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BORON-NITROGEN POLYMERS I MECHANISTIC STUDIES OF
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K J PACIOREK ET AL. 28 SEP 84 SN-2022-11

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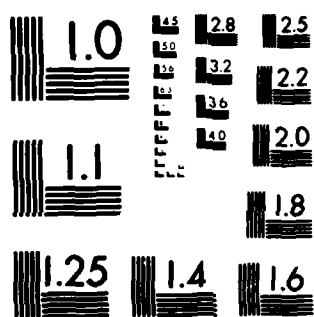
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Boron-Nitrogen Polymers.
I. Mechanistic Studies of Borazine Pyrolyses

by

K. J. L. Paciorek, D. H. Harris,
and R. H. Kratzer

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BORON-NITROGEN POLYMERS.

I. MECHANISTIC STUDIES OF BORAZINE PYROLYSES

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Synopsis

Pyrolyses of B-triamino-N-triphenyl-, B-triamino-N-trimethyl-, and B-trianilino-borazines were performed between 150 and 300°C. Initial stages of degradation were accompanied by liberation of ammonia in addition to the expected aniline or methylamine; this was most pronounced for the methyl borazine. Aniline elimination proceeded more readily with the B-anilino than the B-amino isomer. Data obtained support a ring opening mechanism resulting in telomerization accompanied by aniline liberation and formation first of singly then doubly bridged dimers and finally doubly bridged tetramers. Thermal exposure up to 1000°C failed to give boron nitride; carbon was invariably retained.

INTRODUCTION

The ability of borazines to undergo thermal polymerization has been well proven by the extensive investigations conducted in the late fifties and early sixties¹. Present interest in "polymerizable" borazines was prompted by their potential suitability as boron nitride precursors². A borazine which can be transformed into a readily processible polymer, prior to the final pyrolysis to a ceramic, offers promise for forming fibers, coatings, as well as bulk items. Actually, production of B-N fibers via pyrolysis of B-triamino-N-triphenylborazine has been recently described³.

A review of the literature has revealed conflicting data regarding the nature of the polymerization products derived from different borazines. To mention a few: linear structures were postulated for the materials obtained from B-trichloro-N-triphenylborazine on pyrolysis up to 470°C⁴; B-trisethylamino-N-triethylborazine on heating at 300°C for 4 hr was reported to polymerize to borazine rings joined by ethyl-substituted nitrogen atoms⁵; whereas attachment via ring boron and nitrogen atoms was postulated by Gerrard, et al.⁶ in the interaction of chloroborazine with, e.g. di-s-butylamine. The current study was undertaken to determine the operating processes and their dependence on the nature of the substituents on the ring boron and nitrogen atoms, the ultimate aim being the identification of borazines most suitable for pre-ceramics production.

EXPERIMENTAL

General

All solvents used were reagent grade and were dried and distilled prior to use. Operations involving moisture or air-sensitive materials were carried out either in an inert atmosphere enclosure (Vacuum Atmospheres Model HE-93B), under nitrogen by-pass, or in vacuo. The commercially available starting materials were usually purified by distillation, crystallization, or other appropriate means. Infrared spectra were recorded either neat (on liquids) or as double mulls (Kel-F oil No. 10 and Nujol) using a Perkin-Elmer Corporation Infrared Spectrophotometer Model 1330. The molecular weights were determined in benzene or chloroform using a Mechrolab Model 302 vapor pressure osmometer. The elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, New York.

Starting Materials

The following compounds were prepared using literature procedures: B-triamino-N-triphenylborazine, mp 251-255°C (lit. 255°C⁷), was obtained in an overall 74% yield in a two-step reaction via B-trichloro-N-triphenylborazine^{7,8}. A parallel process was utilized in the synthesis of the B-triamino-N-trimethylborazine^{7,9}, overall yield 70%, mp 88.5-90.5°C (lit. 89°C⁷). B-trichloroborazine, mp 83.5-84.5°C (lit. 83.9-84.5°C¹⁰) was prepared by the hot tube method of Brown and Laubengayer¹⁰ in a 20% yield based on the ammonium chloride consumed.

Preparation of B-trianilinoborazine

This compound has been obtained previously from the reaction of B-tri(butylthio)borazine (prepared from B-trichloroborazine and di(thiobutyl) lead) with aniline (overall yield from B-trichloroborazine was 64%¹¹). Direct interaction of B-trichloroborazine with aniline in the presence of triethylamine gave the desired B-trianilinoborazine in a 76% yield. Under nitrogen by-pass to B-trichloroborazine (9.84 g; 53.52 mmol) in benzene (80 ml) was added, at room temperature over 1.5 hr, a solution of aniline (14.95 g; 160.53 mmol) and triethylamine (16.25 g; 160.58 mmol) in benzene (60 ml). The colorless filtrate obtained after separation of triethylamine hydrochloride (20.60 g, 93% yield) on concentration gave the crude product. Recrystallization from hot benzene afforded B-trianilinoborazine (14.35 g, 76% yield), mp 209.5-212°C (lit. 204-206°C¹¹). Anal. Calcd. for C₁₈H₂₁N₆B₃: C, 61.10; H, 5.98; N, 23.75; B, 9.17; MW, 353.84. Found: C, 60.75; H, 6.05; N, 23.78; B, 9.48; MW 360.

Thermal Degradations

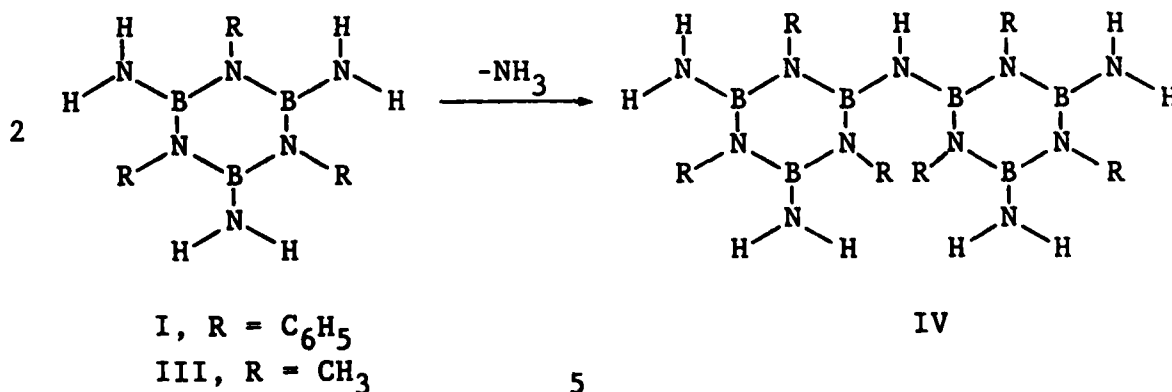
The degradations were performed under nitrogen atmosphere either using nitrogen by-pass or at a slightly lower (~ 550 mm Hg) than atmospheric pressure in a closed system. The volatile condensibles were collected in liquid nitrogen cooled traps; the separations and measurements were performed using standard vacuum line techniques. All the products were weighed, then identified and quantitated by infrared spectral analysis and combined gas chromatography and mass spectrometry. The involatile residues were subjected to infrared spectral analysis, thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). Melting characteristics and molecular weights, the latter of materials soluble in organic solvents, were also determined.

RESULTS AND DISCUSSION

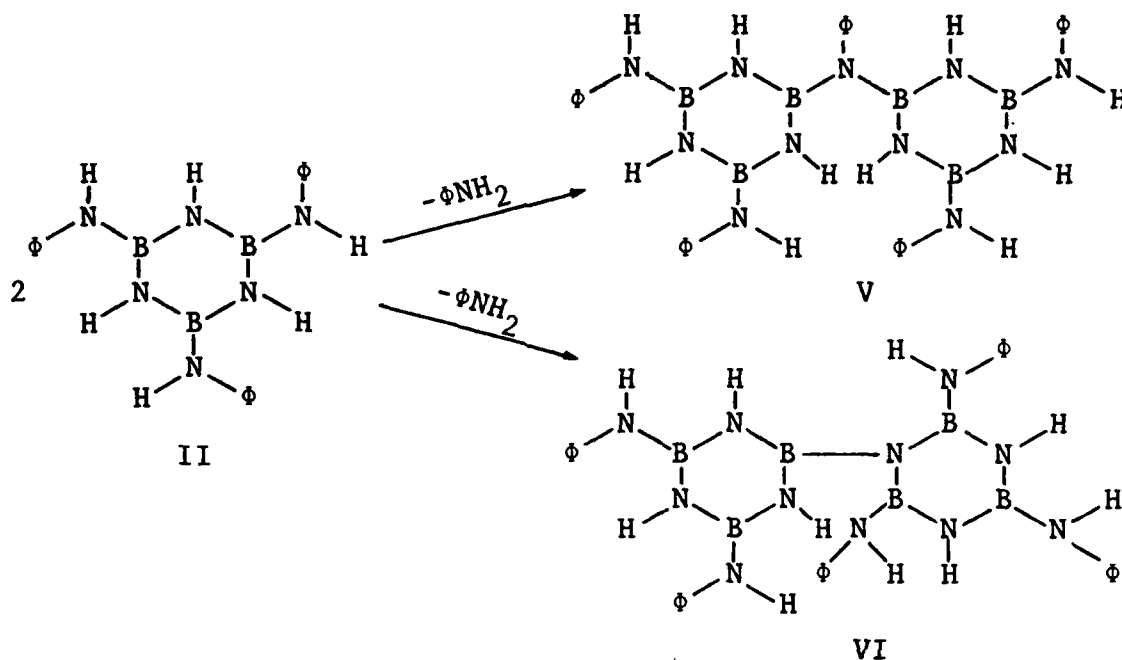
In the first phase of this undertaking, three borazines, namely B-triamino-N-triphenylborazine, I, B-trianilinoborazine, II, and B-triamino-N-trimethylborazine, III, were subjected to controlled stepwise thermal treatments. These are summarized in Table I. The thermal behavior of B-triamino-N-triphenylborazine was investigated by Toeniskoetter and Hall⁷ and Taniguchi, et al.³. The former authors reported to have formed pure boron nitride in a 27% yield from the borazine on heating at 300°C in vacuo for 18 hr; the weight of product was 9.8 mg. Attempts to duplicate this process were unsuccessful due to rapid sublimation of unreacted B-triamino-N-triphenylborazine even at 250°C. Taniguchi, et al.³ claim to have obtained boron nitride fiber by stepwise heating to 1800°C of a low melting (150-200°C) prepolymer formed upon 4 hr exposure to 250°C which was accompanied by a 50% loss of aniline. In our studies the product which resulted from the 250°C treatment

of B-triamino-N-triphenylborazine, following a loss of ~24% of the available aniline, softened only around 350°C (see tests 5 and 9). Based on the material's relative infusibility and its insolubility in organic solvents, it would appear that at 250°C some crosslinking is taking place. Unfortunately, at lower temperatures the reaction rate was negligible (see test 18). The isomer, B-trianilinoborazine, was found to undergo pyrolysis at much lower temperature than the B-amino compound (compare tests 17 and 18). Consequently, efforts were centered on pyrolysis of this compound and its mixtures with the B-amino isomer. It should be emphasized that these materials interacted readily at relatively low temperatures. Whether this behavior is due to the attack of B-trianilinoborazine upon the B-aminoborazine or whether the telomerization products of the B-anilino compound provide a solubilization medium is unknown.

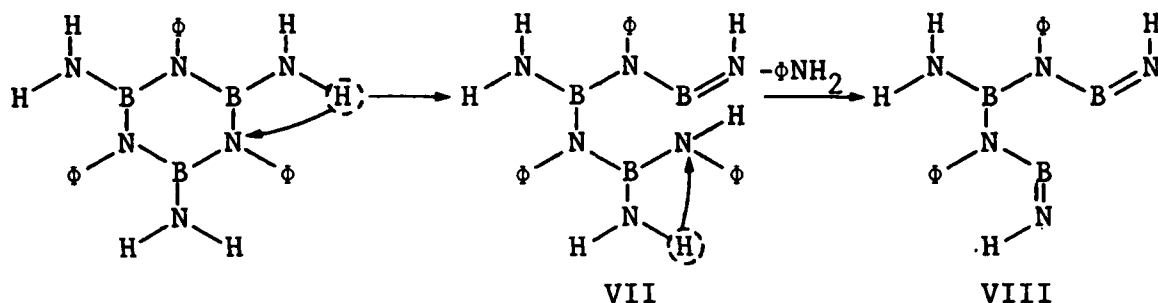
As evident from the Table I compilation, the initial degradation of the three borazines was invariably associated with ammonia formation. This was most pronounced in the case of B-triamino-N-trimethylborazine. On the other hand, none of the borazines under the degradation conditions employed yielded ammonia as the sole elimination product. Any formation of ammonia, of course, necessitates in later stages the elimination of a secondary amine if boron nitride is to be produced. For the B-aminoborazines, compounds I and III, one can visualize ammonia production via an intermolecular process, i.e.,



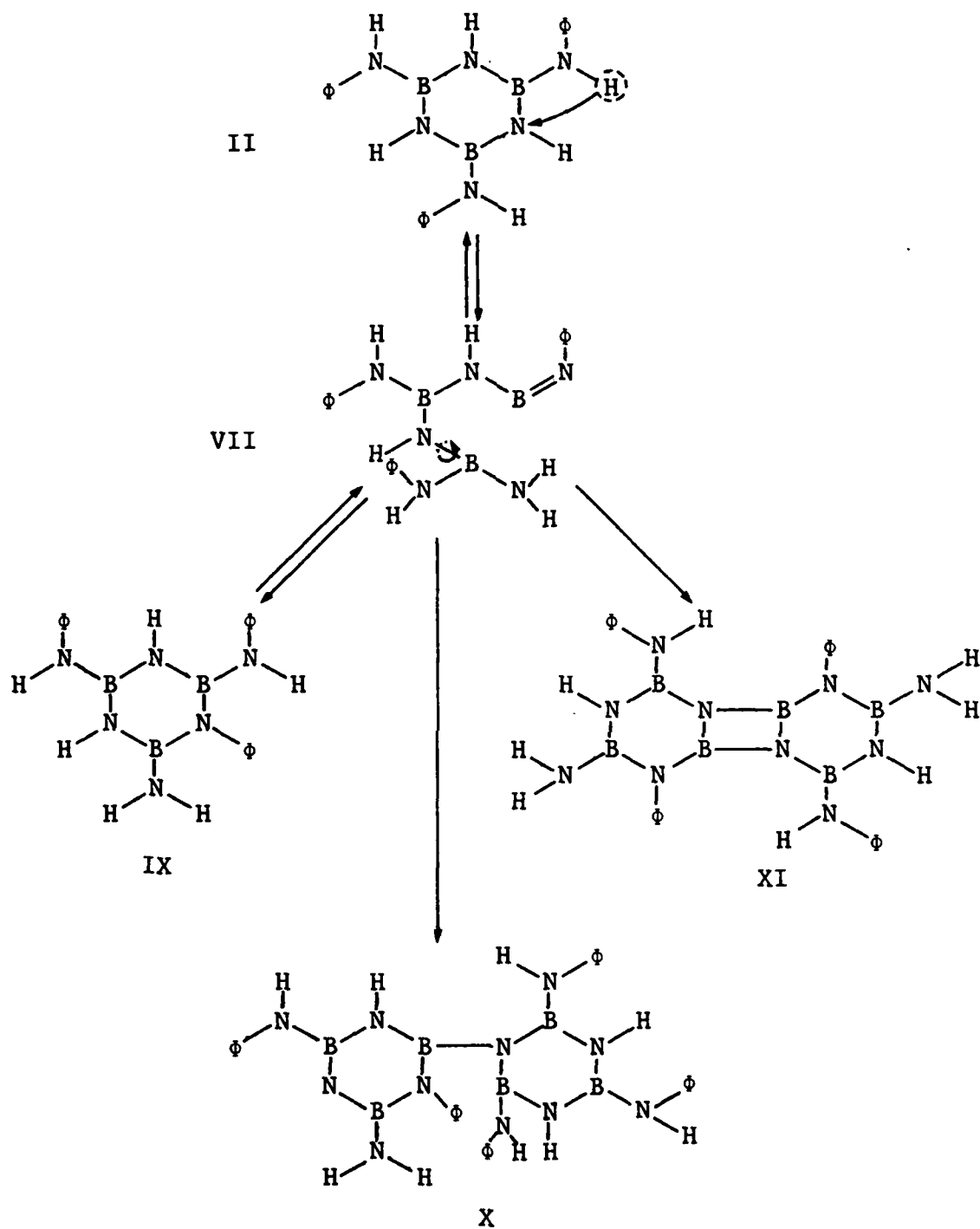
This process should be equally well applicable to aniline liberation from the B-trianilinoborazine where there exists also a possibility of direct B-N linkage establishment, i.e.



Aniline was found to be by far the major product of pyrolysis of the isomeric borazines, compounds I and II. After thermal treatment, the two isomers, following a loss of 18-50% of the theoretically available aniline, exhibited virtually identical infrared spectra, Figures 1 and 2. Yet, the infrared spectra of the two pure isomers are quite distinct as evident from Figures 3 and 4. It is of interest to note that small quantities of B-trianilinoborazine, II, were obtained as a sublimate during heat treatment at 250°C of the B-triamino-N-triphenylborazine. Both of these findings support strongly the ring opening mechanism postulated by Toeniskoetter and Hall⁷ for the aniline production by B-triamino-N-triphenylborazine, i.e.,



Once the ring is opened, it can polymerize by an elimination of ammonia or aniline or reclose to afford either the starting material or an isomer. The polymers, or rather initially dimers, as found from the molecular weight measurements, would be thus comprised of all the possible combinations of ring atoms substituted on boron by amino or phenylamino groups and on ring nitrogens by hydrogen or phenyl groups. The process depicted below for the anilino compound applies equally well to the amino isomer. It is not known whether this ammonia or amine (aniline) elimination takes place via an intra- or inter-molecular process. An intermediate such as VIII would, of course, result from an intramolecular reaction which must be followed either by polymerization or, as the present studies imply, attack of the intermediate upon an unopened ring. On the other hand, attack upon an unopened ring system by the product of the initial ring opening, as depicted by arrangement VII, is probably more plausible, in which case amine elimination would occur by an intermolecular process. Obviously, structures X and XI may be formed by either process; only the intermolecular reaction is shown below:



From the quantity of aniline evolved and the molecular weight of the residual pyrolysate, it is obvious that in the early stages of degradation, when up to 1/6 of the available aniline is eliminated, the singly bridged dimers, e.g. X, are preferentially produced. Further pyrolysis appears to lead to predominant formation of the doubly bridged structures, e.g. XI. A molecular weight of 780 was determined for the pyrolysate of a 1:1 mixture of the two isomeric borazines following the 51% loss of aniline (test 24). A tetramer formed by a single BN linkage between doubly bridged dimers, such as that shown by structure XI, would have a molecular weight of 950 and would result from a 41.7% loss of aniline. A corresponding doubly bridged arrangement would have a molecular weight of 857 and would require an aniline loss of 50%. Based on these data, one is tempted to speculate that the material produced is indeed the tetramer. Beyond this point, the elimination reaction slows down considerably. Up to 300°C, at best 53% of the available aniline could be obtained which corresponds to a 41.9% weight loss.

None of the pyrolysates gave boron nitride up to 1000°C based on thermogravimetric analysis. The highest combined weight loss of 66.7% registered by the B-trianilinoborazine was significantly lower than the 79.0% required for boron nitride formation, as shown in Table II. In all the instances, the residues were black confirming the presence of carbon or carbon-containing moieties.

It is believed that the formation of the rigid fused ring structures is responsible for the lack of further reaction at temperatures below 300°C. Eliminations beyond this stage would be expected to lead preferentially to crosslinking, not chain extension, thus making the subsequent loss of the potential residual aniline difficult with the end result being an arrangement resulting in a high char yield. It must be emphas-

ized that the work conducted here utilized bulk reactions where the surface to volume ratio was relatively low. Toeniskoetter and Hall⁷ worked only with minute quantities of material since the major portion of the borazine simply sublimed out of the hot zone, thus the surface to volume ratio was high. The same would apply to the fiber system employed by Taniguchi, et al.³. It is possible that under these conditions the escape of aniline is, at a minimum, more readily accomplished or that in thin layers the mechanism of final amine (aniline) elimination is entirely different. This does not however explain our inability to reproduce Taniguchi's results up to 250°C, which must have been performed in bulk.

ACKNOWLEDGEMENT

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TABLE I
Thermal Degradations of Borazines

Test No.	Compound ^a Type	mmol	Conditions		Volatile products		NH ₃ mmol	Residue	
			Temp °C	Time hr	RNH ₂ mmol	$\frac{b}{c}$		org. solv.	MW
3	I	2.91	250	4	1.48	17	1.00	soluble	173-177
16	I	1.33	250	4	1.00	24	0.52	insoluble	
5	I	2.89	250	8	3.60	42	0.46	insoluble	~340-350
9	I	2.98	250	4	1.33	15	0.31		
	-ditto-		250	4	1.23	14	0.05		
	-ditto-		300	4	0.76	8	-		
	-ditto-		300	20	0.28	3	-		
	Comb. C		250-300	32	3.60	40	0.36	insoluble	~350 partial
18	I	0.85	170	72	0.05	2	0.04		
10	II	3.07	150	4	0.48	5	-		
	-ditto-		220	5	4.06	44	0.26		
	-ditto-		300	25	0.38	4	-		
	Comb.		150-300	34	4.92	53	0.26		
17	II	2.76	170	72	2.63	32	0.26	soluble	590
23	II	2.15	150	4	0.34	5	0.02		155-158
	-ditto-		220	5	1.17	18	0.15		
	-ditto-		220	5	1.12	18	-		
	Comb.		150-220	14	2.63	41	0.17	soluble	540
24	II+I ^d	10.83	170	72	6.02	18	1.60	soluble	160-185
	-ditto-		220	6	8.09	25	0.48	soluble	560
	-ditto-		250	4	2.49	8	-	soluble	550
	-ditto-		270	4	0.31	1	-	soluble	780
	Comb.		170-270	86	16.91	52	2.08	partl. sol.	
6	III	2.72	105	4	-	-	0.18		
	-ditto-		100-200	11	-	-	0.68		
	-ditto-		300	12	1.20	15	0.46		
	Comb.		105-300	27	1.20	15	1.31		

a) For compounds I and II, R = C₆H₅; for compound III, R = CH₃.

b) Percent of the total amine available.

c) Total products formed during successive heating periods.

d) Equimolar mixture.

TABLE II
Comparison of Degradation Data

Material	Weight loss % up to 300°C	Weight loss % up to 1000°C	Weight loss % theory	Weight stabilization temp °C
$[\text{C}_6\text{H}_5\text{NB}(\text{NH}_2)]_3$ (I)	35.4	58.7	79.0	780
$[(\text{C}_6\text{H}_5\text{NH})\text{BNH}]_3$ (II)	43.6	66.7	79.0	800
1:1 mixture of I & II	40.3 ^a	61.4	79.0	800

a) The temperature here was 260°C.

LEGENDS FOR FIGURES

- Figure 1. Infrared spectrum of B-triamino-N-triphenylborazine after 4 hr at 250°C in N₂ (Nujol mull)
- Figure 2. Infrared spectrum of B-trianilinoborazine after 5 hr at 220-250°C in N₂ (Nujol mull)
- Figure 3. Infrared spectrum of B-triamino-N-triphenylborazine (double mull, Kel F oil/Nujol)
- Figure 4. Infrared spectrum of B-trianilinoborazine (double mull, Kel F oil/Nujol)

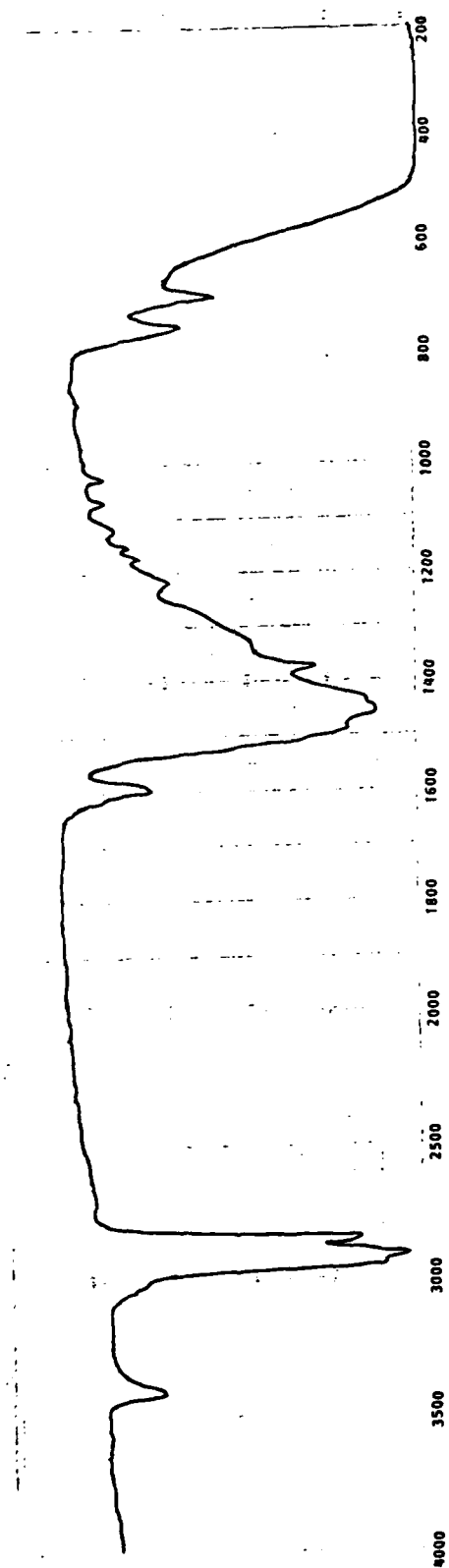


fig 1

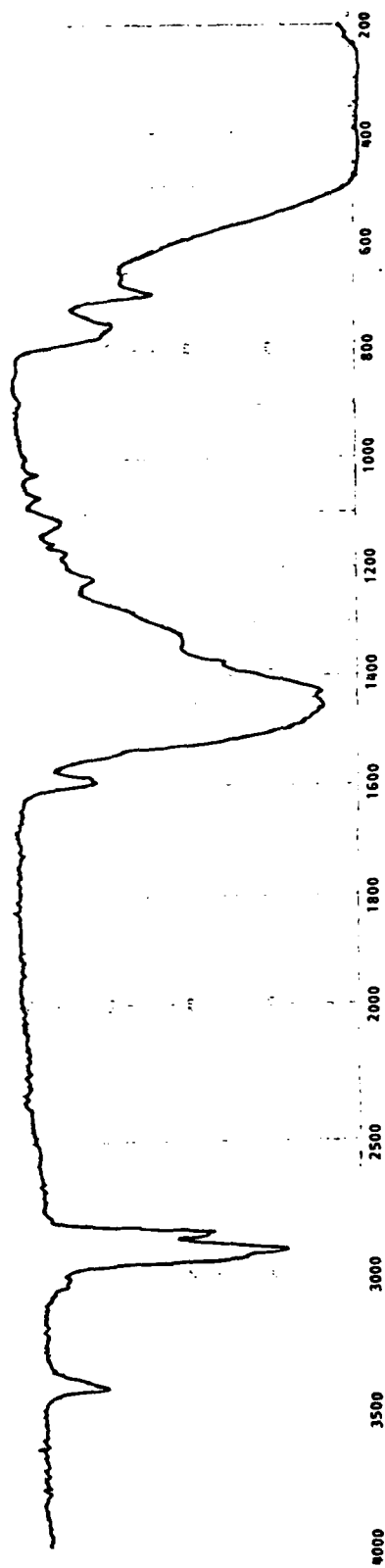


fig 2

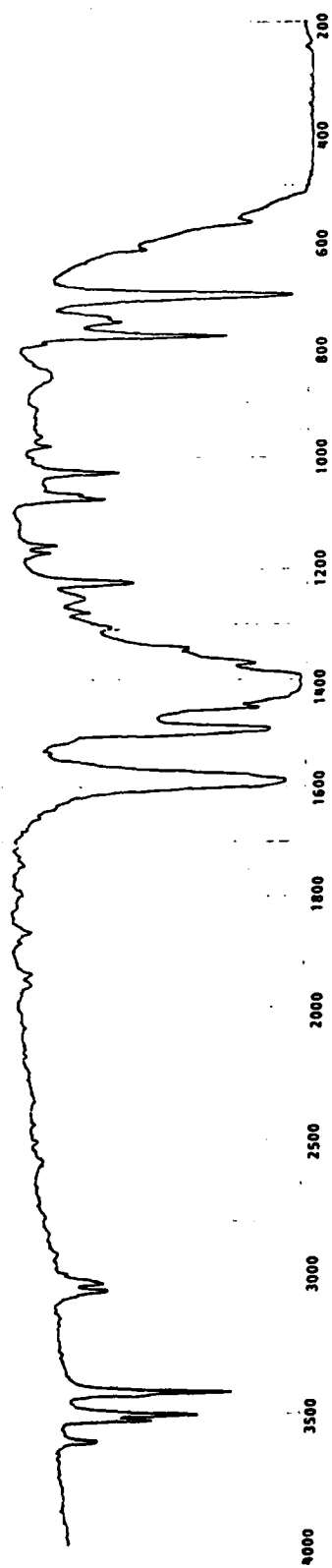


fig 3

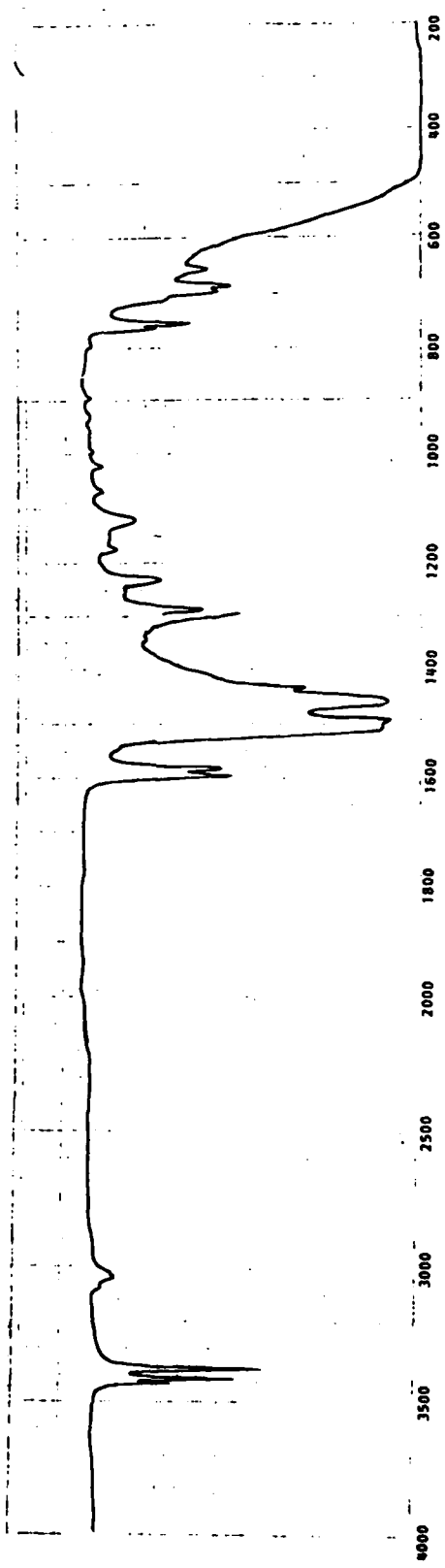


fig 4

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