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Rearrangement of a Phosphorus–Carbon–Phosphorus Bridge to a Phosphorus–Nitrogen–Phosphorus Bridge via Organogermanium- or Organotin-Assisted Cleavage of a Phosphorus–Carbon Bond. Crystal and Molecular Structure of the Imine Salt $(CH_3)Ph_2PNPPh_2(NH_2)^+Cl^-$

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When $Me_3SiN{=}PPh_2CH_2PPh_2{=}NSiMe_3$ is treated with organogermanium halides in the presence of water, the trimethylsilyl groups are removed and the backbone of the molecule is rearranged to yield quantitatively the imine salt $(CH_3)Ph_2PNPPh_2(NH_2)^+Cl^-$. Unassisted hydrolysis gave only the methylene-bridged dioxide $Ph_2(O)PCH_2P(O)Ph_2$. The imine salt structure (crystal data: triclinic $P\bar{1}$, $a = 11.034$ (3)Å, $b = 13.724$ (3) Å, $c = 8.816$ (3) Å, $\alpha = 99.55$ (2)°, $\beta = 109.30$ (3)°, $\gamma = 106.15$ (2)°, $V = 1160.2$ Å^3, $Z = 2$; $R_1 = 0.043$ and $R_2 = 0.052$ for 4079 unique reflections) contains a centrosymmetric dimeric hydrogen-bonded pair of cations in which the hydrogens of the NH_2 groups of the two cations are associated with two chlorides. The cation structure has a bent (142.9°) P=N=P backbone with short P=N bond lengths (1.57 Å) compared to the longer (1.62 Å) P–N bond for the P–NH_2 feature.

Alkyl diphosphines and other tertiary phosphines are extensively employed as versatile complexing ligands with a variety of transition-metal or organometallic substrates because of their ability to stabilize metals in a variety of oxidation states.[2-5] Recently, the propensity of metal–phosphine complexes to suffer phosphorus–carbon bond scission under certain circumstances has been probed[3,6-9] because deactivation of some homogeneous phosphine-based catalyst systems has been attributed to this reaction.[3]

As part of our studies of the reactions of the heterodifunctional methylene-bridged iminophosphorane phosphines $RN{=}PPh_2CH_2PPh_2$[10-14] and the related, doubly oxidized analogue $Me_3SiN{=}PPh_2CH_2PPh_2{=}NSiMe_3$ (**1**), we attempted a transmetalation reaction of the latter, which resulted instead in an unusual rearrangement of the molecular backbone. Further investigation revealed generality of behavior with organogermanium and -tin halides. We describe our results because the reaction yields new insight into an unusual phosphorus–carbon bond scission reaction pathway, which may also provide information illuminating the causes of catalyst deactivation resulting from similar reactions in homogeneous catalysis.[3]

Experimental Section

Reactions were carried out under dry nitrogen with standard precautions taken to dry the solvents and equipment. NMR spectra were obtained on 5–10% solutions in appropriate solvents by using WM-400 and WH 200 Bruker instruments.

Preparation of $Me_3SiN{=}PPh_2CH_2PPh_2{=}N(SiMe_3)GeMe_3{}^+Cl^-$ (3). To a solution of **1** (1.75 g; 3.13 mmol) in toluene was added dropwise with stirring at 25 °C a solution of Ph_3GeCl (1.06 g; 3.13 mol) also in toluene. The mixture was heated under reflux for 2 h before the solvent was removed in vacuo to obtain a white crystalline solid of **3** (yield 97%); mp 230–235 °C dec. Anal. Calcd for $C_{49}H_{55}ClN_2GeP_2Si_2$: C, 65.52; H, 6.13; N, 3.12; Cl, 3.95. Found: C, 65.49; H, 6.10, N, 3.10; Cl, 3.92. ^1H NMR ($CDCl_3$): PCH_2P, δ 3.15, (t, 2 H; $^2J_{PH} = 12.10$ Hz); phenyl rings, δ 7.35–7.80 (m, 35 H); Me_3Si (2 different groups), δ 0.10 (s, 9 H), 0.14 (s, 9 H). ^{31}P NMR (161.98 MHz in $CDCl_3$, ppm vs 85% H_3PO_4): $\delta(P_A)$ (due to $Ph_2P{=}N(SiMe_3)GePh_3$) 5.94; $\delta(P_B)$ (due to $Ph_2P{=}NSiMe_3$) −4.95 ($^2J_{P_AP_B} = 20.05$ Hz).

"One-Pot" Route to 4. A solution of **1** (2.16 g; 3.87 mmol) in wet acetonitrile (50 mL) was added dropwise with stirring at 25 °C to a solution of Ph_3GeCl (1.31 g; 3.87 mmol) also in acetonitrile (50 mL). The mixture was heated under reflux for 6 h before the solvent was removed in vacuo to obtain a shiny white crystalline analytically pure phosphazene salt **4**[17,18] (yield 95%); mp 220 °C. Anal. Calcd for $C_{25}H_{25}ClN_2P_2$: C, 66.60; H, 5.55; N, 6.20; Cl, 7.86. Found: C, 66.47; H, 5.49; N, 6.20; Cl, 7.81. ^1H NMR ($CDCl_3$): CH_3, δ 2.65 (d, 3 H, $^2J_{PH} = 13.0$ Hz); phenyl rings, δ 7.38, 7.60, 7.70 (m, 20 H). ^{31}P NMR (161.98 MHz in $CDCl_3$, ppm vs 85% H_3PO_4: $\delta(P_A)$ 20.90; $\delta(P_B)$ 20.78 ($J_{P_AP_B} = 2.91$ Hz).

Crystal and Molecular Structure of 4. Data were collected at 21 °C on an Enraf-Nonius CAD4 diffractometer with monochromatic Mo Kα ($\lambda = 0.71073$ Å) radiation (graphite-crystal monochromator, Zr foil attenuator (factor 17.8), take-off angle 2.8°, scan rate 2–7°/min in ω, ω–2θ scan technique, detector aperature 2.4-mm horizontal, 4.0-mm vertical). Crystal data: Triclinic $P\bar{1}$ (No. 2) with $a = 11.034$ (3) Å, $b = 13.724$ (3) Å, $c = 8.816$ (3) Å, $\alpha = 99.55$ (2)°, $\beta = 109.30$ (3)°, $\gamma = 106.15$ (2)°, $V = 1160.2$ Å^3, $Z = 2$. Lorentz–polarization, linear decay reflection averaging, and empirical absorption corrections were applied. Direct methods were used for solution. Final indices were $R_1 = 0.043$ and $R_2 = 0.052$ for 4079 unique reflections. Full details are given in the supplementary material. Selected important bonding parameters are given in Table I.

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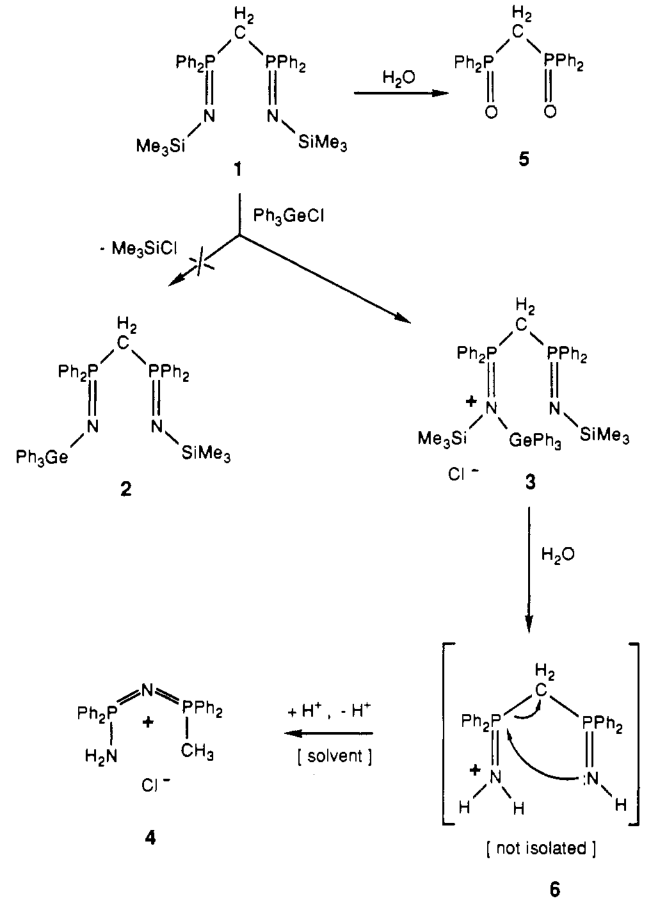
Scheme I



Hydrolysis of 1. An aqueous solution of $Me_3SiN=PPh_2CH_2Ph_2P=NSiMe_3$ (**1**)[19] (1.75 g; 3.13 mmol) in acetonitrile (50 mL CH_3CN; 10 mL H_2O) was refluxed for 0.5 h before the solvents and other volatile residues were removed in vacuo to give **5** as a white crystalline solid (yield 95%; mp 180 °C (lit.[20] mp 181–182 °C)). ^{31}P NMR ($CDCl_3$): δ 24.70 (lit.[20] NMR: δ 24.20).

Reaction of 7 with Water. When $Me_3SiN=PPh_2CH_2PPh_2$ was subjected to experimental conditions similar to those described for **1**, **8** was produced quantitatively as a white crystalline solid. ^{31}P NMR ($CDCl_3$): $\delta(P^{III})$ −28.5; $\delta(P^V)$ 27.3 ppm ($^2J_{PP}$ = 50 Hz) (lit.[21] NMR: $\delta(P^{III})$ −28.4; $\delta(P^V)$ 27.7; ($^2J_{PP}$ = 50 ± 1 Hz)). Reaction of **1** in the presence of Ph_3GeCl or Ph_3SnCl as described above again gave only **8**.

Results and Discussion

We attempted a synthetic approach to the mixed *N*-silyl-*N'*-germylbis(iminophosphoranyl)methane of the type **2** (Scheme I) via treatment of bis(((trimethylsilyl)imino)phosphoranyl)methane (**1**) with Ph_3GeCl in refluxing toluene. The product was a white crystalline solid, which was identified as **3** rather than the desired product **2**[15] (Scheme I). The composition of **3** was confirmed by spectroscopic and analytical data. Hydrolyzing **3** in refluxing wet acetonitrile or in 95% ethanol produced the diphosphazene salt **4** (Scheme I) by means of a rearrangement of the backbone of the molecule in which a P–N–P bridge was formed at the expense of one P–C backbone bond. In contrast, hydrolysis of such compounds frequently splits the molecule at one of the P–C methylene backbone bonds yielding two monophosphorus fragments[16] or (vide infra) converts the imines to oxides. **4** was fully characterized by analysis, ^1H and ^{31}P NMR spectroscopy, and a single-crystal X-ray diffraction study (Figure 1 and Table I). This same salt **4** was reported by Appel et al.[18] as the product of reaction of bis(diphenylphosphino)methane (dppm) with NH_3 and CCl_4 in an autoclave at 40 °C, and subsequently Appel and Ruppert[19] showed that 3 M HCl in ether converted **1** to **4**.

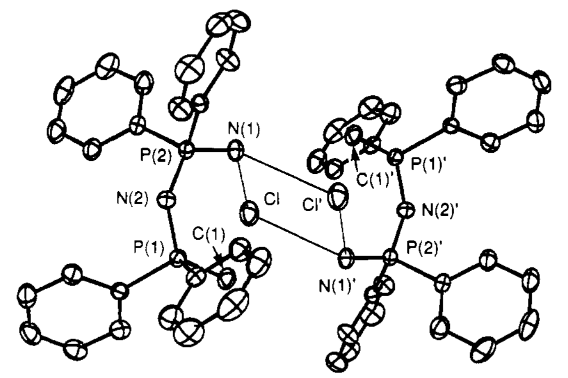


Figure 1. ORTEP drawing of $(CH_3)Ph_2P=N=PPh_2NH_2^+Cl^-$, showing the labeling scheme. Note that the unique methyl carbon atoms (C(1)) are slightly obscured by the phenyl rings. Hydrogen atoms are not shown, and all other atoms are shown as 30% probability ellipsoids.

Table I. Selected Bond Distances[a] (Å) and Angles[a] (deg)

Distances			
P(1)–N(2)	1.573 (3)	P(2)–C(14)	1.786 (3)
P(1)–C(1)	1.777 (3)	P(2)–C(20)	1.788 (3)
P(1)–C(2)	1.793 (3)	N(1)–H(24)	0.88 (3)
P(1)–C(8)	1.796 (3)	N(1)–H(25)	0.91 (4)
P(2)–N(1)	1.624 (2)	Cl–H(24)	2.38 (3)
P(2)–N(2)	1.572 (3)	Cl–H(25)	2.42 (4)
Angles			
N(2)–P(1)–C(1)	114.8 (1)	N(1)–P(2)–C(20)	106.1 (1)
N(2)–P(1)–C(2)	108.8 (1)	N(2)–P(2)–C(14)	108.0 (1)
N(2)–P(1)–C(8)	109.1 (2)	N(2)–P(2)–C(20)	108.2 (1)
C(1)–P(1)–C(2)	108.9 (2)	C(14)–P(2)–C(20)	108.0 (1)
C(1)–P(1)–C(8)	106.9 (1)	P(1)–N(2)–P(2)	142.9 (1)
C(2)–P(1)–C(8)	108.1 (1)	Cl–H(24)–N(1)	169 (3)
N(1)–P(2)–N(2)	121.1 (1)	Cl–H(25)–N(1)	162 (3)
N(1)–P(2)–C(14)	104.9 (1)		

[a] Numbers in parentheses are estimated standard deviations in the least significant digits.

Strong hydrolysis conditions are reported[18] to convert **4** to $Ph_2P(O)N=PPh_2NH_2$. Notably, however the higher homologues with ethylene and propylene backbones gave phosphoranimine ring products with no P–C bond cleavage.[18,19] The susceptibility of compound **3** to hydrolysis and the facility of the reaction with limited quantities of water allows access to **4** via a one-step synthetic route by the reaction of **1** with Ph_3GeCl in undistilled (wet) acetonitrile solvent. Trimethylgermanium chloride also gave **4** in yields similar to those provided by Ph_3GeCl. Parallel reactions of **1** with triphenyltin chloride also gave **4** in yields almost similar to those provided by Ph_3GeCl or Me_3GeCl. Aqueous or alcoholic hydrolysis of **1** alone did not produce **4**; rather, the diphosphine dioxide derivative **5** was isolated quantitatively from this reaction (Scheme I).

It therefore appears that the rearrangement reaction herein observed is mediated in some way by the organometal halide. A plausible mechanism for this transformation is included in Scheme I. The key step appears to be the generation of a phosphinimine intermediate **6**, and the initiation of the reaction may depend on the enhanced hydrolytic sensitivity of the quaternized salt. The nucleophilic attack of the basic imino nitrogen center on the electrophilic phosphorus[19] probably activates the P–C bond. The carbanion so generated can abstract hydrogen either from the imino nitrogen, the amino nitrogen, the solvent, or silyl or germyl sources to give **4**. It does not seem necessary to invoke the bimolecular intermediate proposed earlier.[19]

When $Me_3SiN=PPh_2CH_2PPh_2$[10] (**7**) was treated with Ph_3GeCl or Ph_3SnCl under the experimental conditions that produced **4** from **1**, only the monooxidized phosphine oxide $(O)PPh_2CH_2PPh_2$ was obtained in quantitative yields (eq 1). **8** is also quantitatively produced by the simple hydrolysis reaction of **7** with water or

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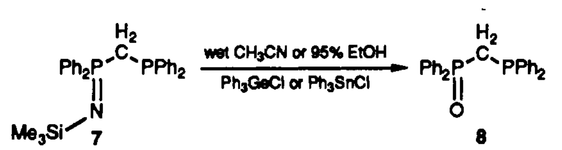

(1)

ethanol. Formation of **8** from the above reaction contrasts the formation of the diphosphazene **4** from **1** (Scheme I), and it is clear that the rearrangement involves a cooperative interaction of two iminophosphorane centers. This result in conjuction with our findings clearly establishes that the P–C–P links in dppm derivatives are more reactive than the phosphorus–aryl carbon bonds in such compounds. In contrast, phosphorus–aryl carbon bond scission reactions are observed for a number of tertiary phosphine–metal complexes.[3]

Several interesting features are provided by the structure of **4**. Most notable is the observation of a hydrogen-bonding interaction between the chloride and the amino hydrogens. This presumably accounts for the dimeric, centrosymmetric solid-state structure exhibited by **4**. The P–N bond lengths (1.57 Å) within the PNP segment are equivalent and are clearly characteristic of P–N double bonds. The long P–N bond (1.62 Å) for the protonated nitrogen also falls within the range for compounds with isolated P–N double bonds,[22-26] but the bond is shorter than the value expected for a single bond. Qualitatively the compound is clearly best formulated as an aminodiphosphinimine

$$H_2N—P(Ph_2){=}N^+{=}PPh_2Me$$

with each P doubly bonded to an imine nitrogen. The charge is probably best placed on the bridge nitrogen. The P=N=P angle is 142.9°. All of these structural features compared well with those of $Ph_3P{=}N{=}PPh_3^+$ (PN bond lengths (1.575 Å (average), P=N=P angle 136°)[25] and the backbone PN bonds of $(H_2N)Ph_2P{=}N{=}PPh_2(NH_2)^+$ (1.575 Å) (average), P=N=P angle 142°)[31]. The terminal NH_2 groups in the latter are similarly bonded to phosphorus ($P{-}NH_2$ = 1.65 Å (average))[26] as in the case of **4**.

Investigations into the chemistry of this interesting compound and into the influence of aryl- or alkylgermanium and tin halides on the hydrolysis pathways of transition-metal–phosphine complexes are underway.

Acknowledgment. We thank the Natural Sciences and Engineering Research Council of Canada for Financial support.

Supplementary Material Available: For **4**, text describing the structure solution, listings of X-ray details, bond distances, bond angles, positional and isotropic equivalent displacement parameters, general displacement (*U*) parameters, and ^{13}C NMR data, and figures showing alternative ORTEP views and a ^{13}C NMR spectrum (14 pages); a table of observed and calculated structure factors (16 pages). Ordering information is given on any current mesthead page.

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Synthesis and Characterization of the Neutral Three-Coordinate Thiolate Complexes $Al[S(2,4,6\text{-}t\text{-}Bu_3C_6H_2)]_3$ and $Ga[S(2,4,6\text{-}t\text{-}Bu_3C_6H_2)]_3$

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The first monomeric, three-coordinate aluminum and gallium thiolate compounds have been synthesized and structurally characterized. The compounds were prepared by the reaction of the corresponding metal halide with 2 equiv of LiSMes* (Mes* = 2,4,6-*t*-$Bu_3C_6H_2$) to give the aluminum derivative $Al(SMes^*)_3$ (**1**) or with 3 equiv of the lithium thiolate to give the gallium species $Ga(SMes^*)_3$ (**2**). The compounds were characterized by ^1H NMR and infrared spectroscopy and X-ray crystallography. Crystal data with Mo Kα (λ = 0.71069 Å) radiation at 130 K: **1**, $Al(SMes^*)_3$, $C_{54}H_{87}AlS_3$, a = 10.229 (5) Å, b = 26.137 (7) Å, C = 19.908 (5) Å, β = 91.260 (3)°, Z = 4, monoclinic, space group $P2_1/n$, $R(F)$ = 0.049; **2**, $Ga(SMes^*)_3$, $C_{54}H_{87}GaS_3$, a = 10.243 (2) Å, b = 26.189 (8) Å, c = 20.031 (3) Å, β = 91.554 (1)°, Z = 4, monoclinic, space grup $P2_1/n$, $R(F)$ = 0.049. The structural data show that **1** and **2** have a very similar three-coordinate, almost planar, geometry at the metal. Distortions from idealized trigonal-planar coordination, as well as other structural features can be explained on the basis of ionic M–S bonding character and of some M···H interactions involving the *o*-*t*-Bu groups of the ligand.

Introduction

In spite of the large amount of research on the inorganic and organometallic chemistry of heavier main-group 3 (Al–Tl) complexes, relatively little is known about their compounds with low (3 or less) coordination numbers. For example, only a small number of such compounds have been structurally characterized in the solid state,[1-14] and most of these have been published within the last 5 years. In the case of Al and Ga derivatives, the compounds $Al[N(SiMe_3)_2]_3$,[1] $MMes_3$ (M = Al,[2] Ga[3]), $Ga(AsMes_2)_3$,[4] $Ga[P(H)Mes^*]_3$,[5] $(\eta^1\text{-}C_5Me_5)_2GaAs(SiMe_3)_2$,[6] $MeAl[O(2,6\text{-}t\text{-}Bu_2\text{-}4\text{-}MeC_6H_2)]_2$,[7] R_2MMR_2 [M = Al,[8] Ga;[9] R = $-CH(SiMe_3)_2$],

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