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(21) It is also clear that the inability of methylviologen and related molecules to catalyze reductions of Co(III) by $Ru(NH_3)_6^{2+}$ is simply a reflection of the weak reduction potential (+0.214 V)[6] of the latter.[22] The equilibrium constant for reduction of methylviologen by this reductant is only 9×10^{-13}. A maximum value of 10^8 M^{-1} s^{-1} for k_1 (the diffusion-controlled limit for bimolecular reactions between +1 and +3 ions[23]) corresponds to an upper limit of 9×10^{-5} M^{-1} s^{-1} for k_1. In contrast, the specific rate for the uncatalyzed reduction of $Co(en)_3^{3+}$ by $Ru(NH_3)_6^{2+}$ is approximately 6×10^{-3} M^{-1} s^{-1},[13] and other Co(III) derivatives are reduced still more rapidly.

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Synthesis and Some Oxidation Reactions of [Bis(trimethylsilyl)amino]dimethylphosphine

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Received February 14, 1977 AIC70118D

The synthesis, characterization, and some reactions of [bis(trimethylsilyl)amino]dimethylphosphine (**1**) are reported. Although compound **1** was routinely prepared via the reaction of $LiN(SiMe_3)_2$ and Me_2PCl, it was also isolated when Me_3SiPMe_2 was treated with Me_3SiN_3. The reactions of the aminophosphine **1** with elemental sulfur, MeI, and Me_3SiN_3 afforded the expected four-coordinate oxidation products $(Me_3Si)_2NP(S)Me_2$, **2**, $[(Me_3Si)_2NPMe_3]^+I^-$, **3**, and $(Me_3Si)_2NP(NSiMe_3)Me_2$, **4**. When **1** was allowed to react with molecular oxygen, however, only the rearranged product, $Me_3SiOP(NSiMe_3)Me_2$, **5**, was obtained. Carbon-13 as well as ^1H NMR data are reported for compounds **1**–**5**.

Introduction

The chemistry and stereochemistry of compounds containing the bis(trimethylsilyl)amino substituent are often markedly different from that of the analogous dialkylamino derivatives. The steric bulk of the $(Me_3Si)_2N$ group and its planar geometry at nitrogen[1] are responsible, in part, for such observations as the stability of unusually low coordination numbers and valence states,[2] high element–nitrogen rotational barriers,[3] and unexpected ground-state structures[4] in many bis(trimethylsilyl)amino-substituted compounds. Moreover, the chemistry of such compounds is sometimes reflective of the lability of the *N*-trimethylsilyl substituent toward intramolecular rearrangements.[5]

As part of a continuing investigation of silicon–nitrogen–phosphorus systems, this paper reports the synthesis and some derivative chemistry of [bis(trimethylsilyl)amino]dimethylphosphine, $(Me_3Si)_2NPMe_2$ (**1**). The primary aim of this study was to assess the effect of a bis(trimethylsilyl)amino substituent on the course of some common oxidative addition reactions of trivalent phosphorus, with particular interest directed toward possible silyl group migrations in the resulting products.

Results and Discussion

When chlorodimethylphosphine was allowed to react with the *N*-lithium derivative of bis(trimethylsilyl)amine in ether solution (eq 1), [bis(trimethylsilyl)amino]dimethylphosphine

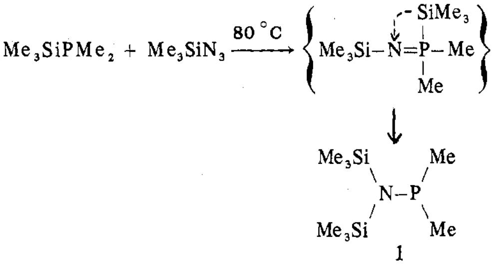
$$(Me_3Si)_2NLi + ClPMe_2 \rightarrow \underset{\mathbf{1}}{(Me_3Si)_2NPMe_2} + LiCl \qquad (1)$$

(**1**) was obtained.

A similar procedure has been used previously[6] for the synthesis of compound **1**; however, only low yields of an impure, poorly characterized product were reported. In the present study, compound **1**, a colorless, air-sensitive (see below) liquid, was routinely obtained in 60–70% yields and was readily purified by fractional distillation. It was fully characterized by NMR (^1H and ^{13}C), infrared, and mass spectroscopy as well as elemental analysis.

Unlike many similar reactions,[7,8] the preparation of compound **1** was found to be extremely sensitive to changes in the reaction conditions. High yields of the product were obtained only when a dilute ether solution of Me_2PCl was added slowly to a vigorously stirred solution of $LiN(SiMe_3)_2$ at 0 °C. Procedures similar to those used for the synthesis of other silylaminophosphines[8] such as the addition of Me_2PCl to $LiN(SiMe_3)_2$ at −78 °C, followed by slow warming to room temperature, gave substantially lower yields (<20%).

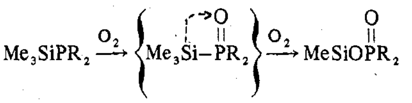
Somewhat surprisingly, the silylaminophosphine **1** was also isolated in a high yield from the reaction (eq 2) of (trimethylsilyl)dimethylphosphine with trimethylsilyl azide.



(2)

This process can be viewed as a combination of two known reactions: the Staudinger phosphinimine synthesis[9] (eq 3) followed by migration of the Me_3Si group as occurs during the oxidation of silylphosphines[10] (eq 4).

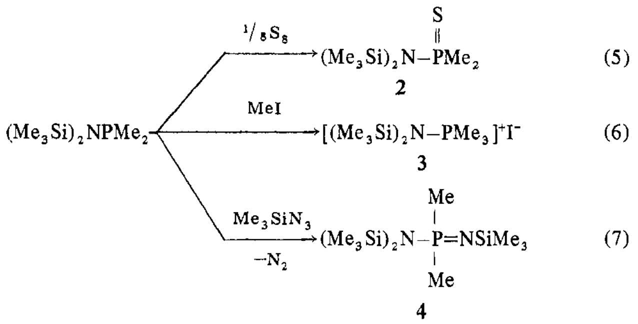
$$R_3P + R'N_3 \rightarrow R_3P{=}NR' + N_2 \qquad (3)$$



$$Me_3SiPR_2 \xrightarrow{O_2} \left\{ Me_3Si{-}\overset{\overset{O}{\|}}{P}R_2 \right\} \xrightarrow{O_2} MeSiO\overset{\overset{O}{\|}}{P}R_2 \qquad (4)$$

The success of this novel synthetic route (eq 2) to silylaminophosphines prompted us to attempt a similar reaction with Me_3SiPH_2 as a possible preparation of $(Me_3Si)_2NPH_2$, a compound not accessible by other methods. When Me_3SiPH_2 and Me_3SiN_3 were heated together in a sealed tube, no reaction occurred below 180 °C, and further heating gave only N_2, Me_3SiH, and unidentified yellow solids. These observations are consistent with the reduced basicity of the primary phosphine Me_3SiPH_2 relative to Me_3SiPMe_2 and the expected thermal instability of the silylaminophosphine $(Me_3Si)_2NPH_2$.

With the exception of its reaction with molecular oxygen, the derivative chemistry of the silylaminophosphine **1** was found to be similar to that of other trivalent phosphorus compounds. Thus, the reactions (eq 5–7) with sulfur, iodomethane, and trimethylsilyl azide proceeded rapidly under mild



$$(Me_3Si)_2NPMe_2 \xrightarrow{1/8\,S_8} (Me_3Si)_2N-P(=S)Me_2 \quad \mathbf{2} \qquad (5)$$

$$(Me_3Si)_2NPMe_2 \xrightarrow{MeI} [(Me_3Si)_2N-PMe_3]^+I^- \quad \mathbf{3} \qquad (6)$$

$$(Me_3Si)_2NPMe_2 \xrightarrow[-N_2]{Me_3SiN_3} (Me_3Si)_2N-P(Me)_2=NSiMe_3 \quad \mathbf{4} \qquad (7)$$

conditions to give the four-coordinate silicon–nitrogen–phosphorus compounds **2**–**4**.

The phosphine sulfide **2** is a low-melting solid which was identified by ^1H and ^{13}C NMR spectroscopy. The extreme moisture sensitivity of the compound, however, made elemental analysis impractical. Even brief exposure of solutions of **2** to the atmosphere resulted in rapid hydrolysis according to eq 8.

$$2(Me_3Si)_2NP(S)Me_2 + H_2O \rightarrow 2Me_3SiN(H)P(S)Me_2 + (Me_3Si)_2O \qquad (8)$$

The hydrolysis product, $Me_3SiN(H)P(S)Me_2$, has been prepared via a different method[11] and its reported properties are identical with those observed in the present study. The increased susceptibility to hydrolysis at the Si–N bond in the four-coordinate compound **2** relative to the parent phosphine **1** is consistent with results reported by Keat[7] for similar silicon–nitrogen–phosphorus systems.

The silylaminophosphonium salt **3**, a moisture-sensitive solid, prepared by simple means and in high yield (eq 6), has been mentioned previously in the literature.[9] It was apparently obtained from the reaction of Me_3SiI and $Me_3SiNPMe_3$ although no experimental details have been published. Nevertheless, the melting points of the products of both reactions are in good agreement.

Scherer and Klusmann[6] have reported that the silylaminophosphinimine **4** was produced in 10% yield when an impure material, thought to be the silylaminophosphine **1**, was heated with Me_3SiN_3. Repetition of this reaction (eq 7) in our laboratory using purified starting materials afforded the same product but in substantially higher yield (75%). In the earlier work,[6] ^1H NMR spectroscopy indicated that compound **4** is undergoing a rapid 1,3-silyl exchange, thus accounting for the observation of a single, broad resonance for all of the Me_3Si protons at room temperature. In our study, this result was verified by the ^{13}C NMR spectrum of **4** which contained only a broad singlet in the Me_3Si region.

It is perhaps interesting to note that some silylated phosphinimines, closely related to **4**, such as the difluoro compound $(Me_3Si)_2NPF_2(NSiMe_3)$,[12] are not fluxional and show the expected nonequivalence in the Me_3Si regions of the ^1H and ^{13}C NMR spectra. Yoder[5a,13] had studied 1,3-silyl migrations from nitrogen to oxygen in amides $RC(O)N(R')SiMe_3$ and concluded that the tendency toward migration is diminished by electron-withdrawing substituents at either carbon or nitrogen. While it seems likely that a similar effect may be operative in the phosphorus systems, no definitive, systematic studies to support this contention have been reported.

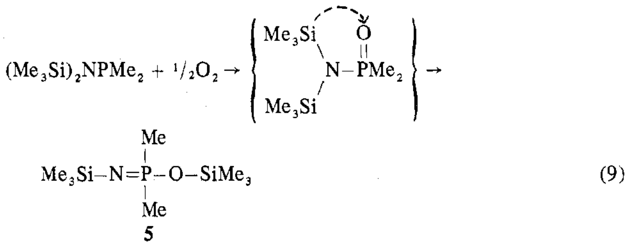
An example of a silyl migration from nitrogen to oxygen has also been observed in the present study. When dry oxygen was bubbled through a solution of the silylaminophosphine **1** in CH_2Cl_2, the product isolated (eq 9) in 49% yield was *P*,*P*-dimethyl-*P*-trimethylsiloxy-*N*-trimethylsilylphosphinimine (**5**) rather than the isomeric phosphine oxide which, presumably, was formed initially.

Compound **5** is a colorless, thermally stable (to at least 130 °C) liquid which was fully characterized by elemental analysis, IR, ^1H and ^{13}C NMR, and mass spectroscopy. The conclusion that the compound has the imine structure **5** is supported by at least three lines of evidence. First, the ^1H NMR spectrum contains two Me_3Si singlets of equal intensity while the ^{13}C NMR spectrum contains two doublets in the Me_3Si region. The value of $J_{PNSiC} = 4.0$ Hz for the doublet is the very similar to that reported for the related compound $Me_3SiNPMe_3$.[14] In order for these NMR data to be accounted for by the isomeric phosphine oxide structure, it must be assumed that the Me_3Si nonequivalence results from hindered rotation about the N–P bond. This possibility can be rejected, however, because the rotational barrier in the starting aminophosphine **1** is very low[15] ($\Delta G^{\ddagger} < 7$ kcal/mol), and it has been shown elsewhere that oxidation results in a lowering of the N–P barrier.[16]

Table I. Proton and $^{13}C\{^1H\}$ NMR Data for [Bis(trimethylsilyl)amino]dimethylphosphine (**1**) and Its Derivatives (**2**–**5**)

		^1H NMR^a		^{13}C NMR^a	
Compd	Signal obsd	δ (^1H)	J_{PCH} or J_{PNSiCH}	δ (^{13}C)	J_{PC} or J_{PNSiC}
$Me_2PN(SiMe_3)_2$ (**1**)	Me_2P	1.34	6.6	19.27	22.0
	Me_3Si	0.24	1.0	4.51	7.9
$Me_2P(S)N(SiMe_3)_2$ (**2**)	Me_2P	1.90	13.0	28.75	67.1
	Me_3Si	0.35	0.0	5.43	2.4
$[Me_3PN(SiMe_3)_2]^+I^-$ (**3**)	Me_3P	2.31	13.0	18.96	64.1
	Me_3Si	0.48	0.0	5.21	1.8
$Me_2P(NSiMe_3)N(SiMe_3)_2$ (**4**)	Me_2P	1.37	13.3	31.35	75.7
	Me_3Si^b	0.05		11.62	
$Me_2P(NSiMe_3)OSiMe_3$ (**5**)	Me_2P	1.44	14.0	20.81	95.2
	Me_3SiN	0.29	0.0	3.52	4.0
	Me_3SiO	0.10	0.0	1.28	1.1

^{a} Chemical shifts in ppm downfield from external Me_4Si; coupling constants in Hz. Solvents (solute concentration): ^1H, CH_2Cl_2 (20%); ^{13}C, $CDCl_3$ (10–50%). ^{b} All Me_3Si groups are equivalent (see text).



$$(Me_3Si)_2NPMe_2 + {}^1/_2O_2 \rightarrow \left\{(Me_3Si)_2N-P(=O)Me_2\right\} \rightarrow Me_3Si-N=P(Me)_2-O-SiMe_3 \quad \mathbf{5} \qquad (9)$$

A second piece of spectroscopic evidence in favor of the imine structure for compound **5** is the observation of a large (relative intensity 90%) peak in the mass spectrum at *m/e* 150 corresponding to $[Me_3SiOPMe_2]^+$, a species most likely to result from fragmentation of the imine **5**.

There is also precedence in the literature for 1,3-silyl migrations from nitrogen to oxygen in systems where the central elements are carbon[5a,b,13] and sulfur,[5c] as well as phosphorus.[5d] This type of rearrangement, like the fluxional behavior of the silylaminophosphinimine **4**, is by no means a general phenomenon since some silylaminophosphine oxides have been prepared.[7,17,18] Again, the factors influencing the course of such reactions have not been fully investigated.

In summary, this study of the preparation and reactions of the silylaminophosphine **1** has revealed three different types of intramolecular silyl rearrangements: from phosphorus to nitrogen (eq 2), from nitrogen to nitrogen (compound **4**), and from nitrogen to oxygen (eq 9). Recently, we have reported a 1,3-silyl migration from nitrogen to carbon in a silicon–nitrogen–phosphorus compound.[19]

Finally, Table I contains some noteworthy observations. For instance, while there is a large increase in the value of the one-bond coupling (J_{PC}) upon oxidation of the aminophosphine **1** to its sulfide **2** or phosphonium salt **3**, there is a decrease in the three-bond coupling (J_{PNSiC}). Similar trends are found in the ^{1}H NMR data for the coupling constants J_{PCH} and J_{PNSiCH}.

Experimental Section

Materials. Sulfur, iodomethane, bis(trimethylsilyl)amine, and *n*-butyllithium were obtained from commercial sources and used without further purification. Chlorodimethylphosphine,[20] trimethylsilyldimethylphosphine,[21] and trimethylsilyl azide[22] were prepared and purified according to published procedures. Ethyl ether and benzene were distilled from calcium hydride prior to use. Other solvents were dried over molecular sieves.

General Procedures. Except where noted, all reactions and other manipulations were carried out under an atmosphere of dry nitrogen. Proton NMR spectra were recorded on Varian T-60 and JEOL MH-100 spectrometers and $^{13}C\{^1H\}$ NMR spectra were recorded on a JEOL FX-60 spectrometer. Infrared spectra were obtained on Perkin-Elmer 297 and 137 spectrophotometers. Mass spectra were obtained on an MS-902 spectrometer at an operating voltage of 70 eV. Elemental analyses were performed by the Schwarzkopf Microanalytical Laboratory, Woodside, N.Y.

Preparation of [Bis(trimethylsilyl)amino]dimethylphosphine (1). (a) From Me_2PCl and $LiN(SiMe_3)_2$. In a typical preparation, a solution of chlorodimethylphosphine (16.1 mmol) in ether (60 mL) was added with stirring over a 1-h period to an equimolar quantity of lithium bis(trimethylsilyl)amide (prepared from 16.1 mmol of $(Me_3Si)_2NH$ and 6.7 mL of a 2.4 M solution of *n*-BuLi in hexane) in ether (10 mL) at 0 °C. The mixture was then allowed to warm to room temperature and stirred for 2 h. After filtration and solvent removal, distillation afforded compound **1** as a colorless liquid (2.18 g, 61% yield; bp 41 °C (0.5 Torr)). IR spectrum (neat liquid): 3000 (s), 2950 (m), 1500 (m), 1340 (s), 1280 (w), 1250 (s), 950 (vs, br), 860 (vs, vbr), 764 (m), 682 (s), 650 (w), 628 (w) cm^{-1}. Mass spectrum [*m*/*e* (relative intensity) tentative assignment]: 221 (32) M^+, 206 (100) $M^+ - CH_3$, 147 (34), 130 (18) $NSi_2Me_4^+$, 118 (44) $PNSiMe_3^+$, 73 (56) Me_3Si^+, 59 (16), 45 (17) PN^+. Anal. Calcd for $C_8H_{24}NPSi_2$: C, 43.39; H, 10.93. Found: C, 42.78; H, 10.50.

(b) From Me_3SiPMe_2 and Me_3SiN_3. By using a conventional high-vacuum system, equimolar quantities (1.63 mmol) of the reactants were condensed together at −196 °C in a 60-mL tube equipped with a glass–Teflon stopcock. The mixture was allowed to warm to room temperature and then heated in a water bath at 80 °C for 2 h. The vessel was then cooled to −196 °C and opened on the vacuum line to remove a noncondensable gas (~1.8 mmol) presumed to be nitrogen. The remaining products were then distilled through a U-trap cooled to −20 °C which retained compound **1** as a colorless liquid (0.25 g, 70% yield). The IR and ^{1}H NMR spectra of this material were identical with those observed for the product of the $Me_2PCl + LiN(SiMe_3)_2$ reaction described above.

Preparation of [Bis(trimethylsilyl)amino]dimethylphosphine Sulfide (2). Compound **1** (9.01 mmol) was added slowly to a stirred suspension of sulfur (9.06 mmol) in benzene (10 mL) at 25 °C. After 40 min of stirring, a ^{1}H NMR spectrum of the reaction mixture indicated quantitative formation of the phosphine sulfide and no detectable side products. The solvent was removed under vacuum leaving **2** as a white solid (mp 40–45 °C). Further manipulations of the product were invariably accompanied by hydrolysis. The hydrolysis product, $Me_3SiN(H)P(S)Me_2$, was precipitated from a solution of **2** after exposure to the atmosphere. Recrystallization from hexane gave a white solid which was identified by its mp 70–75 °C (lit.[11] mp 75 °C) and its ^{1}H NMR spectrum (20% v/v in CH_2Cl_2): Me_2Si singlet (δ 0.24), Me_2P doublet (δ 1.77, J_{PCH} = 13.2 Hz), and NH singlet (δ 2.39 broad).

Preparation of [Bis(trimethylsilyl)amino]trimethylphosphonium Iodide (3). A solution of iodomethane (4.82 mmol) in benzene (0.5 mL) was added with stirring to a solution of compound **1** (4.58 mmol) in benzene (2 mL) at 0 °C. A white precipitate formed immediately. The solvent was removed under vacuum leaving **3** as a white solid which was identified by its mp 156.5–158.5 °C (lit.[9] mp 154–155 °C) and NMR spectroscopy (Table I). No side products or residual starting material were detected in the NMR spectra of **3**. Further characterization was precluded by the extreme moisture sensitivity of the compound.

Preparation of *P*-Bis(trimethylsilyl)amino-*P*,*P*-dimethyl-*N*-trimethylsilylphosphinimine (4). A mixture of compound **1** (28.8 mmol) and trimethylsilyl azide (30.1 mmol) was heated (55–60 °C) with stirring for 24 h, during which time evolution of a gas was observed. Distillation afforded compound **4** as a colorless liquid (6.65 g, 75% yield; bp 78–80 °C (0.4 Torr), lit.[6] bp 70–74 °C (0.1 Torr)). IR spectrum (neat liquid): 2950 (s), 1420 (w, br), 1255 (s), 1230 (s, br), 980 (s), 930 (s), 840 (s, br), 765 (w), 740 (w), 710 (w), 665 (m), 650 (m) cm^{-1}.

Preparation of *P*,*P*-Dimethyl-*P*-trimethylsiloxy-*N*-trimethylsilylphosphinimine (5). Oxygen, dried by passing slowly through a trap cooled to −78 °C, was bubbled through a solution of compound **1** (23.4 mmol) in methylene chloride (20 mL) at 25 °C. As determined by ^{1}H NMR spectroscopy, the reaction was complete in about 45 min. After removal of the solvent under vacuum, distillation afforded **5** as a colorless liquid (2.72 g, 49% yield; bp 38–41 °C (1.7 Torr)). IR spectrum (20% v/v in CCl_4): 2900 (m), 1320 (s, br), 1287 (vs), 1255 (s), 1000 (s, br), 953 (m), 850 (vs, br), 740 (m, br) cm^{-1}. Mass spectrum [*m*/*e* (relative intensity) tentative assignment]: 237 (15) M^+, 222 (100) $M^+ - CH_3$, 166 (12), 150 (90) $Me_3SiOPMe_2^+$, 134 (21) Me_3SiOPN^+, 120 (10) Me_3SiOP^+, 77 (28) Me_2PO^+, 75 (22) Me_2PN^+, 73 (46) Me_3Si^+, 59 (15) $MeSiO^+$, 47 (12) PO^+, 45 (15) PN^+. Anal. Calcd for $C_8H_{24}NOPSi_2$: C, 40.47; H, 10.19. Found: C, 40.38; H, 10.34.

The thermal stability of compound **5** was determined by heating a neat sample under vacuum at 130 °C for 3 days. Proton NMR analysis indicated that the material was unchanged.

Registry No. 1, 63744-11-6; **2**, 63744-10-5; **3**, 63744-08-1; **3** (ionic form), 63744-09-2; **4**, 21385-93-3; **5**, 63744-07-0; $Me_3SiN(H)P(S)Me_2$, 42346-45-2; Me_2PCl, 811-62-1; $LiN(SiMe_3)_2$, 4039-32-1; Me_3SiPMe_2, 26464-99-3; Me_3SiN_3, 4648-54-8; iodomethane, 74-88-4; ^{13}C, 14762-74-4.

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