubt, responsible. Materials (IV), (V), and (VI) lost much weight after 3—4 hours' refluxing. In this respect, as in its plasticity, the last material, which has piperazine links, is anomalous.

All hydrolysates showed strong infrared absorptions caused by hydroxyl groups, secondary amino-groups, and all groups contained in the borazole rings except boron–hydrogen groups. The latter remained only in those from (III), (experiment 3), (V), and (VI). Since all hydrolysates contained secondary amino-groups, hydrolysis in each case was incomplete. Absorptions at 805 cm.$^{-1}$ were always present. When unaccompanied by absorptions at 3200—3050 cm.$^{-1}$, these were assigned solely to two adjacent free ring-hydrogen atoms, which meant, as explained in Part I,[1] that boron–carbon bonds formed through cross-linking or chain-branching were present, as expected, in the hydrolysates. The hydrolysates of (I) and (V) absorbed at 3180 and 3170 cm.$^{-1}$ as well as at 800 cm.$^{-1}$. Hence, amine hydrochloride or intermolecularly hydrogen-bonded amino-groups were present. The hydrolytically unstable points in these materials were the bridging groups by which exocyclic (in relation to the borazole rings) boron–nitrogen groups were introduced.

Hydrolytic stabilities of other linear polyborazoles recorded in the literature were compared in Part I.[1]

Linear polyborazole (III) (from experiment 3) was the most thermally stable, but less stable than one which contained trimethylenediamine links.[1] There was a slight levelling of the thermograms in air of (III) and (IV) at 15—17·5 and 17% weight loss, respectively. These steps coincide fairly well with the percentage weight of methyl groups in these materials (III, 17·8%; IV, 15·2). Hence degradation may commence at the methyl groups of the 2,6-dimethylphenyl substituents.[1] The stability of the insoluble borazole material from experiment 5 was slightly less than that of its linear counterpart (III). Linear polyborazole (VI) began to lose weight at 140° in air and 160° in nitrogen. Both these temperatures are close to that of 145° which is the boiling point of piperazine. The *X*-ray pattern suggested that *B*-dichloro-*N*-tri-(2,6-dimethylphenyl)-borazole was present. From these two results, (VI) may contain free piperazine and the free borazole, whose presence would help to explain the very wide range of plasticity (85—215°) observed; pure piperazine melts at 104° and the pure borazole at 240—244°.

EXPERIMENTAL

General.—All reagents were dried as described in Part I.[1] *m*- and *p*-Phenylenediamine were distilled in nitrogen and

TABLE 3

Infrared vibrational frequencies

Expt.:	1	2	3	4	6	7	8	3	5
A *	(I)	(II)	(III)	(III)	(IV)	(V)	(VI)	†	†
B									
C	3450	3500	3530	3500	3500	3460	3300	3520	3530
	3330	3300	3320	3330	3330	3300		3330	3330
D	3140	3160	3160	3190		3170			3150
		↓							
		3060							
E	3020	3020	3020	2990	3000	2970	3000	2990	3000
F	Masked by Nujol								
G		2490		2520		2520	2500		
H	1605					1605	1605		
		1505	1505	1505	1505			1505	1509
I	1455	1437	1460	1460	1460	1455	1460	1460	1460
	↓	↓	↓	↓	↓	↓	↓	↓	↓
	1375	1372	1375	1390	1380	1375	1385	1380	1378
J	1260	1258	1260	1263	1260	1260	1259	1260	1260
K	1165	1165	1165	1165	1175	1165	1165	1145	1160
	1093	1092	1093	1095	1090	1095	1092	1095	1095
	1032	1030	1032	1035	1020	1030	1030	1032	1030
	982	985							
L	805	805	805	805		805			805
M	805	805	805	805	805	805	805	805	805
N	762	762	758	765	765	765	765	763	763
	735	742		735					
O	705	710	717	710	717	713	715	710	715
	695	697	708	705	705	709	702	705	700

* *A*, Material. *B*, OH stretch. *C*, NH and NH_2 stretch. *D*, NH_3^+ stretch or intermolecularly hydrogen-bonded amino-groups. *E*, Aromatic CH stretch of monosubstituted nucleus. *F*, Aliphatic CH stretch. *G*, BH stretch. *H*, C=C skeletal in-plane vibrations. *I*, BN stretch. *J*, CN stretch. *K*, 1,2,3-substituted aromatic rings. *L*, NH_3^+ rock. *M*, 2 Adjacent free ring-H atoms. *N*, 3 Adjacent free ring-H atoms. *O*, BN out-of-plane deformation characteristic doublet.

† Insoluble borazole material.

TABLE 4

Infrared vibrational frequencies

Expt.:	1	3	4	6	7	8	5
A *	Hydrolysate of linear polyborazole						Hydrolysate of insoluble borazole material
B	3520	3500	3500	3500	3500	3500	3480
	3430	3420	3400			3420	3400
P	3345	3340	3340	3310	3300	3330	3320
D	3180				3170		3180
E	3000	3000	3000	3000	3000	3000	2980
F	Masked by Nujol						
G		2520			2500	2510	2510
H	1590				1605	1605	
		1510	1510	1505			1505
I	1460	1460	1460	1455	1455	1460	1455
	↓	1405	1405	1370 ↓	1405	1378 ↓	1375 ↓
	1380	1375 ↓	1375 ↓		1375 ↓		
J	1265	1262	1260	1260	1265	1260	1262
K	1165	1163	1165	1165	1160	1163	1165
	1095	1095	1095	1092	1092	1092	1095
	1035	1035	1035	1035	1035	1035	1035
	990	990					
L	805				805		805
M	805	805	805	805	805	805	805
N	765	760	765	765	770	765	765
O	715	717	713	713	715	715	710
	700	710	705	705	705	702	695

* See footnotes, Table 3.

other diamines were used as supplied. The handling of materials, analyses, molecular weights, *X*-ray spectra, infrared spectra (those of linear polyborazoles are in Table 3 and of the hydrolysates in Table 4), thermogravimetric analyses, plasticities in nitrogen (Table 5), stabilities in air, and hydrolytic stabilities were as in Part I.[1]

Preparation of Monomeric Borazoles.—*B*-Dihydroxy-*N*-tri-(2,6-dimethylphenyl)borazole and *B*-dichloro-*N*-tri-(2,6-dimethylphenyl)borazole were synthesised [1] by the method of Bartlett *et al.*[3]

Linear Polymerisation of B-*Dichloro*-N-*tri*-(2,6-*dimethylphenyl*)*borazole through Aromatic and Heterocyclic diamines.*—*Control experiments: stabilities in refluxing solvents.* See Part I.

Linear polymerisation through aromatic and heterocyclic

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[3] R. K. Bartlett, H. S. Turner, R. J. Warne, M. A. Young, and (in part) W. S. McDonald, *Proc. Chem. Soc.*, 1962, 153.

diamines. *B*-Dichloro-*N*-tri-(2,6-dimethylphenyl)borazole and the diamine were in a molar ratio of 1 : 1. In all experiments (1—9), the borazole was dissolved in 20—30 ml. of tetrahydronaphthalene by refluxing under nitrogen. When this solution was cool the solid aromatic diamine was added. It was then refluxed for a known time and hydrogen chloride was collected in distilled water by bubbling the outgoing gases through two traps containing distilled water for titration with sodium hydroxide. Insoluble materials were separated under nitrogen, dried, and weighed.

In experiments 2, 3, 5, and 8 there were two types of insoluble product; borazole material and aromatic diamine hydrochloride. In experiments 2, 3, and 5 the insoluble borazole material stuck to the sides of the flask, whereas *p*-phenylenediamine hydrochloride was formed as a cake on the surface of the solution and was filtered off under nitrogen. In experiment 8, the two insoluble solids were filtered off under nitrogen and the hydrochloride sublimed from the insoluble borazole *in vacuo*. All filtrates were evaporated to dryness under reduced pressure. The *X*-ray photographs of all unsoluble borazole materials (boron indicated by flame test) were similar to those of the soluble linear polyborazoles, except for the material from experiment 8. These *X*-ray photographs contained lines together with dark, diffuse halos.

TABLE 5

Plasticities in nitrogen

Expt.	Temp. at which material is: Stiff dough	Like putty	Stuck to glass rod	Pulled into threads	Tacky	Very mobile	M. p.
1 *	190°	220—230°	235°				250°
2, 3, 4 *	190	220—230	230—250				260
6	155	195	230				260
7	175		180—215				220
8	85	95	110	115°	165°		215

* In these cases the terms " stiff dough " and " like putty " should be replaced by " slight softening " and " adhered together," respectively. With all polymers except no. 8, there was no true m. p.; decomposition occurred first and the approximate temperature at which this commenced is that recorded under m. p.

In experiment 1, 1·23 g. of *m*-phenylenediamine and 5·256 g. of the borazole were refluxed for 3 hr. 0·7125 g. of hydrogen chloride, 0·38 g. of insoluble borazole material (Cl, 0·0%), and 5·4538 g. of soluble brown *solid* [Found: C, 69·4; H, 6·7; B, 7·0; Cl, 2·2; N, 13·25%; *M*, 1353. $C_{90}H_{103}B_9ClN_{15}$ (I) requires: C, 70·7; H, 6·7; B, 6·4; Cl, 2·3; N, 13·75%; *M*, 1527] were obtained. The last was stable in air but after being refluxed in water for 8 hr., it had lost 22·4% by weight and the hydrolysate had *M* = 824. Its *X*-ray pattern contained two dark diffuse halos, the strongest of which corresponded to 8·92 Å. In air, decomposition commenced at 240° and in nitrogen at 220°.

In experiment 2, 0·8164 g. of *p*-phenylenediamine and 3·4887 g. of borazole were refluxed for 3 hr. 0·3950 g. of hydrogen chloride, 0·3255 g. of amine hydrochloride (Found: Cl, 39·3. $C_6H_{10}Cl_2N_2$ requires Cl, 39·2%; *X*-ray data indicated the presence of *p*-phenylenediamine dihydrochloride), 0·09 g. of insoluble borazole compound (Cl, 0·0%) and 3·5287 g. of soluble brown *solid* [Found: C, 69·8; H, 6·6; B, 6·4; Cl, 1·5; N, 13·2%; *M*, 2244. $C_{150}H_{171}B_{15}ClN_{25}$ (II) requires: C, 71·4; H, 6·8; B, 6·45; Cl, 1·4; N, 13·9%; *M*, 2521] were obtained. The last was stable in air but after being refluxed in water for 8 hr., lost 18·6% by weight.

In experiment 3, 0·9837 g. of *p*-phenylenediamine and 4·205 g. of the borazole were refluxed for 5 hr. 0·5067 g. of hydrogen chloride, 0·558 g. of amine hydrochloride (Cl, 14·9%; *X*-rays indicated the presence of *p*-phenylenediamine dihydrochloride), 0·925 g. of insoluble, borazole compound (Cl, 2·0%; its infrared spectra is recorded in Table 3), and 3·113 g. of soluble brown solid [Found: C, 68·9; H, 7·0; B, 6·8; Cl, 1·8; N, 13·45%; *M*, 1866. $C_{120}H_{137}B_{12}ClN_{20}$ (III) requires: C, 71·1; H, 6·8; B, 6·5; Cl, 1·75; N, 13·8%; *M*, 2024] were obtained. The last was stable in air but after 8 hours' refluxing in water had lost 14·8% by weight and the hydrolysate had *M* = 837. Its *X*-ray photograph contained two dark diffuse halos and some lines; the strongest halo corresponded to 7·89 Å. In air, decomposition began at 250° and there was a levelling at 15—17·5%; in nitrogen, decomposition commenced at 230°.

In experiment 4, 0·9526 g. of *p*-phenylene diamine and 4·071 g. of the borazole were refluxed for 5½ hr. 0·6190 g of hydrogen chloride, 2·746 g. of insoluble, borazole compound (Cl, 0·0%), and 1·610 g. of soluble brown solid [Found: Cl, 1·7%; *M*, 1957. $C_{120}H_{137}B_{12}ClN_{20}$ (III) requires Cl, 1·75%; *M*, 2024] were obtained. After 8 hours' refluxing in water, the soluble brown solid lost 19·7% by weight. Its *X*-ray photograph contained two dark diffuse halos together with some lines; the strongest halo corresponded to 8·05 Å.

In experiment 5, 1·046 g. of *p*-phenylenediamine and 4·469 g. of the borazole were refluxed for 7 hr. 0·6016 g. of hydrogen chloride, 0·089 g. of amine hydrochloride (Found: Cl, 23·4. $C_6H_9ClN_2$ requires Cl, 24·7%; *X*-ray data indicated the presence of *p*-phenylenediamine dihydrochloride), 3·777 g. of insoluble borazole compound (Found: C, 51·0; H, 6·2; B, 4·9; Cl, 1·35; N, 11·4%, its infrared spectrum is recorded in Table 3) and 0·91 g. of soluble brown *solid* [Found: Cl, 1·4. $C_{150}H_{171}B_{15}ClN_{25}$ (II) requires Cl, 1·4%] were obtained. After 8 hours' refluxing in water, the soluble brown solid had lost 15·3% by weight. The insoluble borazole compound began to decompose at 230° in air and at 190° in nitrogen.

In experiment 6, 1·274 g. of *p,p'*-diaminodiphenyl methane and 2·9690 g. of the borazole were refluxed for 3¼ hr. 0·4375 g. of hydrogen chloride, 0·4479 g. of insoluble borazole compound (Found: Cl, 4·15%), and 3·4902 g. of soluble brown *solid* [Found: C, 73·3; H, 6·6; B, 5·4; Cl, 1·2; N, 11·5%; *M*, 2624. $C_{185}H_{201}B_{15}ClN_{25}$ (IV) requires C, 74·8; H, 6·8; B, 5·5; Cl, 1·2; N, 11·8%; *M*, 2969] were obtained. The last was stable in the air but after 3—4 hours' refluxing had lost 30·1% by weight. Its *X*-ray photograph contained two dark diffuse halos together with some lines; the strongest halo corresponded to 4·26 Å. In air, decomposition commenced at 230° and there was a levelling at 17% weight loss. In nitrogen, initial decomposition occurred at 200°.

In experiment 7, 1·331 g. of benzidine and 3·3390 g. of the borazole were refluxed for 4½ hr. 0·4466 g. of hydrogen chloride, 0·3814 g. of insoluble borazole compound [Found: Cl, 2·66%), and 3·6787 g. of soluble brown *solid* (Found: C, 74·0; H, 6·6; B, 6·1; Cl, 1·2; N, 11·4%; *M*, 3089. $C_{180}H_{191}B_{15}ClN_{25}$ (V) requires C, 74·5; H, 6·6; B, 5·6; Cl, 1·2; N, 12·1%; *M*, 2899] were obtained. The soluble brown solid was stable in the air but after 3—4 hours' refluxing in water, it had lost 41·3% by weight. Its *X*-ray photograph contained two dark, diffuse halos together with some lines; the line about the strongest halo corresponded to 6·01 Å. In air, it began to decompose at 210° and at 230° in nitrogen.

In experiment 8, 0·6701 g. of piperazine and 3·5957 g. of the borazole were refluxed for 3 hr. 0·2997 g. of hydrogen chloride, 0·3740 g. of amine hydrochloride (Found: Cl, 45·7. $C_4H_{12}Cl_2N_2$ requires Cl, 44·7%; *X*-rays indicated this material to be piperazine dihydrochloride), 0·7626 g. of insoluble borazole compound (Found: Cl, 7·1%) and 2·6590 g. of soluble brown *solid* [Found: C, 68·30; H, 7·40; B, 7·3; Cl, 1·4; N, 13·9%; *M*, 2341. $C_{140}H_{181}B_{15}ClN_{25}$ (VI) requires C, 69·8; H, 7·5; B, 6·75; Cl, 1·5; N, 14·5; *M*, 2409] were obtained. The last was stable in air but after being refluxed in water for 3—4 hr. it had lost 25·4% by weight. It had a good *X*-ray pattern with distinct lines, the strongest corresponding to 7·17 Å. In air, decomposition began at 140° and in nitrogen at 160°.

In experiment 9, 0·6901 g. of ammeline and 2·5082 g. of the borazole were refluxed for 5 hr. 0·1111 g. of hydrogen chloride, 0·6877 g. of insoluble solid (*X*-ray data indicated that this material was ammeline), and 2·5409 g. of soluble borazole material (Found: Cl, 11·3%) were obtained. The *X*-ray pattern of the last was identical to that of the borazole.

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