hydrogen peroxide addition was resumed until a total of 35 ml. (0.35 mole) had reacted after a total of 63 min. Only intermittent cooling was needed after the sulfoxide state to maintain the temperature at 70–75°. Hydrogen peroxide was then added dropwise very slowly with slight heating to a total of 41 ml. (0.41 mole) over a further 65-min. period while testing with starch–iodide paper to find the point where there is slight excess over the theoretical stoichiometric amount (0.40 mole) of hydrogen peroxide needed to obtain the sulfone. The reaction was stopped when the last milliliter of hydrogen peroxide persisted for 15 min. Two layers formed on cooling, the pH of the water layer being 4.4. A trace of hydrogen peroxide was destroyed with 0.5 g. of sodium bisulfite. The reaction product was isolated and purified by dissolving the organic layer in benzene, extracting the aqueous layer with benzene, drying the combined extracts over anhydrous sodium sulfate and fractionally distilling to give 35.3 g. of **2-phenylsulfonylethanol** in 94.5% yield, b.p. 184° (4 mm.), $n^{24}\text{D}$ 1.5555. Literature constants[16] are b.p. 177° (2 mm.), $n^{11}\text{D}$ 1.5573. The structure was confirmed by infrared spectrum.

Anal. Calcd. for $C_8H_{10}O_3S$: C, 51.60; H, 5.41; S, 17.22. Found: C, 52.04; H, 5.57; S, 17.22.

A *vanadium* catalyst solution was prepared by dissolving 0.20 g. (0.0017 mole) of sodium orthovanadate (Na_3VO_4) in 65 ml. of distilled water. The pH of 12.5 was lowered to 1.8 by the addition of a few drops of dilute sulfuric acid to give a yellow solution. The catalyst solution was immediately added to a reactor with 30.8 g. (0.20 mole) of 2-phenylmercaptoethanol and heated to 67°. Hydrogen peroxide (31%) was added as before to the rapidly stirring mixture. The solution became clear after 16.2 ml. (0.162 mole) hydrogen peroxide had been added. Each time a drop of hydrogen peroxide entered the reaction solution, a momentary reddish color appeared which tended to persist when the addition rate was too fast. A negative starch–iodide test resulted when a pale yellow color was present. If there was no hydrogen peroxide present for a short time, a haze formed which was cleared by hydrogen peroxide addition. After the halfway point of the reaction had been reached, the temperature maintained itself spontaneously at 68–78° only when a relatively large excess of hydrogen peroxide was present. When a total amount of 39 ml. (0.39 mole) of hydrogen peroxide had been added over a 90-min. period, a negative starch–iodide test was obtained. The reaction was completed by adding 6 cc. more hydrogen peroxide in the course of another 65 min. At the end the solution had a reddish tinge; the pH was 1.3. Sodium bisulfite was added to destroy excess hydrogen peroxide. The The reaction product was isolated as before to give 31.8 g. (85%) of 2-phenylsulfonylethanol, b.p. 177–183° (3 mm.), $n^{25}\text{D}$ 1.5564–1.5570. This time employing ammonium metavanadate,[17] a second vanadium-catalyzed reaction was perfored by commencing at a pH of 5.45 and adding 18 ml. (0.18 mole) of hydrogen peroxide over a period of 42 min. A clear solution was obtained after this time which approximates the sulfoxide stage. The second reaction step did not proceed in a satisfactory fashion. The reaction was kept at 73–77° for a total reaction time of 298 min. The final pH was 4.4; no layering occurred on standing. The solution was salted and the product was isolated as before. Infrared spectra and C and H analyses on the distillation fractions showed the product to be a mixture of sulfoxide and sulfone.

Preparation of 3,3′-Sulfonyldipropionic Acid.—The preparation was carried out using a tungstic acid catalyst solution in water as described above by using 3,3′-thiodipropionic acid to react with essentially a stoichiometric amount of 31% hydrogen peroxide at 60° in the course of 2.5 hr. A white precipitate formed during the second half of the peroxide addition. After cooling to room temperature, the white solid was filtered, and dried in a vacuum at 80° to give a 91.3% yield, m.p. 222–224° (lit.,[18] m.p. 223–225°). The pH during the oxidation was approximately 3.

2,2′-Sulfonyldiethanol was prepared from 2,2′-thiodiethanol and hydrogen peroxide using a tungstic acid catalyst, prepared as already described, to give yields of 97–99%. A molybdic acid catalyst could be substituted for the tungstic acid under the same pH and temperature conditions to give similar results.

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[16] A. H. Ford-Moore, R. A. Peters, and R. W. Wakelin, *J. Chem Soc.*, 1754 (1949).

[17] N. V. Sidgwick, *loc. cit.*, p. 811 discloses that ortho-, pyro- or metavanadate in solution interconvert into the same form, the form being a function of salt and hydrogen ion concentration.

[18] R. Dahlbom, *Acta Chem. Scand.*, **5**, 690 (1951); *Chem. Abstr.*, **46**, 431 (1952).

Vinyl Derivatives of Metals. XVII. Radical Additions to B-Trivinyl-N-triphenylborazine[1]

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B-Trivinyl-N-triphenylborazine (I) has been shown to undergo radical-initiated copolymerization with other vinyl monomers, although homopolymerization was inhibited by steric interference by the phenyl groups attached to the nitrogen atoms.[3] We report here the addition of a number of reagents to the vinyl groups of I in the presence of radical initiators.

The radical addition of bromotrichloromethane and carbon tetrachloride to the dibutyl ester of vinylboronic acid has been reported.[4] In a similar manner, we were able to effect smooth addition of bromotrichloromethane and of carbon tetrabromide to the vinylborazine I in the presence of benzoyl peroxide.

$$3CX_3Br + [CH_2{=}CHBNC_6H_5]_3 \longrightarrow [CX_3CH_2CHBrBNC_6H_5]_3 \ (X = \text{Cl and Br})$$

Isolation of the carbon tetrabromide adduct in the pure state was complicated by the tendency of this borazine to form relatively stable benzene and carbon tetrachloride solvates. Carbon tetrachloride and chloroform did not react with I at 100° in the presence of benzoyl peroxide. A reaction between I and carbon tetrachloride did occur at 140° when *t*-butyl peroxide was used as initiator, but no stable, crystalline product could be isolated. Attempted addition of $(CH_3)_3Si$-$CH{=}CHCBr_3$ to I in the presence of either peroxide also was unsuccessful.

Benzenethiol addition to I, initiated by benzoyl peroxide, also proceeded readily, giving $[C_6H_5SCH_2CH_2BNC_6H_5]_3$. Addition of aliphatic mercaptans to vinylboronate esters had been recorded previously.[4]

The products of the reaction of I with anhydrous hydrogen bromide in the presence of benzoyl peroxide depended on the solvent used. In benzene the expected B-tris(β-bromoethyl)-N-triphenylborazine was obtained in good yield. In diethyl ether, on the other hand, complete rupture of the borazine ring system was observed, the only crystalline product isolated being anilinium bromide. The fate of the boron fragment of the ring was not established. This difference in behavior very likely is a reflection of the different states of HBr in these solvents, HBr being present essentially in the molecular form in benzene and as the strongly acidic oxonium salt in ether. The structure of the HBr-I adduct was shown to be $[BrCH_2CH_2BNC_6H_5]_3$. Its n.m.r. spectrum in the aliphatic region consisted of two triplets at 1.20 and 2.76 p.p.m. downfield from tetramethylsilane,[5] which is consistent with the $—CH_2CH_2—$

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[1] Part XVI: D. Seyferth and M. Takamizawa, *Inorg. Chem.*, to be published; also Part IV of the series "Borazine Derivatives."

[2] (a) Alfred P. Sloan Research Fellow; (b) Fellow of the M.I.T. School for Advanced Study, 1961–1962; on leave from the Shin-Etsu Chemical Industry Company, Ltd., Tokyo.

[3] J. Pellon, W. G. Deichert, and W. M. Thomas, *J. Polymer Sci.*, **55**, 153 (1961).

[4] D. S. Matteson, *J. Am. Chem. Soc.*, **82**, 4228 (1960).

[5] The n.m.r. spectrum was recorded in carbon tetrachloride solution using a Varian Associates A60 n.m.r. spectrometer.

structure, but not with the alternative —CH(CH_3)— structure. This terminal addition of Br to the vinyl groups of I suggests strongly that the assumed structures in the other cases mentioned above, [CX_3CH_2-$CHBrBNC_6H_5$]$_3$ and [$C_6H_5SCH_2CH_2BNC_6H_5$]$_3$, are the correct ones.

Attempted condensation of [$BrCH_2CH_2BNC_6H_5$]$_3$ with Grignard reagents resulted in β-elimination. Thus the action of phenyl- and ethylmagnesium bromide on I led to hexaphenylborazine and B-triethyl-N-triphenylborazine, respectively.

$$3C_6H_5MgBr + [BrCH_2CH_2BNC_6H_5]_3 \longrightarrow [C_6H_5BNC_6H_5]_3 + 3CH_2{=}CH_2 + 3MgBr_2$$

Bromine reacted with I in carbon tetrachloride solution at room temperature to give [$BrCH_2CHBr$-BNC_6H_5]$_3$ in good yield.

Experimental[6]

Additions to B-Trivinyl-N-triphenylborazine (I).—All radical reactions performed at atmospheric pressure were carried out in a 100-ml. three-necked flask equipped with a magnetic stirrer, reflux condenser, thermometer and dropping funnel under a nitrogen atmosphere.

(1) **Bromotrichloromethane.**—To a mixture of 3.86 g. (0.01 mole) of I[3], m.p. 186–187°, and 5.95 g. (0.03 mole) of bromotrichloromethane in 12 g. of toluene at 80–85° was added slowly with stirring a solution of 0.12 g. of benzoyl peroxide in 5.95 g. of bromotrichloromethane. A vigorous, exothermic reaction commenced, and the temperature rose rapidly to 110°. The mixture was maintained at 110–115° for 90 min. Excess volatiles were removed at reduced pressure. Addition of hexane to the residue gave a yellow solid, m.p. 208–209.5°, 7.27 g. (74%). Recrystallization from ether–methanol resulted in white crystals of the desired compound, m.p. 212–213°.

Anal. Calcd. for $C_{27}H_{24}N_3Cl_9Br_3B_3$: C, 33.02; H, 2.46; N, 4.31. Found: C, 33.13; H, 2.46; N, 4.08.

(2) **Carbon Tetrabromide.**—To a mixture of 5 mmoles of I and 15 mmoles of carbon tetrabromide in 6 g. of toluene at 90° was added a solution of 0.06 g. of benzoyl peroxide and 15 mmoles of carbon tetrabromide in 6 g. of toluene. The temperature rose spontaneously to 114°, and the mixture was stirred at this temperature for 2 hr. Toluene was removed at reduced pressure. Addition of methanol to the residue gave a light yellow solid, m.p. 222–223° dec., 6.3 g. Recrystallization from benzene resulted in white crystals, m.p. 224–225° dec. The analytical results indicated that the substance was a benzene solvate of the expected product.

Anal. Calcd. for $C_{27}H_{24}N_3Br_{12}B_3$: C, 23.46; H, 1.75; N, 3.04. Calcd. for $C_{27}H_{24}N_3Br_{12}B_3 \cdot C_6H_6$: C, 26.54; H, 2.09. Found: C, 26.32; H, 1.96.

Recrystallization of the crude reaction product from carbon tetrachloride–methanol gave a carbon tetrachloride solvate, m.p. 237–238° dec.

Anal. Calcd. for $C_{27}H_{24}N_3Br_{12}B_3 \cdot CCl_4$: Cl, 9.23; Br, 62.44. Found: Cl, 9.45; Br, 62.31.

The carbon tetrachloride solvate was heated for 8 hr. at 140° *in vacuo* to give the unsolvated compound, m.p. 241–241.5° dec.

Anal. Found: C, 23.66; H, 1.88; N, 3.04.

(3) **Attempted Addition of Carbon Tetrachloride.**—A mixture of 5 mmoles of I and 30 mmoles of carbon tetrachloride containing 0.07 g. of *t*-butyl peroxide was sealed in a thick-walled Pyrex tube and heated at *ca.* 90–100° for 24 hr. A yellow solution resulted. Evaporation of volatiles left a yellow solid, melting range 146–190° dec. Attempted recrystallization from ether–methanol gave a white solid, which, however, decomposed rapidly to a red liquid. A similar reaction was carried out at 130–140° for 4 hr., and a yellow solid of melting range 123–173° dec. was obtained. Two attempts (atmospheric pressure in toluene solution at *ca.* 100° and in a sealed tube at higher temperature) were made to effect the addition of carbon tetrachloride to I using benzoyl peroxide as initiator. In both cases the starting borazine was recovered unchanged in high yield.

(4) **Addition of Benzenethiol.**—To 5 mmoles of I and 15 mmoles of benzenethiol in 6 g. of toluene at 80° was added slowly a solution of 0.1 g. of benzoyl peroxide and 15 mmoles of benzenethiol in 4 g. of toluene. The mixture was heated at 115° for 2.5 hr. Volatiles were removed at reduced pressure, and methanol added to the residue to give a white solid, m.p. 127–131°, 3.3 g. (90%). Recrystallization from ether–methanol gave white crystals, m.p. 134–135°.

Anal. Calcd. for $C_{42}H_{42}N_3S_3B_3$: C, 70.31; H, 5.90; N, 5.86. Found: C, 70.65; H, 5.65; N, 5.96.

(5) **Addition of Hydrogen Bromide.** (a) **In Benzene.**—Anhydrous hydrogen bromide was bubbled into a solution of 13 mmoles of I and 0.1 g. of benzoyl peroxide in 80 ml. of benzene for 2 hr. at room temperature. The mixture was stirred for another 2 hr. and unchanged hydrogen bromide then was removed at reduced pressure. The reaction mixture was filtered to remove 0.12 g. of white solid. The latter, m.p. 289–291° dec. after recrystallization from methanol–toluene, was identified as anilinium bromide (see part b). The light yellow filtrate was concentrated *in vacuo* and methanol was added to the residue to precipitate 6.5 g. (80%) of white crystalline solid, m.p. 164–165°. Recrystallization from benzene–hexane gave material of m.p. 165–166°, [$BrCH_2CH_2BNC_6H_5$]$_3$.

Anal. Calcd. for $C_{24}H_{27}N_3Br_3B_3$: C, 45.77; H, 4.32; N, 6.67. Found: C, 45.81; H, 4.44; N, 6.90.

(b) **In Diethyl Ether.**—Anhydrous hydrogen bromide was passed for 2 hr. at room temperature into a solution of 2.5 mmoles of I and 0.06 g. of benzoyl peroxide in 25 ml. ether. A white solid precipitated. Unchanged hydrogen bromide and the ether solvent were removed *in vacuo,* and hexane was added to the residue. Light brown crystals, 1.2 g., m.p. 285–289° dec. were obtained. Recrystallization from methanol-benzene gave white, easily sublimable crystals, m.p. 289–291° dec., which were insoluble in non-polar solvents. A mixture melting point with authentic material and combustion analysis established their identity as anilinium bromide.

Anal. Calcd. for C_6H_8NBr: C, 41.40; H, 4.63; N, 8.05. Found: C, 41.06; H, 4.56; N, 8.17.

Reactions of B-Tris(β-bromoethyl)-N-triphenylborazine with Grignard Reagents.—To *ca.* 23 mmoles of phenylmagnesium bromide in 20 ml. of tetrahydrofuran was added a solution of 2.1 g. (3.3 mmoles) of [$BrCH_2CH_2BNC_6H_5$]$_3$ in 20 ml. of tetrahydrofuran at room temperature, and the resulting mixture was heated at reflux for 4.5 hr., cooled, and subsequently hydrolyzed with saturated ammonium chloride solution. Evaporation of the organic layer and addition of methanol to the residue gave 1.45 g. of white solid. Recrystallization from toluene resulted in white needles, m.p. >400°. The compound's infrared spectrum suggested that it is hexaphenylborazine (m.p. 413–415°[7]).

Anal. Calcd. for $C_{36}H_{30}N_3B_3$: C, 80.51; H, 5.63; N, 7.83. Found: C, 80.72; H, 5.87; N, 7.39.

A similar reaction with ethylmagnesium bromide in tetrahydrofuran gave 0.79 g. of B-triethyl-N-triphenylborazine, m.p. 162–163.5°.

Anal. Calcd. for $C_{24}H_{30}N_3B_3$: C, 73.34; H, 7.69; N, 10.69. Found: C, 73.31; H, 7.80; N, 10.59.

Bromination of I.—To 0.96 g. (2.5 mmoles) of I in 20 ml. of carbon tetrachloride was added with stirring 1.25 g. (7.8 mmoles) of bromine in 15 ml. of carbon tetrachloride. Evaporation of the resulting light orange solution left a white solid, which was recrystallized from benzene–methanol to give [$CH_2BrCHBr$-BNC_6H_5]$_3$, m.p. 257–258°, 1.63 g. (76%).

Anal. Calcd. for $C_{24}H_{24}N_3Br_6B_3$: C, 33.26; H, 2.80; N, 4.85. Found: C, 33.35; H, 2.75; N, 5.06.

Preparation of 3,3,3-Tribromo-1-propenyltrimethylsilane.—A mixture of 5.5 g. (0.055 mole) of trimethylvinylsilane, 3.6 g. of carbon tetrabromide and 0.1 g. of benzoyl peroxide was added to 13 g. of carbon tetrabromide (0.05 mole total) in 10 g. of toluene at 80–100°. The reaction mixture was heated at 114–115° for 2 hr. Toluene was removed from the yellow-brown solution. The residue was distilled to give 19.0 g. of colorless liquid distilling between 55–113° at 4 mm. Fractional distillation of the crude distillate resulted in a small forerun and 16 g. (74%) of $(CH_3)_3SiCHBrCH_2CBr_3$, b.p. 93–98° at 0.7 mm. Another distillation gave pure material, b.p. 96–97° at 0.7 mm., n^{25}_D 1.5602.

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[6] All reactions were carried out under an atmosphere of prepurified nitrogen. Analyses by Dr. S. M. Nagy (M.I.T.), the Schwarzkopf Microanalytical Laboratory, Woodside, N. Y., and A. Schoeller, Kronach, Ofr.

[7] S. J. Groszos and S. F. Stafiej, *J. Am. Chem. Soc.*, **80**, 1357 (1958).

Anal. Calcd. for $C_6H_{12}Br_4Si$: C, 16.68; H, 2.80. Found: C, 16.86; H, 2.92.

A mixture of 21.6 g. (0.05 mole) of $(CH_3)_3SiCHBrCH_2CBr_3$ and 20.2 g. (0.2 mole) of triethylamine was heated at *ca.* 90° for 20 hr. After a short time white solid precipitated, and the solution became yellow, later brown. The mixture was filtered to remove 11.7 g. of triethylammonium bromide (m.p. 252–254° after recrystallization from methanol–ether). The filtrate was distilled to give 12 g. of light yellow liquid, b.p. 106–115° at 6 mm. Fractional distillation of the latter resulted in 9 g. (51%) of pure product, b.p. 94° at 3 mm., $n^{25}D$ 1.5458.

Anal. Calcd. for $C_6H_{11}Br_3Si$: C, 20.53; H, 3.16. Found: C, 21.11; H, 3.07.

The infrared spectrum was consistent with the assumed structure, $(CH_3)_3SiCH{=}CHCBr_3$, showing bands at 1592 cm.$^{-1}$ ($\nu_{C=C}$), 1250, 840 and 760 cm.$^{-1}$ (trimethylsilyl group). The absorption at 990 cm.$^{-1}$, indicative of a *trans*-1,2-disubstituted olefin, was very weak.

This compound did not react with I in toluene solution in the presence of benzoyl or *tert*-butyl peroxide at temperatures up to 145°.

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The Lithium Aluminum Hydride Reduction of Various Tetrahydropyridazine-1,2-dicarboxylic Acid Esters

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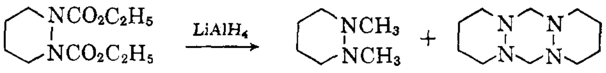
Several authors have described the Diels–Alder reaction between various dienes and azodicarboxylic acid esters and the subsequent reduction of the adducts to the tetrahydropyridazine-1,2-dicarboxylic esters.[1–8] This paper reports our investigation into the lithium aluminium hydride ($LiAlH_4$) reduction of certain of these esters.

The lithium aluminum hydride reduction of carbamates to the corresponding N-methyl compounds has been described,[9] but apparently this reaction has not been extended to hydrazino diesters. When diethyl tetrahydropyridazine-1,2-dicarboxylate[3,7] (I) was subjected to reduction by lithium aluminum hydride in ether, the major product formed was indeed 1,2-dimethylhexahydropyridazine (II). In addition, a small amount of a second product was obtained, which by analysis and infrared studies seemed to be 6*H*,13*H*-octahydrodipyridazino[1,2-*a*:1′,2′-*d*]-*s*-tetrazine (III).



I II III

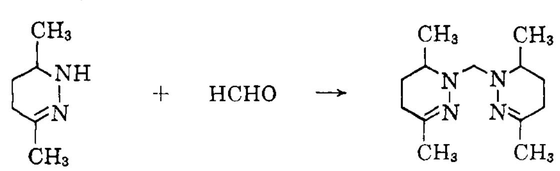
Confirmation of structure III was obtained by qualitatively identifying formaldehyde (as its 2,4-DNPH derivative) as one of the acid hydrolysis products of III and also by synthesis of III from hexahydropyridazine[3,7] and formaldehyde, as has been recently reported.[10] The product from the latter reaction was shown to be identical with that from the lithium aluminum hydride reaction by infrared comparison and mixed melting point.

Other esters of type I reduced by lithium aluminum hydride were the 4-methyl,[7] 4-isohexyl and 3,6-endomethylene[1] analogs. The products obtained along with several methiodide salts, are listed in Table I. The Diels–Alder adduct from chloroprene and diethyl azodicarboxylate was found to be unstable, liberating acidic vapors on standing. Catalytic reduction of the double bond in this adduct resulted in concurrent removal of the chlorine atom as well to give compound I.

The Diels–Alder addition between 1-methoxy-1,3-butadiene and diethyl azodicarboxylate gave the desired adduct, diethyl 3-methoxy-3,6-dihydro-1,2-pyridazinedicarboxylate, in high yield. Saturation of the double bond in this compound also proceeded satisfactorily. Attempted hydrolysis and decarboxylation of the diethyl 3-methoxy-3,4,5,6-tetrahydro-1,2-pyridazinedicarboxylate to the hexahydropyridazine caused complete disruption of the compound, due, undoubtedly to its hemiacetal type structure.

The reaction between diethyl azodicarboxylate and 1-diethylamino-1,3-butadiene[11] appeared to take place since the color of the diester was discharged, but no product could be isolated from the reaction mixture.

Substituted analogs of III were made by condensing various hexahydropyridazines with formaldehyde. (See Table II.) Using 3,6-dimethylhexahydropyridazine,[12] unsuccessful attempts were made to carry out this condensation with acetone, heptaldehyde, furfural, and thiophene carboxaldehyde in place of formaldehyde. The condensation of 2,3-diazabicyclo[2.2.1]heptane[1] with formaldehyde was likewise unsuccessful. It was found possible however, to effect the formaldehyde condensation with 3,6-dimethyl-1,4,5,6-tetrahydropyridazine[12] (IV).



IV

This reaction may thus have some utility in determining the positions of unsaturation in certain partially unsaturated nitrogen heterocycles.

It seems likely that the process by which III is formed from I must be similar to that by which III is

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