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Triphosphane formation from the terminal zirconium phosphinidene complex $[Cp_2Zr{=}PDmp(PMe_3)]$ $(Dmp = 2,6\text{-}Mes_2C_6H_3)$ and crystal structure of $DmpP(PPh_2)_2$

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Abstract

The new terminal phosphinidene complex $[Cp_2Zr{=}PDmp(PMe_3)]$ ($Dmp = 2,6\text{-}Mes_2C_6H_3$; **1**) was prepared in 81% yield by the reaction of $[Li(Et_2O)][P(H)Dmp]$ with $[Cp_2Zr(Me)Cl]$ in the presence of excess PMe_3. Compound **1** reacts with Ph_2PCl to produce selectively the sterically congested triphosphane $DmpP(PPh_2)_2$ (**2**) and $[Cp_2ZrCl_2]$ in high yields. The structure of **2** obtained by X-ray diffraction analysis of a single crystal reveals phosphorus–phosphorus bond lengths of 2.251(2) and 2.234(2) Å and a PPP bond angle of 105.46(6)°. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Phosphorus; Phosphinidene complexes; Triphosphane; Crystal structures; Electrophile addition

1. Introduction

Organozirconium complexes have found widespread use in the synthesis of new organophosphorus compounds [1]. In particular, a number of fascinating materials have been prepared using the readily prepared terminal phosphinidene complex $[Cp_2Zr{=}PMes^*(PMe_3)]$ ($Mes^* = 2,4,6\text{-}{}^tBu_3C_6H_2$) to deliver the phosphinidene ‘PMes*’ [2,3]. The high oxo- and chlorophilicity of the zirconium center in $[Cp_2Zr{=}PMes^*(PMe_3)]$ will readily allow exchange of its phosphinidene unit for two chlorine atoms or an oxygen atom of a substrate to form either $[Cp_2ZrCl_2]$ or $[Cp_2ZrO]_n$, respectively. For example, reaction of $[Cp_2Zr{=}PMes^*(PMe_3)]$ with dichloromethane affords a rapid and convenient synthesis of the methylenephosphaalkene $Mes^*P{=}CH_2$ in good yields (Eq. (1)).

$$[Cp_2Zr{=}PMes^*(PMe_3)] + CH_2Cl_2 \rightarrow [CP_2ZrCl_2] + Mes^*P{=}CH_2\ (86\%) \qquad (1)$$

Phosphinidene transfers using this reagent are also equally effective for other phosphorus–heteroatom bond forming processes using germanium, silicon, and tin halides [2].

We are interested in developing zirconium-based transfer chemistry for phosphinidenes bearing *meta*-terphenyl [4,5] protected phosphorus centers [6–21]. In particular, we sought new methods for constructing phosphorus–phosphorus bonds that might utilize zirconium phosphinidene complexes [13]. Herein we present the synthesis of a terminal phosphinidene complex bearing a *meta*-terphenyl ligand and its reaction with a chlorophosphine to generate a novel triphosphane.

2. Results and discussion

Stephan and Breen have illustrated that the terminal phosphinidene complex $[Cp_2Zr{=}PMes^*(PMe_3)]$ is produced in high yields by reaction of $[Li(THF)_3]$-$[P(H)Mes^*]$ with $[Cp_2Zr(Me)Cl]$ in the presence of excess PMe_3 [2]. Accordingly, this method was chosen for synthesis of the desired complex bearing the *meta*-terphenyl Dmp ($Dmp = 2,6\text{-}Mes_2C_6H_3$) in place

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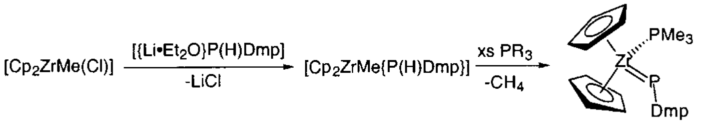
of the supermesityl (Mes*) group. The new terminal phosphinidene complex [Cp_2Zr=PDmp(PMe_3)] (**1**) was thus isolated as a deep green crystalline material by reaction of [Cp_2Zr(Me)Cl] and [Li(Et_2O)][P(H)Dmp] in the presence of excess PMe_3 in 81% yield (Eq. (2)).

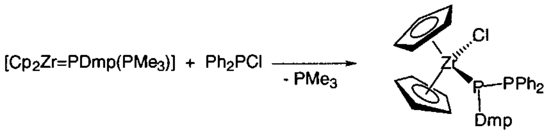


1

(2)

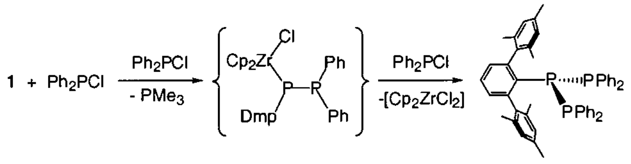
Compound **1** is thermally stable in the absence of air or water. The presumed precursor to **1**, [Cp_2Zr(Me)-{P(H)Dmp}], can be isolated as a somewhat unstable orange solid if the reaction is performed in the absence of trialkylphosphine (^{31}P-NMR (C_6D_6): δ 33.3 (d, $J_{PH} = 255$ Hz)). However, attempts to purify this material completely were unsuccessful. The ^{1}H- and ^{31}P{^{1}H}-NMR spectra of **1** are in accord with the previously characterized zirconium phosphinidene complex [3]. For example, the phosphinidene signal in the ^{31}P{^{1}H}-NMR of **1** is found at δ 771.0 (d, $J_{PP} = 23$ Hz), which can be compared to data for [Cp_2Zr=PMes*(PMe_3)]: δ 792.4 (d, $J_{PP} = 23$ Hz). Unfortunately, a high quality X-ray structure of **1** could not be obtained. A preliminary structure, however, confirmed the expected gross structural features of **1**. In particular, a short Zr=P bond length of $d_{Zr=P} = 2.446(8)$ Å (compare to $d_{Zr=P} = 2.505(4)$ Å for [Cp_2Zr=PMes*(PMe_3)]) and a significantly bent zirconium phosphinidene array with a Zr=P–C angle of 123.2(8)° could be ascertained.

We previously reported on the structural characterization of the stable phosphanido complexes [Cp_2Zr(Cl)-{P(H)Dmp}] and [Cp_2Zr{P(H)Dmp}$_2$] [20]. Although Group 4 phosphanido complexes are well-known species, species of the form [Cp_2M(Cl){P(PR_2)R}] are rare or unknown [22]. Thus the reaction of **1** with Ph_2PCl was examined with the anticipation that [Cp_2Zr(Cl)-{P(PPh_2)Dmp}] might be isolable (Eq. (3)).



(3)

Reaction of equimolar amounts of **1** and Ph_2PCl is rapid and affords a single new organophosphorus species. Surprisingly, the new species is not a zirconium phosphanido complex but the novel triphosphane DmpP(PPh_2)$_2$ (**2**; Eq. (4)). The triphosphane displays very diagnostic ^{31}P resonances at δ −18.0 (d, $J_{PP} = 221$ Hz) and −52.3 (t, $J_{PP} = 221$ Hz). As expected for the stoichiometry of the reaction, the ^{31}P-NMR spectrum of the reaction mixture shows remaining **1**. If the reaction is performed with two equivalents of Ph_2PCl, then **1** is consumed completely and the triphosphane **2** is produced in essentially quantitative yield (> 95% by ^{1}H-NMR).



2

(4)

Crystals of **2** were grown from a THF–hexanes mixture at −35°C and the results of a single-crystal X-ray structure determination is presented in Fig. 1 and Tables 1 and 2. Despite the severe steric crowding in the molecule, the geometry at each phosphorus atom is

Table 1
Crystal data and structure refinement for DmpP(PPh_2)$_2$ (**2**)

Empirical formula	$C_{48}H_{45}P_3$
Formula weight	714.75
Temperature (K)	173(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	
a (Å)	11.2205(2)
b (Å)	21.8735(4)
c (Å)	16.0223(3)
α (°)	90
β (°)	93.7125(8)
γ (°)	90
V (Å^3)	3924.14(17)
Z	4
D_{calc} (Mg m^{-3})	1.210
Absorption coefficient (cm^{-1})	1.85
F(000)	1512
Crystal size (mm)	0.45 × 0.25 × 0.15
θ Range for data collection	2.04–24.00°
Index ranges	$-11<h<12$, $-25<k<22$, $-18<l<17$
Reflections collected	13 597
Independent reflections	5620 [$R_{int} = 0.0599$]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	5620/0/460
Goodness-of-fit on F^2	2.103
Final R indices [$I>2\sigma(I)$]	$R_1 = 0.0784$, $wR_2 = 0.1749$
R indices (all data)[a,b]	$R_1 = 0.1026$, $wR_2 = 0.1860$
Largest difference peak and hole (e Å^{-3})	0.442 and −0.441

[a] $R(F) = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.

[b] $R_w(F^2) = [\Sigma\{w(F_o^2 - F_c^2)^2\}/\Sigma\{w(F_o^2)^2\}]^{0.5}$; $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, where $P = [F_o^2 + 2F_c^2]/3$ and a and b are constants adjusted by the program.

Table 2
Selected bond lengths and bond angles for $DmpP(PPh_2)_2$ (**2**)

P(1)–P(2)	2.2516(15)	C(1)–P(1)–P(3)	102.96(13)
P(1)–P(3)	2.2343(15)	C(1)–P(1)–P(2)	104.15(13)
P(1)–C(1)	1.859(5)	P(3)–P(1)–P(2)	105.46(6)
P(2)–C(25)	1.867(4)	C(2)–C(1)–P(1)	111.5(3)
P(2)–C(31)	1.846(5)	C(6)–C(1)–P(1)	128.9(3)
P(3)–C(37)	1.842(4)	C(31)–P(2)–C(25)	99.4(2)
P(3)–C(43)	1.844(4)	C(31)–P(2)–P(1)	95.84(14)
		C(25)–P(2)–P(1)	98.90(13)
		C(43)–P(3)–C(37)	101.68(19)
		C(43)–P(3)–P(1)	100.33(14)
		C(1)–C(6)–C(16)	124.3(4)
		C(3)–C(2)–C(7)	120.5(4)
		C(37)–P(3)–P(1)	105.60(13)

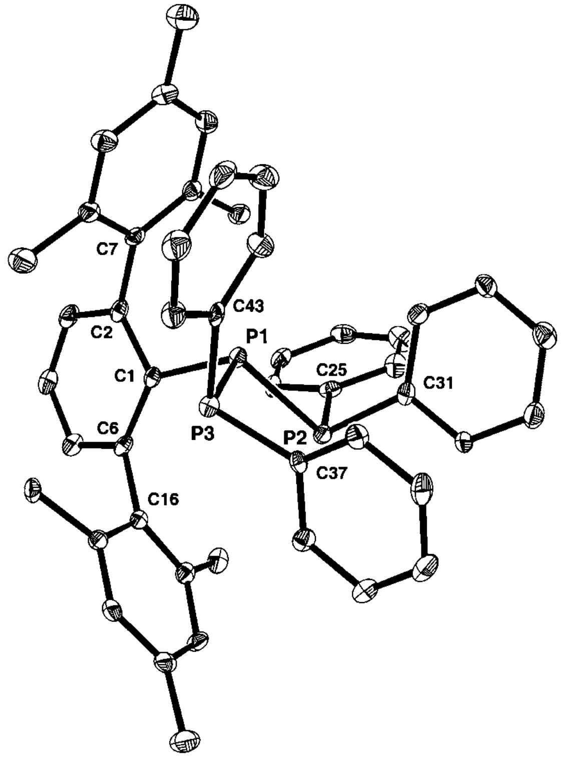


Fig. 1. Thermal ellipsoid plot for $DmpP(PPh_2)_2$.

essentially pyramidal. The PP bond lengths of 2.251(2) and 2.234(2) Å and the PPP bond angle of 105.46(6)° are comparable to values reported for two recent crystallographically characterized silyltriphosphanes. For example, in $^{i}Pr_3SiP(PPh_2)_2$ the PP bond lengths are 2.243(1) and 2.217(1) Å and the PPP bond angle is 100.0(1)° [23]. Likewise, $Me_3SiP(P^{t}Bu_2)_2$ displays PP bond lengths of 2.1895(4) and 2.1964(4) Å and a PPP bond angle of 118.72(2)° [24]. All of the values are in rough agreement with ab initio calculations (HF/6-31G** level of theory) for hypothetical P_3H_5 that predict the most stable isomer to have PP bond lengths of 2.211 Å and a PPP bond angle of 110.27° [25].

Several other structural features of **2** merit comment. First, two pairs of nearly coplanar aromatic rings can be discerned (Fig. 1) that might suggest π stacking. The pair of phenyl rings containing atoms C(31) and C(37) deviate from coplanarity by only 18.9°, and have a centroid–centroid distance of 4.02 Å. The pair of aromatic rings involving atoms C(7) and C(43) are also nearly coplanar (13.7°), but lie at a slightly greater distance from one another (centroid–centroid distance of 4.54 Å). Second, the two PPh_2 groups are directed toward one of the two mesityl rings, and the resulting steric clashes force the $P(PPh_2)_2$ unit further away from this mesityl ring. This effect is evinced by a 17° disparity in the corresponding C(*n*)–C(1)–P(1) bond angles (C(*n*) = C(2) and C(6)). Third, steric clashes between the $P(PPh_2)_2$ unit and the Dmp group also force P(1) out of the plane of the phenyl ring containing C(1) by 0.43 Å. The Dmp ligand also reacts to the steric pressures by moving the mesityl rings away from the PPh_2 unit, as indicated by movement of the *ipso*-carbons of the mesityl rings to the opposite side of the central phenyl ring by 0.28 and 0.31 Å for C(7) and C(16), respectively. These structural distortions give rise to a P(1)···C(7) ring centroid distance of 3.74 Å, which is longer than the Menshutkin-type interactions observed in the related *meta*-terphenyls $2,6\text{-}Ar_2\text{–}C_6H_3ECl_2$ (Ar = Mes or $2,4,6\text{-}^{i}Pr_3C_6H_2$; E = As, Bi or Sb; $d_{E\cdots centroid} \sim 3.4$ Å) [14].

A reasonable pathway for the formation of $DmpP(PPh_2)_2$ is outlined in Eq. (4). The putative intermediate $[Cp_2Zr(Cl)\{P(PPh_2)Dmp\}]$, although not stable, finds precedent in the related reaction of $[Cp_2Zr{=}PMes^*(PMe_3)]$ and Me_2SiCl_2. This reaction produces $Mes^*P(SiMe_2Cl)_2$, but if the reaction stoichiometry is 0.5 $[Cp_2Zr{=}PMes^*(PMe_3)]$ to 1.0 Me_2SiCl_2, the intermediate phosphanido complex $[Cp_2Zr(Cl)\{P(Mes^*)Si(Cl)Me_2\}]$ can be isolated in 71% yield [2].

The inability to isolate $[Cp_2Zr(Cl)\{P(Dmp)PPh_2\}]$ is probably not a consequence of steric factors. Related phosphanido complexes $[Cp_2Zr(Cl)\{P(H)Dmp\}]$ and $[Cp_2Zr\{P(H)Dmp\}_2]$ have been prepared and characterized structurally [20]. The latter material is quite thermally robust. Likewise, the extremely hindered mono-phosphanido complex $[Cp_2Zr(Cl)\{P(Mes^*)SiMe_3\}]$ shows noteworthy thermal and photochemical stability [26]. The failure to isolate $[Cp_2Zr(Cl)\{P(Dmp)PPh_2\}]$ probably lies in its enhanced reactivity with Ph_2PCl (relative to **1**).

Alternative mechanisms for formation of **2** involving radical intermediates (such as $Ph_2P^{\bullet}$) are unlikely, as the reaction is exceptionally clean and Ph_2PPPh_2 (the anticipated coupling product of two free $Ph_2P^{\bullet}$ radicals) is not detected. The reaction of $DmpPCl_2$ with two equivalents of $LiPPh_2$ (or $KPPh_2$) was investigated briefly as an independent means for generating $DmpP(PPh_2)_2$. This reaction gives multiple products, the major species actually being Ph_2PPPh_2.

3. Conclusions

The terminal phosphinidene complex [Cp_2Zr=PDmp(PMe_3)] (**1**) was prepared and found to react cleanly with Ph_2PCl to produce the sterically congested triphosphane DmpP(PPh_2)$_2$ (**2**). The structure of **2** obtained by single-crystal X-ray diffraction analysis confirms the sterically congested nature of the central phosphorus atom.

4. Experimental

4.1. General procedures

All reactions, unless stated separately, were performed in a Vacuum Atmospheres dry-box under dry N_2. All solvents were distilled from purple Na–benzophenone solutions. $DmpPH_2$ [6], [Li(Et_2O)][P(H)Dmp] [20], and [Cp_2Zr(Me)Cl] [27] were prepared as reported. ^{1}H-NMR spectra were recorded on Varian Gemini 300 or 200 MHz instruments and referenced using residual solvent proton signals. ^{31}P-NMR spectra were recorded on the Varian Gemini 300 MHz instrument operating at 121.47 MHz and referenced to external H_3PO_4 standard. Mass spectrometry was performed at the CWRU departmental facility. Elemental analyses were preformed by Oneida Research Services.

4.2. Synthesis of [Cp_2Zr=PDmp(PMe_3)] (1)

[Li(Et_2O)][P(H)Dmp] (1.40 g, 3.29 mmol) and [Cp_2Zr(Me)Cl] (0.87 g, 3.18 mmol) were dissolved in 20 ml of a 1.0 M toluene solution of PMe_3 (20.0 mmol) at room temperature. The solution turned orange–yellow immediately upon dissolution and progressively turned to a dark green solution over a period of 2 days. The solution was filtered and the volume of the filtrate was reduced in vacuo until substantial amounts of a dark green solid had precipitated. The solid was filtered off, washed with hexanes and dried in vacuo to afford 1.66 g of dark green **1a** (81%). Analytical quality samples were obtained by crystallization from hot toluene–hexanes (ca. 1:1) mixture. ^{1}H-NMR (C_6D_6): δ 7.15 (m, 3H), 6.82 (s, 4H), 5.43 (s, 5H), 5.42 (s, 5H), 2.43 (s, 12H), 2.23 (s, 6H), 0.41 (d, 9H, $J_{HP} = 7.0$ Hz). ^{31}P{^{1}H}-NMR (C_6D_6): δ 771.0 (d, $J_{PP} = 23$ Hz), -6.7 (d, $J_{PP} = 23$ Hz). Anal. Found: C, 69.44; H, 7.00. Calc. for $C_{37}H_{44}P_2Zr$: C, 69.23; H, 6.91%.

4.3. Synthesis of [$Cp_2Zr(CH_3)$\{P(H)Dmp\}]

To a mixture of [Li(Et_2O)][P(H)Dmp] (0.59 g, 1.38 mmol) and [$Cp_2Zr(CH_3)Cl$] (0.374 g, 1.375 mmol) was added a mixture of toluene (4 ml)–Et_2O (3 ml). The reaction mixture was stirred for 30 min and then filtered to afford 0.62 g of orange solid which was ascertained to be predominantly [$Cp_2Zr(CH_3)$\{P(H)Dmp\}] (ca. 95%) by ^{1}H- and ^{31}P-NMR spectroscopy (crude yield 77%). ^{1}H-NMR (C_6D_6): δ 7.07 (m, 1H), 6.99 (m, 2H), 6.94 (s, 4H), 5.49 (s, 10H), 4.37 (d, 1H, $J_{HP} = 255$ Hz), 2.28 (s, 12H), 2.24 (s, 6H), 0.44 (d, 3H, $^3J_{HP} = 8.3$ Hz). ^{31}P-NMR (C_6D_6): δ 33.3 (d, $J_{HP} = 255$ Hz).

4.4. Synthesis of DmpP(PPh_2)$_2$ (2)

To a stirred solution of **1** (0.12 g, 0.18 mmol) in 6 ml of benzene was added PPh_2Cl (66.0 µl, 0.37 mmol) via a microliter syringe. The reaction mixture gradually turned pale yellow. After 30 min, the reaction mixture was filtered through alumina plug and all volatiles were removed in vacuo, affording 0.131 g (95% yield based on ^{1}H-NMR vs internal *para*-dimethoxybenzene) of pale yellow DmpP(PPh_2)$_2$ (**2**). ^{1}H-NMR (C_6D_6): δ 7.04 (m, 9H), 6.93 (m, 2H), 6.83 (m, 16H), 2.31 (s, 6H), 2.15 (s, 12H). ^{31}P{^{1}H}-NMR (C_6D_6): δ -18.0 (d, $J_{PP} = 221$ Hz), -52.3 (t, $J_{PP} = 221$ Hz). HRMS (EI) *m/z* Found: 714.2741. Calc. for $C_{48}H_{45}P_3$: 714.2737. X-ray quality crystals were obtained after slow crystallization from hot THF–hexanes mixture (ca. 1:5) at −35°C.

4.5. X-ray crystallography for 2

The single-crystal X-ray diffraction experiment was performed on a Siemens P4/CCD diffractometer for **2**. Systematic absences and diffraction symmetry for **2** were consistent with the monoclinic space group $P2_1/n$. The structure was solved by direct methods, completed by subsequent difference Fourier syntheses and refined by full-matrix, least-squares procedures. All non-hydrogen atoms were refined with anisotropic coefficients and all hydrogen atoms were treated as idealized contributions.

All software and sources of the scattering factors were contained in the SHELXTL (5.03) program library (G. Sheldrick, Siemens XRD, Madison, WI).

5. Supplementary material

Crystallographic data for structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 158176 for **2**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

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