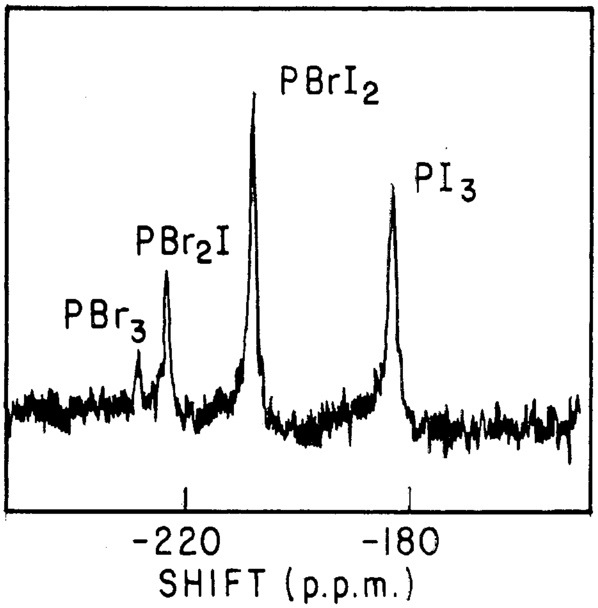

Figure 1.—P^{31} n.m.r. spectrum of a P_2I_4–bromine reaction mixture (pure liquid).

The bromo iodides can also be detected in PBr_3–PI_3 mixtures. A 2:1 mole ratio mixture of PI_3 and PBr_3 undergoes an endothermal reorganization reaction within a few minutes to yield a product with P^{31} n.m.r. and mass spectra that are very similar to those of the P_2I_4–bromine reaction product. The rate of reorganization seems to increase along the series $F < Cl < Br < I$. Thus, the PBr_3–PCl_3 system[10] requires 1.5 hr. for equilibration while the PBr_2F–PCl_3 system[10] requires 60 hr. Further evidence for the relative lability of the bromo–iodo system is our study of the mass spectra of the P_2I_4–bromine reaction product at various temperatures. Upon raising the temperature of the heated inlet system in steps of 30 to 40° one finds that the PI_3 and $PBrI_2$ peaks increase in intensity at the expense of the PBr_2I and PBr_3 peaks. The lability of the system, plus its lack of volatility, has precluded a separation of the reaction mixture into its components.

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CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF FLORIDA, GAINESVILLE, FLORIDA

Further Studies in the Chloramination of Some Aminophosphines

BY DONALD F. CLEMENS AND HARRY H. SISLER

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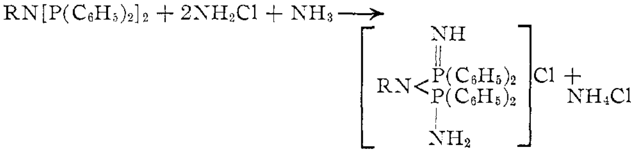
It has recently been shown that chloramine reacts with aminophosphines to form aminophosphonium salts.[1] All the previously reported chloraminations were, however, concerned with phenylphosphino derivatives and none of them contained alkyl–phosphorus bonds. Furthermore, with the exception of 1,1-bis-(diphenylphosphino)-2,2-dimethylhydrazine,[2] none of the previously chloraminated nitrogen–phosphorus derivatives contained more than one phosphorus atom attached to a nitrogen atom.

The present work was undertaken to determine if, as in the case of the previously studied[1] aminophenylphosphines, chloramination of aminoalkylphosphines and of bis(diphenylphosphino)amines occurs on the phosphorus atom(s) rather than the nitrogen atom. The results have established that this is indeed the case and that the chloramination of the bis(diphenylphosphino)amines occurs in accordance with the equation



$$RN[P(C_6H_5)_2]_2 + 2NH_2Cl + NH_3 \longrightarrow \left[RN{<}\begin{matrix} \overset{\displaystyle NH}{\overset{\|}{P}}(C_6H_5)_2 \\ \underset{\underset{\displaystyle NH_2}{|}}{P}(C_6H_5)_2 \end{matrix} \right] Cl + NH_4Cl$$

This work has further shown that if the R group on the nitrogen atom in the bisphosphinoamines is a hydrogen atom the compound obtained on chloramination is $[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl$, which has been synthesized by the ammonolysis of diphenyltrichlorophosphorane[3] and by the ammonolysis and chloramination of diphenylchlorophosphine.[4]

Experimental

Materials.—Solvents used were dried over calcium hydride. The anhydrous amines were obtained from The Matheson Company, Inc., and were used as received. Diphenylchlorophosphine was provided by the Victor Chemical Works. Tributylphosphine was obtained from Food Machinery and Chemical Corp. Dibutylchlorophosphine was prepared by the pyrolysis of tributyldichlorophosphorane.[5]

Analyses.—Elemental analyses were done by Galbraith Microanalytical Laboratories. Melting points were obtained in wax-sealed capillary tubes in a Thomas-Hoover capillary melting point apparatus and are uncorrected.

Infrared Spectra.—The infrared spectra were recorded on a Perkin-Elmer Model 137 spectrometer using sodium chloride optics. The spectra of the solids were obtained from Nujol mulls. A summary of the data is found in Table I.

Nuclear Magnetic Resonance Spectra.—Nuclear magnetic resonance spectra were obtained on a Varian high-resolution spectrometer, Model V-4300-2, equipped with field homogeneity control, magnet insulation, and field stabilizer. A summary of the data is found in Table II.

Preparation of Aminophosphines.—Some of the aminophosphines used in this work have been reported previously.[6,7] Using similar methods two new aminophosphines have been prepared. These are ethylaminodibutylphosphine, $C_2H_5NHP(C_4H_9)_2$, and bis(diphenylphosphino)amine, $HN[P(C_6H_5)_2]_2$. Analytical and melting point data are listed in Table III.

Procedure for Chloramination Reactions.—The procedure out-

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[1] W. A. Hart and H. H. Sisler, *Inorg. Chem.*, **3**, 617 (1964).

[2] R. P. Nielsen, J. F. Vincent, and H. H. Sisler, *ibid.*, **2**, 760 (1963).

[3] I. I. Bezman and J. H. Smalley, *Chem. Ind.* (London), 839 (1960).

[4] H. H. Sisler, H. S. Ahuja, and N. L. Smith, *Inorg. Chem.*, **1**, 84 (1962).

[5] S. E. Frazier, R. P. Nielsen, and H. H. Sisler, *ibid.*, **3**, 292 (1964).

[6] A. B. Burg and P. J. Slota, *J. Am. Chem. Soc.*, **80**, 1107 (1958).

[7] G. Ewart, A. P. Lane, J. McKechnie, and D. S. Payne, *J. Chem. Soc.* 1543 (1964).

TABLE I

INFRARED ABSORPTION (CM.$^{-1}$)[a]

$(CH_3)_2NP(CH_3)_2$, neat

3030 (vs), 2960 (vs), 2900 (sh, vs), 2840 (vs), 1470 (sh, s), 1450 (s), 1430 (s), 1420 (sh, m), 1290 (s), 1270 (s), 1190 (vs), 1140 (m), 1100 (vw), 1060 (s), 971 (vs), 936 (vs), 889 (vs), 845 (s), 811 (m), 692 (s)

$[(H_2N)\{(CH_3)_2N\}P(CH_3)_2]Cl$, Nujol

3250 (sh, vs), 3170 (sh, vs), 2940 (vs), 2650 (m), 2170 (vw), 1630 (vw), 1570 (m), 1460 (vs), 1420 (s), 1380 (m), 1310 (vs), 1270 (s), 1180 (s), 1080 (s), 987 (b, vs), 951 (s), 899 (m), 856 (s), 793 (m), 752 (s)

$C_2H_5NHP(C_4H_9)_2$, neat

3450 (sh, w), 3390 (w), 2940 (vs), 2900 (sh, s), 1460 (s), 1400 (m), 1370 (s), 1340 (w), 1290 (w), 1260 (w), 1180 (b, w), 1120 (vs), 1090 (sh, m), 1060 (s), 1030 (b, sh, w), 1000 (w), 966 (w), 935 (m), 897 (m), 810 (b, sh, w), 781 (sh, m), 725 (b, m)

$[(H_2N)\{(C_2H_5)HN\}P(C_4H_9)_2]Cl$, Nujol

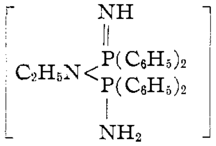
3190 (s), 3120 (sh, s), 2940 (vs), 2760 (sh, w), 1560 (w), 1460 (vs), 1380 (s), 1310 (vw), 1270 (vw), 1230 (w), 1190 (vw), 1130 (s), 1100 (m), 1080 (sh, m), 1060 (w), 1020 (b, sh, w), 1000 (sh, m), 988 (s), 957 (m), 901 (b, w), 846 (b, w), 802 (w), 781 (b, sh, w), 720 (b, m)

$C_2H_5N[P(C_6H_5)_2]_2$, Nujol

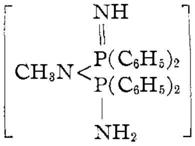
3110 (sh, m), 2960 (vs), 2900 (sh, vs), 1970 (vw), 1890 (vw), 1820 (vw), 1590 (w), 1460 (s), 1430 (s), 1370 (s), 1300 (w), 1180 (vw), 1150 (m), 1090 (s), 1060 (s), 1030 (m), 998 (w), 966 (w), 924 (m), 879 (s), 854 (w), 766 (m), 749 (s), 742 (s), 738 (sh, s), 695 (b, s)

$CH_3N[P(C_6H_5)_2]_2$, Nujol

3110 (sh, vs), 2990 (vs), 1960 (vw), 1890 (vw), 1820 (vw), 1650 (vw), 1580 (m), 1470 (vs), 1430 (vs), 1420 (sh, m), 1380 (s), 1310 (m), 1270 (m), 1170 (m), 1160 (sh, m), 1090 (vs), 1070 (vs), 1020 (m), 998 (m), 966 (vw), 917 (d, w), 851 (vs), 743 (vs), 695 (vs)


$\left[C_2H_5N{<}\begin{array}{l}P(C_6H_5)_2{=}NH\\P(C_6H_5)_2{-}NH_2\end{array}\right]Cl$, Nujol

3210 (sh, s), 2940 (vs), 1980 (vw), 1890 (vw), 1820 (vw), 1590 (m), 1560 (sh, w), 1470 (sh, m), 1460 (vs), 1430 (vs), 1370 (s), 1280 (b, sh, s), 1250 (sh, s), 1220 (vs), 1180 (sh, s), 1160 (sh, m), 1120 (vs), 1090 (m), 1070 (m), 1030 (w), 998 (m), 969 (s), 937 (m), 861 (w), 850 (w), 798 (w), 765 (m), 746 (s), 727 (s), 719 (vs), 690 (b, vs)


$\left[CH_3N{<}\begin{array}{l}P(C_6H_5)_2{=}NH\\P(C_6H_5)_2{-}NH_2\end{array}\right]Cl$

3180 (sh, s), 3100 (sh, vs), 2970 (vs), 1910 (vw), 1820 (vw), 1680 (vw), 1580 (d, m), 1480 (sh, m), 1470 (s), 1450 (vs), 1420 (sh, m), 1380 (m), 1270 (b, vs), 1180 (d, m), 1160 (sh, m), 1120 (vs), 1090 (s), 1030 (m), 1000 (s), 947 (s), 907 (w), 862 (m), 849 (sh, w), 781 (w), 756 (s), 724 (vs), 691 (b, vs)

$HN[P(C_6H_5)_2]_2$, Nujol

3250 (m), 3080 (sh, m), 2960 (vs), 2880 (sh, s), 1960 (vw), 1890 (vw), 1810 (vw), 1480 (sh, s), 1460 (s), 1430 (s), 1380 (s), 1300 (vw), 1270 (vw), 1250 (m), 1160 (b, vw), 1100 (s), 1070 (sh, w), 1020 (m), 997 (w), 916 (sh, m), 905 (sh, s), 897 (vs), 794 (m), 749 (s), 736 (vs), 692 (b, vs)

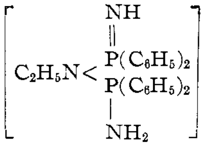
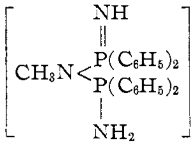
$[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl\cdot HCCl_3$, Nujol

3250 (sh, m), 3130 (sh, s), 2990 (vs), 1590 (w), 1570 (sh, w), 1470 (vs), 1440 (sh, s), 1380 (s), 1280 (d, vs), 1250 (sh, s), 1180 (w), 1130 (s), 1120 (sh, s), 1030 (w), 998 (w), 952 (b, m), 922 (sh, w), 747 (b, vs), 724 (vs), 690 (vs)

[a] s, strong; m, medium; w, weak; sh, shoulder; d, doublet; b, broad.

TABLE II

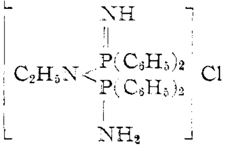
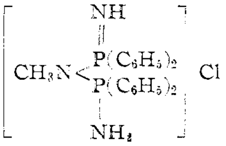
NUCLEAR MAGNETIC RESONANCE DATA

	^1H shifts in approximate τ values[a]								^{31}P shifts, p.p.m. from H_3PO_4
Compound	C_6H_5[b]	NH	NH_2	NCH_2	NCH_3	PCH_3	CH_2	CH_3	
$(CH_3)_2NP(CH_3)_2$					7.00 (d 10.1)	8.34 (d 5.7)			−39
$[(CH_3)_2NP(CH_3)_2(NH_2)]Cl$			3.70		7.08 (d 11.1)	7.80 (d 14.5)			−54
$C_2H_5NHP(C_4H_9)_2$				7.34					−37
$[C_2H_5NHP(C_4H_9)_2(NH_2)]Cl$			4.19 4.46	6.85					−54
$C_2H_5N[P(C_6H_5)_2]_2$	2.97			6.79 (q 7.5)				9.30 (t 7.5)	−61
 $\left[C_2H_5N{<}\begin{array}{l}P(C_6H_5)_2{=}NH\\P(C_6H_5)_2{-}NH_2\end{array}\right]Cl$	2.07 2.53	3.31	4.48	7.29 (q)				8.85 (t)	−21 150 c.p.s. broad
$CH_3N[P(C_6H_5)_2]_2$	2.78				7.70 (t)				−72
 $\left[CH_3N{<}\begin{array}{l}P(C_6H_5)_2{=}NH\\P(C_6H_5)_2{-}NH_2\end{array}\right]Cl$	2.32 2.79	3.46 (t 4.6)	4.49		7.60 (d 5.0) (d 14.4)				−24 150 c.p.s. broad
$[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl$	2.45		4.63						−20

[a] The chemical shift values were obtained by sweeping through the field slowly and interchanging a reference sample, acetaldehyde, and the sample being studied. Coupling constants in c.p.s. are in parentheses; d, doublet; t, triplet; q, quartet. [b] The chemical shift values of the phenyl peaks were obtained by measuring to the highest part of the peak.

TABLE III

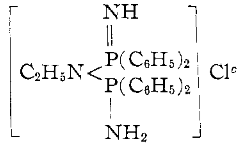
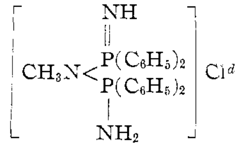
ANALYTICAL AND MELTING POINT DATA

Compound	B.p., °C. (mm.) or m.p., °C.	% C Calcd.	% C Found	% H Calcd.	% H Found	% N Calcd.	% N Found	% N Calcd.	% N Found	% Cl Calcd.	% Cl Found
$C_2H_5NHP(C_4H_9)_2$	75 (2.0)	63.45	63.27	12.78	12.86	7.40	7.24	16.37	16.20		
$HN[P(C_6H_5)_2]_2$	145–146	74.79	74.88	5.49	5.48	3.64	3.62	16.08	16.03		
$[(H_2N)\{(CH_3)_2N\}(CH_3)_2P]Cl$	190–193	30.67	30.66	9.01	9.20	17.89	18.06	19.78	19.89	22.64	22.27
$[(H_2N)\{(C_2H_5)HN\}(C_4H_9)_2P]Cl$	103–104	49.88	49.71	10.89	11.02	11.64	11.49	12.87	12.67	14.73	14.41
 $\left[\begin{matrix} NH \\ \Vert \\ C_2H_5N<^{P(C_6H_5)_2}_{P(C_6H_5)_2} \\ \vert \\ NH_2 \end{matrix}\right]Cl$	176 dec.	65.06	65.03	5.88	6.02	8.76	8.92	12.91	12.93	7.39	7.60
 $\left[\begin{matrix} NH \\ \Vert \\ CH_3N<^{P(C_6H_5)_2}_{P(C_6H_5)_2} \\ \vert \\ NH_2 \end{matrix}\right]Cl$	166–168	64.44	64.34	5.63	5.57	9.02	8.85	13.30	13.25	7.61	7.72
$[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl\cdot HCCl_3$[a]		52.56	52.59	4.41	4.39	7.36	7.43	10.85	11.21		

[a] Decomposes to yield the material $[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl$ which melts at 245–246°.

TABLE IV

CHLORAMINATION REACTION CONDITIONS

Compound	Solvent	Chloramination time at room temp., min.	Standing time at room temp., hr.	Product	Yield
$C_2H_5NHP(C_4H_9)_2$ (1.10 g.)	C_6H_6 (50 ml.)	6	4	$[H_2N\{(C_2H_5)NH\}P(C_4H_9)_2]Cl$[a]	1.18 g., 84%
$(CH_3)_2NP(CH_3)_2$ (1.62 g.)	C_6H_6 (30 ml.)	11	12	$[H_2N\{(CH_3)_2N\}P(CH_3)_2]Cl$[b]	1.47 g., 61%
$C_2H_5N[P(C_6H_5)_2]_2$ (2.37 g.)	C_6H_6 (50 ml.)	13	3	 $\left[\begin{matrix} NH \\ \Vert \\ C_2H_5N<^{P(C_6H_5)_2}_{P(C_6H_5)_2} \\ \vert \\ NH_2 \end{matrix}\right]Cl$[c]	1.75 g., 64%
$CH_3N[P(C_6H_5)_2]_2$ (1.94 g.)	C_6H_6 (30 ml.)	10	36	 $\left[\begin{matrix} NH \\ \Vert \\ CH_3N<^{P(C_6H_5)_2}_{P(C_6H_5)_2} \\ \vert \\ NH_2 \end{matrix}\right]Cl$[d]	1.31 g., 58%
$HN[P(C_6H_5)_2]_2$ (2.20 g.)	C_6H_6 (50 ml.)	10	24	$[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl\cdot HCCl_3$[e]	1.74 g., 53%

[a] Recrystallized from benzene–hexane. [b] Recrystallized from chloroform–hexane. [c] Washed rapidly with small portions of water followed by ether. [d] Recrystallized from benzene. [e] Recrystallized from chloroform–benzene.

lined in ref. 1 was followed. The effluent gas from the chloramine generator yielded about 0.1 mole of chloramine per hour. The experimental details from the various chloraminations are outlined in Table IV.

Hydrolysis of Aminodimethylaminodimethylphosphonium Chloride.—Aminodimethylaminodimethylphosphonium chloride was hydrolyzed with aqueous sodium hydroxide in a calibrated vacuum system. The volatile products were separated by fractional condensation and identified as ammonia and dimethylamine.

The Isolation and Identification of $[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl\cdot HCCl_3$.—The reaction products from the chloramination of $HN[P(C_6H_5)_2]_2$ were extracted with hot chloroform to separate the phosphonium salt from the ammonium chloride which is also formed in the reaction. The chloroform adduct[1] was then precipitated from the extract by the addition of benzene. Warming this adduct, under vacuum, to 75° for 2 hr. resulted in the removal of the chloroform yielding $[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl$. Identification was carried out by comparing the infrared spectrum, the n.m.r. spectrum, and by obtaining a mixture melting point with a known sample.

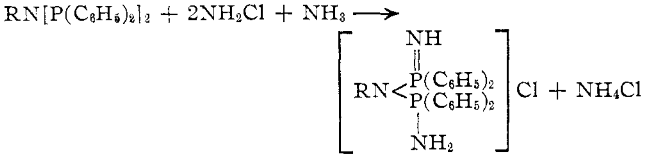
Discussion

The infrared, n.m.r., and hydrolytic data for the various chloramination products reported herein confirm the fact that the chloramination occurs on the phosphorus rather than the nitrogen atom. For example, hydrolysis of the chloramination product of dimethylaminodimethylphosphine yields only dimethylphosphinic acid, ammonia, and dimethylamine with no trace of a hydrazine derivative. This hydrolysis may be represented by the equation

$$[(H_2N)\{(CH_3)_2N\}P(CH_3)_2]Cl + H_2O + OH^- \longrightarrow (CH_3)_2P(O)(OH) + NH_3 + (CH_3)_2NH + Cl^-$$

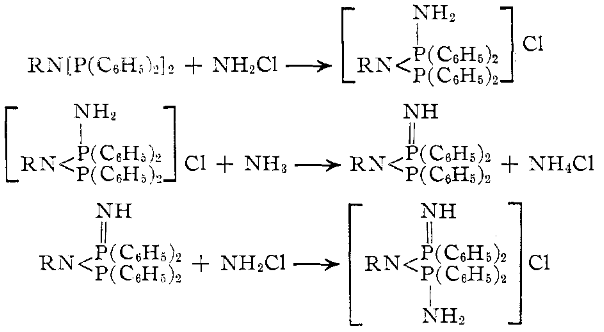
A comparison of ^{31}P n.m.r. chemical shift values from Table II of the aminophosphines having alkyl–phosphorus bonds and their chloramination products shows that the phosphorus peaks have been shifted downfield in the phosphonium salts relative to the starting material. This change in the chemical shift value is evidence that the environment of the phosphorus atom has changed considerably. If the chloramine had attacked the nitrogen atoms, the change in chemical shift values would probably have been small. Further, the infrared spectra of the chloramination products which contain P–phenyl bonds all exhibit the strong peak at about 1120 cm.$^{-1}$ which has been assigned to a tetracoordinate phosphorus atom having a P–phenyl bond.[1]

Analytical, n.m.r., and infrared data show clearly that the chloramination of compounds of the type $RN[P(C_6H_5)_2]_2$ results in the combination of the bisphosphinoamine with 2 moles of chloramine in accordance with the equation



$$RN[P(C_6H_5)_2]_2 + 2NH_2Cl + NH_3 \longrightarrow \left[RN{<}\begin{matrix} \overset{NH}{\overset{\|}{P(C_6H_5)_2}} \\ \underset{NH_2}{\underset{|}{P(C_6H_5)_2}} \end{matrix} \right] Cl + NH_4Cl$$

A reasonable rationalization of this result would involve the assumption that the initial monochloramination product undergoes dehydrohalogenation and thus remains in solution and available for the second chloramination step.



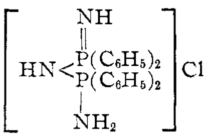
$$RN[P(C_6H_5)_2]_2 + NH_2Cl \longrightarrow \left[RN{<}\begin{matrix} \overset{NH_2}{\overset{|}{P(C_6H_5)_2}} \\ P(C_6H_5)_2 \end{matrix} \right] Cl$$

$$\left[RN{<}\begin{matrix} \overset{NH_2}{\overset{|}{P(C_6H_5)_2}} \\ P(C_6H_5)_2 \end{matrix} \right] Cl + NH_3 \longrightarrow RN{<}\begin{matrix} \overset{NH}{\overset{\|}{P(C_6H_5)_2}} \\ P(C_6H_5)_2 \end{matrix} + NH_4Cl$$

$$RN{<}\begin{matrix} \overset{NH}{\overset{\|}{P(C_6H_5)_2}} \\ P(C_6H_5)_2 \end{matrix} + NH_2Cl \longrightarrow \left[RN{<}\begin{matrix} \overset{NH}{\overset{\|}{P(C_6H_5)_2}} \\ \underset{NH_2}{\underset{|}{P(C_6H_5)_2}} \end{matrix} \right] Cl$$

This scheme is analogous to that previously postulated by our laboratory for the chloramination of 1,1-bisdiphenylphosphino-2,2-dimethylhydrazine, $[(C_6H_5)_2P]_2NN(CH_3)_2$.[2]

In the case of the compound $HN[P(C_6H_5)_2]_2$ the chloramination product obtainable according to this reaction scheme, *viz.*



$$\left[HN{<}\begin{matrix} \overset{NH}{\overset{\|}{P(C_6H_5)_2}} \\ \underset{NH_2}{\underset{|}{P(C_6H_5)_2}} \end{matrix} \right] Cl$$

is a tautomer of the previously prepared compound $[(C_6H_5)_2P(NH_2)NP(NH_2)(C_6H_5)_2]Cl$[4] and would be expected to rearrange to yield that compound. Analytical, infrared, and n.m.r. data show that this is indeed the case and provide further evidence of the preference of the chloramine molecule for attack on the phosphorus rather than the nitrogen atom.

Acknowledgments.—The authors are pleased to acknowledge the support of much of this work by a grant from the Petroleum Research Fund administered by the American Chemical Society. We also wish to express our appreciation to Professor Wallace S. Brey, Jr., for his assistance in obtaining and interpreting the nuclear magnetic resonance data included in this paper.

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INDIANA UNIVERSITY, BLOOMINGTON, INDIANA

Synthesis of a New Deuterium-Labeled Tetraborane, μ-Deuteriotetraborane-10[1]

BY ARLAN D. NORMAN AND RILEY SCHAEFFER

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Isotopically labeled boranes are convenient compounds for studies of interconversion and other reaction mechanisms of boron hydrides; however, only two reports of specifically labeled tetraboranes have appeared. Schaeffer and Tebbe reported a reaction in which labeling of either the boron atom or the hydrogen atoms at the 2 position[2] could be effected.[3] Koski and co-workers have also presented some evidence for 1,3-dideuteriotetraborane-10 obtained during exchange reactions between tetraborane-10 and deuterated diborane.[4,5] Recently, by substituting deuterium oxide for water in the pentaborane-11 water reaction described by Boone and Burg,[6] we have prepared the first monodeuterium labeled tetraborane, μ-deuteriotetraborane-10.

Experimental

Preparation of Starting Materials.—All operations and manipulations performed in these investigations involve standard high-vacuum techniques described elsewhere.[7,8]

Pentaborane-11 used in these preparations was obtained by the pyrolysis of diborane in a hot–cold reactor in which the hot and cold surfaces were maintained at 155 ± 5° and −80°, respectively.[9] Carrying out the pyrolysis at a pressure of approximately 1 atm. for a 6- or 7-hr. period resulted in conversion of 30 to 40% of the diborane to tetraborane-10 and pentaborane-11. Final purification of pentaborane-11 was carried out by repeated fractionation at −98 to −101° using a low-temperature fractional distillation column.[10] Pentaborane-11 was obtained in 70 to 75% yield based on the amount of diborane converted.

Deuterium oxide (isotopic purity stated at >99.8%) obtained from the Atomic Energy Commission was used without further purification.

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(1) Studies of Boranes. XVIII. For paper XVII of this series see P. C. Keller, D. MacLean, and R. Schaeffer, *Chem. Commun.* (London), in press. Correspondence regarding this paper should be addressed to R. Schaeffer.

(2) The numbering system is that recommended for boron compounds by the Preliminary Report of the Advisory Committee on the Nomenclature of Organic Boron Compounds, available through *Chemical Abstracts.*

(3) R. Schaeffer and F. N. Tebbe, *J. Am. Chem. Soc.*, **84**, 3974 (1962); F. N. Tebbe, Ph.D. Thesis, Indiana University, 1963.

(4) J. E. Todd and W. S. Koski, *J. Am. Chem. Soc.*, **81**, 2319 (1959).

(5) T. P. Fehlner and W. S. Koski, *ibid.*, **85**, 1905 (1963).

(6) J. L. Boone and A. B. Burg, *ibid.*, **80**, 1519 (1958).

(7) A. Stock, "Hydrides of Boron and Silicon," Cornell University Press, Ithaca, N. Y., 1933.

(8) R. T. Sanderson, "High Vacuum Manipulations of Volatile Compounds," John Wiley and Sons Inc., New York, N. Y., 1948.

(9) M. J. Klein, B. C. Harrison, and I. J. Solomon, *J. Am. Chem. Soc.*, **80**, 4149 (1958).

(10) J. Dobson and R. Schaeffer, "Studies of Boron–Nitrogen Compounds, X" to be published elsewhere.