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Chemistry of Decaborane-Phosphorus Compounds. IV. Monomeric, Oligomeric and Cyclic Phosphino-Carboranes.

by

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Olin Mathieson Chemical Corporation
Organics Division
New Haven, Connecticut

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CONTRIBUTION FROM THE ORGANICS DIVISION

OLIN MATHIESON CHEMICAL CORPORATION

Chemistry of Decaborane-Phosphorus Compounds. IV. Monomeric, Oligomeric and Cyclic Phosphino-Carboranes.[1,2]

By Roy P. Alexander and Hansjuergen Schroeder

Received............

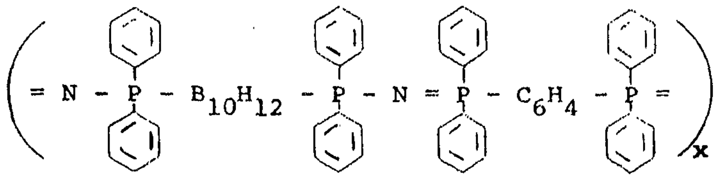
Dilithiocarborane, $B_{10}H_{10}C_2Li_2$, was allowed to react with chlorophosphines containing one, two, and three chlorine atoms. $(C_6H_5)_2PCl$ and $C_6H_5PCl_2$ gave the expected disubstituted compounds, $B_{10}H_{10}C_2[P(C_6H_5)_2]_2$ and $B_{10}H_{10}C_2(C_6H_5P{\cdot}Cl)_2$, respectively. $B_{10}H_{10}C_2Li_2$ and PCl_3, however, formed a compound having two phosphorus atoms and two carborane nuclei in a six membered ring. This unique ring system also arose from the reaction of $B_{10}H_{10}C_2Li_2$ with $B_{10}H_{10}C_2$-$(C_6H_5P{\cdot}Cl)_2$. Subsequent reactions of such molecules with ammonia and NaN_3 are described. The cyclic bis-azide, $(B_{10}H_{10}C_2P{\cdot}N_3)_2$, and p-$[P(C_6H_5)_2]_2C_6H_4$ reacted to form a PNP bonded oligomer.

In previous papers of this series we reported the synthesis of bifunctional decaborane-phosphine monomers[3] and their conversion into linear polymers[1]. The high-thermal and hydrolytical stability of PNP bonded decaborane-phosphine polymers[1], especially of compound I, made them an obvious object for further investigation.

(1) Preceding communication: H. Schroeder, J. R. Reiner, and T. A. Knowles, Inorg. Chem., 2, 393 (1963).

(2) Presented before the Inorganic Division of The American Chemical Society at the 145th Meeting at New York, N.Y., Sept. , 1963.

(3) H. Schroeder, J. R. Reiner, and T. L. Heying, Inorgan. Chem., 1, 618 (1962).

I  (x = ~ 20)

Since it was established[1] that compound I lost hydrogen from the decaborane nuclei under crosslinking at 340°, a logical extension of this work with regard to improving heat stability was the replacement of those hydrogens by atoms more firmly bonded to boron such as chlorine and fluorine. Initial attempts to chlorinate the starting monomer $B_{10}H_{10}\left[(C_6H_5)_2P{\cdot}Cl\right]_2$ met with failure. However, before a more thorough halogenation study of compounds containing the $B_{10}H_{12}$ unit was conducted, attention was turned to the easily chlorinated carborane[4] moiety instead.

Interest in this laboratory has been focused recently on the chlorination of carborane resulting in polychlorocarboranes such as $B_{10}Cl_{10}C_2H_2$.[5] The potential utility of the chlorinated carborane nucleus for the formation of PNP bonded carborane polymers led us to initiate an investigation aimed at the preparation of difunctional phosphorus containing carborane and polychlorocarborane derivatives. The present study describes the first part of our research dealing with the synthesis of chlorophosphino-carboranes followed by the application of the PNP polymer scheme to those compounds obtained containing the unchlorinated carborane nucleus.

The ability of carborane to undergo direct lithiation upon treatment with n-butyllithium[6] provided dilithiocarborane, an appropriate starting compound for reactions with various chloro-

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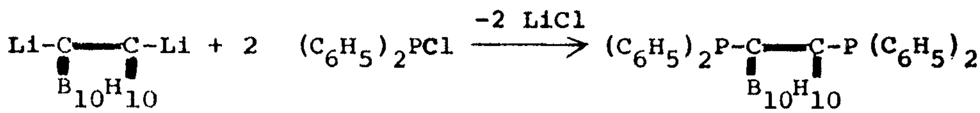
(4) For brevity, the term carborane is used for 11,12-dicarba-dodecaborane; for structure of carborane see reference 4.

(5) H. Schroeder, T. L. Heying, and J. R. Reiner, Inorg. Chem., ... (1963).

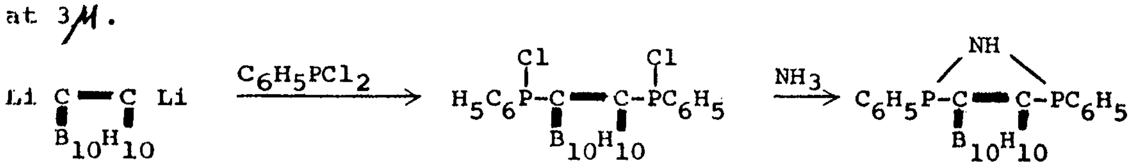
(6) T. L. Heying, J. W. Ager, Jr., S. L. Clark, R. P. Alexander, S. Papetti, J. A. Reid, and S. I. Trotz, submitted for publication.

phosphines. In a prototype experiment, methathesis was found to occur between dilithiocarborane and diphenylchlorophosphine to give bis(diphenylphosphino)-carborane.[7]



Having demonstrated the fundamental reaction, the dilithio salt was reacted with phenyldichlorophosphine. As expected, two molar equivalents of $C_6H_5PCl_2$ reacted with one equivalent of $B_{10}H_{10}C_2Li_2$ to form the desired bis-chloro compound $B_{10}H_{10}C_2(C_6H_5P{\cdot}Cl)_2$.

To determine the reactivity of the phosphorus bound chlorine atoms, ammonia was passed, at room temperature, into a solution of $B_{10}H_{10}C_2(C_6H_5P{\cdot}Cl)_2$ in benzene. Ammonolysis occured, but instead of the expected bis-amino compound $B_{10}H_{10}C_2\left[P(C_6H_5)NH_2\right]_2$, a cyclic diphospha(III)azane, $B_{10}H_{10}C_2\left[(PC_6H_5)_2NH\right]$, was recovered in 70% yield. The infrared spectrum was consistent with this formulation in showing only a single N-H stretching absorption at 3μ.



On the other hand, treatment of $B_{10}H_{10}C_2(C_6H_5P{\cdot}Cl)_2$ with sodium azide in ethanol at 0° gave the desired bis-azide, $B_{10}H_{10}C_2(C_6H_5P{\cdot}N_3)_2$, m.p. 126-128° dec. In benzene solution the compound undergoes decomposition at approximately 40° with evolution of nitrogen yielding a product, the analysis of which is in general agreement with the formula $(=NP(C_6H_5)-CB_{10}H_{10}C-(C_6H_5)PN=)_x$. The decomposition is promoted by the addition of

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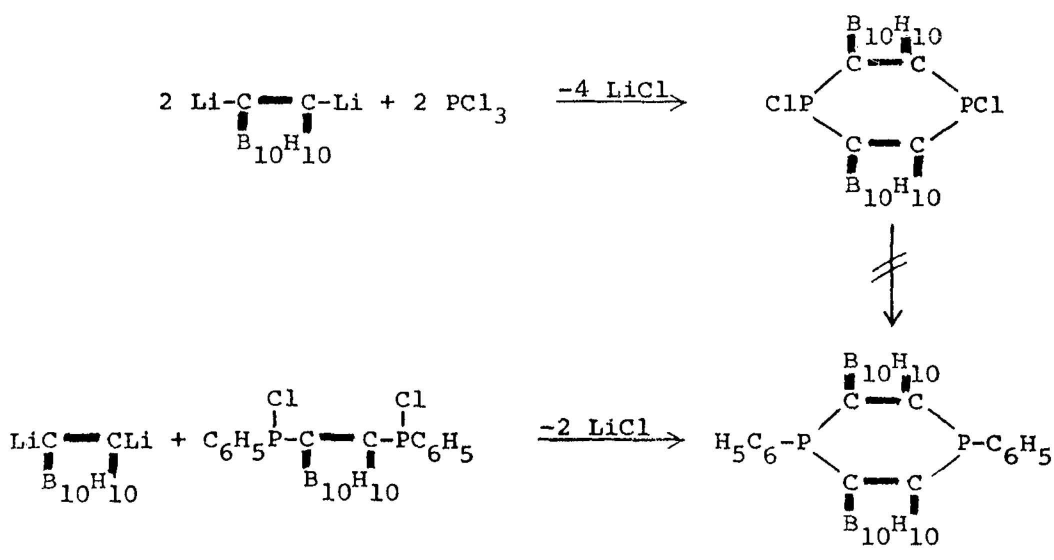
(7) The notation $-C{-}C-$ over $B_{10}H_{10}$ will be employed throughout this publication to designate the structure of the carboranyl grouping.

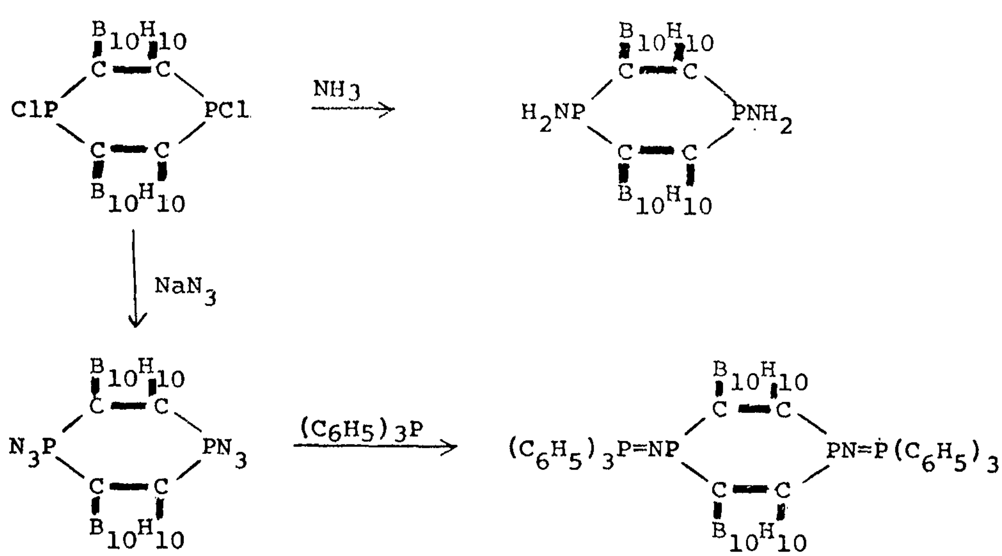
a phosphine such as $(C_6H_5)_3P$, since in their presence nitrogen evolution begins at 10° to 20°. The phosphines do not interact with this phosphorus(III)bis-azide, a finding in sharp contrast to the easy formation of phosphorus-nitrogen double bonds[1] observed in the reaction of $B_{10}H_{10}\left[(C_6H_5)_2P \cdot N_3\right]_2$, a compound having pentavalent phosphorus, with phosphines.

In view of these results it appeared of interest to investigate next the reaction of $B_{10}H_{10}C_2Li_2$ with PCl_3. It was found that equimolar amounts of these reactants form a product which analyzes as $(B_{10}H_{10}C_2PCl)_x$. On the evidence of an accurate cryoscopic molecular weight determination corroborated by mass spectral analysis, a cyclic structure containing two carborane moieties and two phosphorus atoms in the ring was assigned for this reaction product. Another example of this diphosphacyclohexane system was obtained by reaction of $B_{10}H_{10}C_2(C_6H_5P \cdot Cl)_2$ with $B_{10}H_{10}C_2Li_2$ to give the diphenyl substituted derivative. However, the dichloro-compound has resisted attempts to convert to the diphenyl compound by either Friedel-Crafts, Grignard, or aryllithium procedures.



In a comparison of the carborane infrared spectrum with those of $B_{10}H_{10}C_2(C_6H_5P\cdot Cl)_2$ and $(B_{10}H_{10}C_2P\cdot Cl)_2$ (see Fig. 1) it is of interest to note that not only does the C-H stretching frequency disappear, but also the carborane and chlorocarborane characteristic absorptions[5] between 8 and 9 μ are not present. The simplicity of the spectrum of the cyclic bis-chloro-compound also indicates high symmetry for this molecule.

Treatment of the cyclic bis-chloro compound with ammonia and with sodium azide, respectively, afforded in good yields the cyclic bis-amino compound, $(B_{10}H_{10}C_2P\cdot NH_2)_2$, and the cyclic bis-azide, $(B_{10}H_{10}C_2P\cdot N_3)_2$. Unlike the preceding bis-azide, $B_{10}H_{10}C_2(C_6H_5P\cdot N_3)_2$, it proved to be stable in refluxing benzene and reacted with triphenylphosphine according to the following equation:

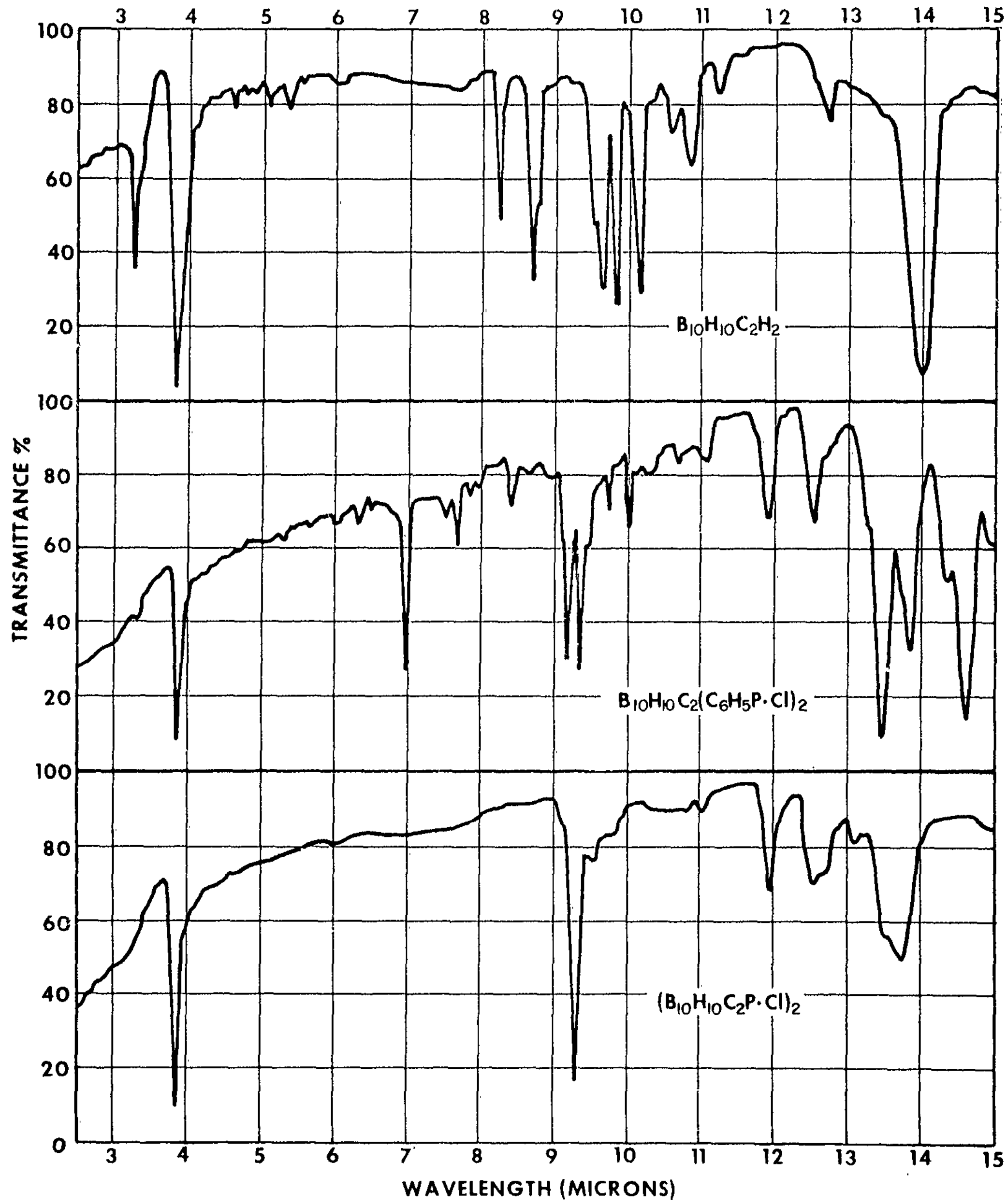


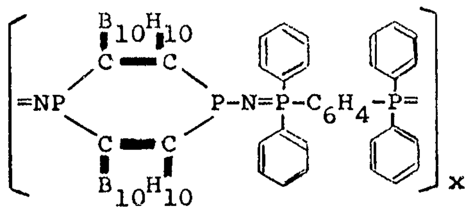
This is the first example of a phosphorus(III)azide reacting with a phosphine to form a phosphineimino compound.

Upon extension of this reaction to the use of a diphosphine following the PNP-polymer pattern, equimolar amounts of $(B_{10}H_{10}C_2P\cdot N_3)_2$ and p-$[(C_6H_5)_2P]_2C_6H_4$ reacted with evolution of nitrogen to form compound II.

FIGURE 1.

INFRARED SPECTRA OF CARBORANE, BIS(CHLOROPHENYLPHOSPHINO)-CARBORANE, AND DIMERIC CHLOROPHOSPHA (III) CARBORANE (Kel-F and Nujol Mulls)





II

This reaction was repeated several times and in each instance the elemental analysis was precise for the structure shown, and molecular weight determination gave x a value 3 ($\pm$ 5%). A rigorous structural determination of this apparently trimeric substance is in progress.

EXPERIMENTAL[8]

Starting Materials.- $B_{10}H_{10}C_2H_2$ was prepared as described in ref. 6 in n-propyl ether (Eastman Kodak) from resublimed $B_{10}H_{14}$, diethylsulfide (Matheson, Coleman & Bell) and acetylene which was purified by standard procedures.[9] Diphenylchlorophosphine was obtained from Victor Chemical Company.

Molecular Weights.- These were obtained in benzene either using a Mechrolab Osmometer, Model 301, or by cryoscopic determination.

Dilithiocarborane[6].- A 100 ml. three-neck flask fitted with a magnetic stirrer, a nitrogen inlet and outlet, and an addition funnel, is purged with nitrogen and then charged with a solution of butyllithium (0.066 mole) in ethyl ether (approx. 60 ml.). With ice-cooling a solution of carborane (4.33 g., 0.3 mole) in ethyl ether (20 ml.) is added over a period of 10 minutes, and the resulting white slurry is stirred under nitrogen and ice-cooling for another 30 minutes. Ether and excess butyllithium are then filtered from the precipitate through a coarse sintered-glass filter stick. The remaining $B_{10}H_{10}C_2Li_2$ is washed with ethyl ether (50 ml.) in two portions, dried under vacuum and then stored in an inert atmosphere.

Bis(diphenylphosphino)carborane.- A solution of diphenylchlorophosphine (14.6 g., 0.066 mole) in ether (15 ml.) was added, with ice-cooling, within 25 minutes to a dilithiocarborane slurry in ether prepared from 4.33 g. (0.3 mole) of carborane as described

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(8) Melting points are uncorrected; elemental analyses were by R. Culmo, A. C. Mayer, and J. D. Giunta of the Olin Mathieson Microchemical Section; mass spectral analyses were obtained and interpreted by Herman Hoberecht.

(9) We thank Mr. R. W. Blundon for preparing a continuing supply of carborane for this research.

in the preceding example. The slightly yellow mixture was stirred for 20 minutes at 20°, then refluxed for 30 minutes and, after cooling to 10°, hydrolyzed with 20 ml. of water. The insoluble reaction product was recovered by filtration, dried in vacuo and recrystallized from petroleum ether (b.p. 60-110°) to give 9.0 g. (58.5%) of $B_{10}H_{10}C_2[P(C_6H_5)_2]_2$, m.p. 219°.

Anal. Calcd. for $C_{26}H_{30}B_{10}P_2$ (512.7): C, 60.91; H, 5.90; B, 21.11; P, 12.09. Found: C, 61.15; H, 6.23; B, 20.63; P, 11.68.

Bis(chlorophenylphosphino)carborane.- A dilithiocarborane (0.173 mole) slurry in ether prepared from 25 g. of carborane was slowly added to a cooled solution of phenyldichlorophosphine (77.5 g., 0.43 mole) in ether (600 ml.). The mixture was stirred at room temperature for 90 minutes, then refluxed for 1 hour. Upon evaporation of the ether a yellow solid remained which was extracted with hot petroleum ether (800 ml., b.p. 60-110°). After concentration and cooling, 43.7 g. (58%) of $B_{10}H_{10}C_2(C_6H_5P{\cdot}Cl)_2$, m.p. 172-174°, precipitated from the solution.

Anal. Calcd. for $C_{14}H_{20}B_{10}Cl_2P_2$ (429.4): C, 39.16; H, 4.70; B, 25.20; Cl, 16.51; P, 14.43. Found: C, 38.87; H, 4.67; B, 24.95; Cl, 16.81; P, 14.18; mol. weight: 420.1.

C,C'-Bis phenylphospha(III) -azanyl(1,3)-carborane.- Gaseous ammonia was bubbled into a solution of $B_{10}H_{10}C_2(C_6H_5P{\cdot}Cl)_2$ (4.3 g., 0.01 mole) in anhydrous benzene (100 ml.) for 30 minutes. The temperature was maintained at 20° by external cooling. The reaction mixture was then evaporated to dryness, the solid residue was refluxed with 150 ml. of hot petroleum ether (b.p. 60-110°). After filtration, concentration, and cooling, the imino-compound precipitated and was recrystallized from petroleum ether. Yield of $B_{10}H_{10}C_2\left[(PC_6H_5)_2NH\right]$: 2.6 g. (70%); m.p. 222-24°.

Anal. Calcd. for $C_{14}H_{21}B_{10}NP_2$ (373.5): C, 45.02; H, 5.67; B, 28.97; N, 3.75; P, 16.59. Found: C, 45.00; H, 5.82; B, 28.46; N, 3.72; P, 16.65.

Bis(azidophenylphosphino)carborane.- A mixture of $B_{10}H_{10}C_2(C_6H_5P{\cdot}Cl)_2$ (10.7 g., 0.025 mole), sodium azide (6.5 g., 0.1 mole), and ethanol (150 ml.), was stirred with ice-cooling for 1 hour. The solid reaction products were recovered by filtration and thoroughly washed with water. The remaining bis-azide was dried in vacuo over diphosphorus pentoxide; m.p. 126-28° (dec.); yield: 9.1 g. (82%).

Anal. Calcd. for $C_{14}H_{20}B_{10}N_6P_2$ (442.5): C, 38.00; H, 4.56; B, 24.45; N, 18.99; P, 14.00. Found: C, 38.29; H, 4.60; B, 24.55; N, 17.80; P, 13.85.

Pyrolysis: A solution of 6 g. of $B_{10}H_{10}C_2(C_6H_5P{\cdot}N_3)_2$ in 125 ml. dry benzene was slowly warmed. At a bath temperature of approximately 50° gas evolution started. After keeping the mixture for 90 minutes at 55°, it was cooled to 20° and the precipitate formed (2.5 g.) was recovered by filtration. It did not show the characteristic azide absorption at 4.8 μ ; m.p. 170-180°.

Anal. Calcd. for $(C_{14}H_{20}B_{10}N_2P_2)_x$ (386.4): C, 43.51; H, 5.22; B, 28.00; N, 7.23; P, 16.03. Found: C, 41.93; H, 4.82; B, 26.44; N, 6.22; P, 14.89.

Dimeric Chlorophospha(III)carborane.- A dilithiocarborane (0.139 mole) slurry in ether (500 ml.) prepared from 20 g. of carborane was added in small portions, with ice-cooling, to a stirred solution of phosphorus trichloride (41.1 g., 0.3 mole) in ether (1000 ml.). The mixture immediately became purple and a colorless precipitate began to form. After stirring for 20 hours at 20°, the mixture had turned blue and was evaporated to dryness. The green solid residue was extracted with 300 ml. of

<u>Dimeric Triphenylphosphineiminophospha(III)carborane</u>.- To a mixture of $(B_{10}H_{10}C_2P{\cdot}N_3)_2$ (2.15 g., 0.005 mole) and triphenylphosphine (2.62 g., 0.01 mole) was added, in one portion, cold benzene (50 ml.). Solution was effected accompanied by vigorous foaming. After 25 minutes, gas evolution subsided and the solution was gradually heated to reflux temperature over a 30 minute-period. The hot reaction mixture was filtered and cooled to 10°. The colorless reaction product precipitated and was washed with petroleum ether. Yield of $[(C_6H_5)_3P{=}NPC_2B_{10}H_{10}]_2$: 2.4 g. (53.5%), m.p. 311-12°.

Anal. Calcd. for $C_{40}H_{50}B_{20}N_2P_4$ (899.2): C, 53.43; H, 5.61; B, 24.07; N, 3.12; P, 13.78. Found: C, 53.89; H, 5.92; B, 24.08; N, 3.54; P, 13.73.

<u>Reaction of Dimeric Azidophospha(III)carborane with 1,4-Bis-(diphenylphosphino)-benzene</u>.- Addition of cold benzene (75 ml.) to a mixture of $(B_{10}H_{10}C_2P{\cdot}N_3)_2$ (3.45 g., 0.008 mole) and p-$[(C_6H_5)_2P]_2C_6H_4$ (3.55 g., 0.008 mole) produced, with evolution of nitrogen, a clear, purple solution. Stirring at 20° for 1 hour followed by refluxing for 2 hours changed the color to pale-blue. After filtration from a small amount of solid, the filtrate was poured into 500 ml. of petroleum ether. A solid separated, was recovered by filtration and purified by washing with hot petroleum ether. Yield of product: 4.6 g. (70%); m.p. 235-50°.

Anal. Calcd. for $C_{34}H_{44}B_{20}N_2P_4$ (821.0): C, 49.74; H, 5.40; B, 26.36; N, 3.41; P, 15.09. Found: C, 50.20; H, 5.65; B, 26.06; N, 3.05; P, 14.59; molecular weight: 2367(experiment 1), 2585 (experiment 2), 2498(experiment 3).

Dimeric Phenylphospha(III)carborane.- A solution of $B_{10}H_{10}C_2(C_6H_5P\cdot Cl)_2$ (6.9 g., 0.016 mole) in ether (200 ml.) was added, with stirring, to an ice-cooled dilithiocarborane (0.016) slurry in ether (100 ml.) prepared from 2.32 g. of carborane. After stirring the mixture for 12 hours at room temperature, it was filtered to give 3.6 g. of insoluble residue. Extraction with hot benzene afforded 2.0 g. of product which was recrystallized from benzene to give 1.6 g. (20%) of $(B_{10}H_{10}C_2P\cdot C_6H_5)_2$, m.p. 356-358°.

Anal. Calcd. for $C_{16}H_{30}B_{20}P_2$ (500.8): C, 38.38; H, 6.04; B, 43.21; P, 12.37. Found: C, 38.28; H, 5.79; B, 42.36; P, 11.93.

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