## Insertion of ClPPh<sub>2</sub> into a Ta-H bond: Synthesis and Structure of the First Phosphido Derivative of Tantalocene

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Summary:  $Ph_2PCl$  easily inserts into the Ta-H bond of  $Cp_2TaH_3$  affording an ionic complex  $[Cp_2TaH_2(HPPh_2)]$ -Cl (4). This species was deprotonated to give the first phosphido-substituted tantalocene complex  $Cp_2TaH_2$ - $PPh_2$  (5), which was characterized by NMR spectroscopy and X-ray crystallography. Thermal decomposition of 5 as well as its reaction with  $HPPh_2$  was studied.

Recently there has been an increased interest in phosphido-substituted metallocene chemistry.<sup>1-6</sup> The usual approaches to these species are metathesis<sup>1</sup> and oxidative addition of HPR<sub>2</sub>;<sup>2</sup> however, for the group 5 metallocenes these methods have yet shown little application due to the difficulties of isolation and low yields.<sup>3,4</sup> For the basic d<sup>2</sup> metallocenes Cp<sub>2</sub>MH(L) (M = Nb, Ta, L = two-electron donor; M = Mo, W, L = H) an alternative synthetic route was suggested,<sup>5</sup> leading to [Cp<sub>2</sub>ML(PHPh<sub>2</sub>)]X and supposed to be a nucleophilic substitution of halogen in XPR<sub>2</sub> by a nucleophilic metallocene center.<sup>5a</sup> Our recent investigation of the reaction of Cp<sub>2</sub>NbH<sub>3</sub> with ClPR<sub>2</sub> (Scheme 1) has shown

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(6) Nikonov, G. I.; Lemenovskii, D. A.; Lorberth, J. Organometallics 1994, 13, 3127. that direct insertion of a halogenophosphine into the Nb-H bond of  $Cp_2NbH_3$  is also possible, especially for complexes lacking metal-centered lone pairs.<sup>6</sup> Deprotonation of the insertion product  $[Cp_2NbH_2(PHPh_2)]Cl$  (1) yields, however, only the phosphine hydride complex  $Cp_2NbH(HPPh_2)$  (3), whereas the possible intermediate isomeric dihydro phosphide  $Cp_2NbH_2PPh_2$  (2) was not observed directly. The tantalum analogue of 2, i.e.  $Cp_2$ -TaH<sub>2</sub>PPh<sub>2</sub>, was mentioned earlier in literature; however, neither the synthesis nor characterization was provided.<sup>4</sup> Here we report the application of our insertion/deprotonation approach<sup>6</sup> to the synthesis of  $Cp_2$ -TaH<sub>2</sub>PPh<sub>2</sub>, together with its characterization and X-ray structure.

## **Results and Discussion**

Similar to the case of niobocene,  $ClPPh_2$  very smoothly inserts into the Ta-H bond of  $Cp_2TaH_3$  yielding the ionic compound  $(Cp_2TaH_2(HPPh_2))Cl$  (4) (eq 1). The

$$Cp_2TaH_3 + CIPPh_2 \xrightarrow{\text{toluene}} (Cp_2TaH_2(HPPh_2))CI \downarrow \xrightarrow{\text{NaOH} eq.} toluene 4$$
  
4  
 $Cp_2TaH_2PPh_2$  (1)

compound 4 immediately precipitates as white voluminous flakes after mixing toluene solutions of  $Cp_2TaH_3$ and  $ClPPh_2$  at ambient temperature. Even at low temperatures (up to -20 °C) the rate of the reaction is very high.

The structure of **4** was established by comparison of its IR and NMR data with those of its niobocene analogue 1. In the <sup>1</sup>H NMR spectrum of 4, the P-Hresonance appeared as a doublet of triplets at  $\delta$  7.09 ppm ( ${}^{1}J_{P-H} = 389 \text{ Hz}, {}^{3}J_{H-H} = 4.3 \text{ Hz}$ ). This signal does not diminish rapidly in intensity with time (days), indicating that 4 is much more resistant to proton exchange with the solvent (CD<sub>3</sub>OD), when compared to its niobium analogue 1 (hours).<sup>6</sup> However, deprotonation of 4 in aqueous sodium hydroxide (0.25 M) results in a quite different product compared to that found in the case of 1. Thus the dihydride phosphido complex  $Cp_2TaH_2PPh_2$  (5) was produced exclusively rather than the tantalocene analogue of 3. Complex 5 was isolated in high yield (90%) as pale yellow crystals after recrystallization from diethyl ether. Formation of pentavalent 5 versus trivalent 3 indicates an interesting isomerism existing for the complexes of HPPh<sub>2</sub>. An analogous

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Figure 1. Molecular structure of Cp<sub>2</sub>TaH<sub>2</sub>PPh<sub>2</sub>.

Table 1. Selected Bond Distances (Å) and Angles (deg) for Cp<sub>2</sub>TaH<sub>2</sub>PPh<sub>2</sub>

Ta-P(1)	2.595(3)	Ta-H(1)	1.70
Ta-H(2)	1.70	$Ta-X(1)^a$	2.058
Ta-X(2)	2.039	P(1) - C(21)	1.85(1)
P(1) - C(11)	1.85(1)		
P(1)-Ta-H(1)	54.0	P(1)-Ta-H(2)	53.5
H(1)-Ta-H(2)	102	C(11)-P(1)-C(21)	101.1(5)
Ta-P(1)-C(21)	108.8 (4)	Ta-P(1)-C(11)	113.9 (3)
$Cp(1)-Cp(2)^b$	137.3		

 $^{a}$  X(1) and X(2) are the gravity centers of the cyclopentadienyl rings.  $^{b}$  Cp(1)-Cp(2) denotes the angle between the normals to the cyclopentadienyl rings.

isomerism has recently been observed by Baker et al. for the pair Cp\*W(PMe<sub>3</sub>)(PPh<sub>2</sub>)<sub>2</sub>H and Cp\*Mo(PMe<sub>3</sub>)-(HPPh<sub>2</sub>)PPh<sub>2</sub>.<sup>7</sup> In our opinion, the stabilization of hydrido phosphide complexes for the 3rd row transition metals is mainly due to the higher metal-hydrogen bond energy observed for the third transition row as compared with the second.8

An X-ray diffraction study of 5 confirmed the above formulation. Thus 5 was found to be a trisubstituted tantalocene with a centrally positioned PPh<sub>2</sub> substituent and two lateral hydride ligands. Structurally analogous silicon<sup>9</sup> and tin<sup>10</sup> compounds have been previously studied; a related disilicon hydride tantalocene has been reported by Berry et al.<sup>11</sup> The molecular structure of 5 is shown in Figure 1, and selected bond distances and angles are given in Table 1. It is a common bent tantalocene substituted by a pyramidal PPh<sub>2</sub> ligand. The Ta-P bond distance is 2.595(3) Å, which is in perfect agreement with the calculated distance of 2.60



Å obtained by combining the tantalum radius in  $d^0$ tantalocene  $(1.50 \text{ Å})^9$  with the P radius of 1.10 Å.<sup>12</sup> This distance is somewhat longer than the Ta-P(phosphine)bond in Cp<sub>2</sub>TaSiMe<sub>3</sub>(PMe<sub>3</sub>) (2.516(4) Å)<sup>11</sup> and reflects the absence of any Ta-P multiple bond. In another structurally characterized phosphide derivative of tantalum, TaH(PPh<sub>2</sub>)<sub>2</sub>(dmpe)<sub>2</sub>,<sup>13</sup> having trigonal-planar phosphide ligands, the Ta-P(phosphide) bond distances are 2.430(6) and 2.404(6) Å, and the average bond order was assigned to be 1.5. Two hydrides were also located (but were not refined) at the distances of 1.700 Å, which are close to those found for Cp<sub>2</sub>TaH<sub>3</sub> (average 1.774 (3) Å).14

The compound 5 was proposed earlier to be an intermediate in the reaction between  $Cp_2TaH_3$  and HPPh<sub>2</sub> to give the diphosphene compound  $Cp_2TaH(\eta^2 -$ PPhPPh) (6).<sup>4</sup> According to these authors, 5 "is readily converted into 6 by heating with HPPh2". In this context it would be of interest to compare the thermal decomposition of the tantalum complex 5 and niobium complex 3. The latter was showed to lose easily dihydrogen giving an orthometalated complex 7, probably, via an intermediate complex 2', isomeric to 2.6



Wishing to obtain other possible intermediates on the way to 6, we have studied the thermolysis of 5. We have found that 5 does not decompose upon heating at 85 °C over 8 h (by <sup>1</sup>H and <sup>13</sup>C NMR). However, when 5 was heated at 100 °C in the presence of an equivalent amount of HPPh<sub>2</sub> for 3.5 h, very small but detectable amounts of another phosphorus derivative of tantalocene were found. Its <sup>1</sup>H, <sup>13</sup>C, and <sup>31</sup>P NMR data were identical with those of Cp<sub>2</sub>Ta(HPPh<sub>2</sub>)PPh<sub>2</sub> (8), which was also synthesized by another method;<sup>15</sup> the tantalum analogue of 7 was not found. Small amounts of Cp2- $TaH_3$  were also observed in the NMR spectra of the reaction mixture. This indirectly supports that elimination of dihydrogen from 5 proceeds via complex Cp<sub>2</sub>-TaH<sub>2</sub>PPh<sub>2</sub> with a lateral position of the PPh<sub>2</sub> group, which is analogous to 2' and isomeric to 5. This compound can be formed by subsequent reductive elimination/oxidative addition steps of HPPh<sub>2</sub> from 5, producing Cp2TaH as an intermediate. Trapping of the latter by  $H_2$  accounts for the formation of  $Cp_2TaH_3$ .

Complex 6 was, however, formed after refluxing 5 in toluene for 2 days in the presence of  $HPPh_2$  with a

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<sup>(15)</sup> Preparation of this compound as well as further development of the chemistry described here will be reported in a following full paper.



periodical removal of dihydrogen. The <sup>1</sup>H and <sup>31</sup>P NMR spectra of the reaction mixture revealed the presence of **8** and **6** in the ratio 3:7. Thus the reactions between Cp<sub>2</sub>TaH<sub>3</sub> and HPPh<sub>2</sub> and between **5** and HPPh<sub>2</sub> can be presented by Scheme 2. The search for other intermediates of this interesting transformation of Cp<sub>2</sub>TaH<sub>3</sub> to Cp<sub>2</sub>TaH( $\eta^2$ -PPhPPh) (**6**) continues.

## **Experimental Section**

All manipulations were carried out *in vacuo* using conventional Schlenk techniques. Solvents were dried over sodium or sodium benzophenone ketyl and distilled into the reaction vessel by high-vacuum gas phase transfer. ClPPh<sub>2</sub> was purchased from Merck. NMR spectra were recorded on a Varian VXR-400 spectrometer (<sup>1</sup>H, 400 MHz; <sup>13</sup>C, 100.4 MHz, <sup>31</sup>P, 162 MHz). IR spectra were obtained with an IKS-26 spectrometer. Elemental analysis was performed in the analytical laboratory of the Chemistry Department of Moscow University.

**Preparation of [Cp<sub>2</sub>TaH<sub>2</sub>(HPPh<sub>2</sub>)]Cl (4).** Excess of ClPPh<sub>2</sub> (1 mL, 5.57 mmol) was added dropwise to 100 mL of toluene solution of Cp<sub>2</sub>TaH<sub>3</sub> (1.05 g, 3.35 mmol). A white voluminous precipitate was formed immediately. After filtration of the reaction solution, this powder was washed with an additional 10 mL of toluene and dried in vacuo. Yield: 1.30 g (2.61 mmol, 77.9%). IR (KBr):  $\nu_{P-H} = 2290 \text{ cm}^{-1}$ ,  $\nu_{Ta-H} = 1780 \text{ cm}^{-1}$ . <sup>1</sup>H NMR (methanol-d<sub>4</sub>):  $\delta$  7.70 (m, 4, Ph) and 7.58 (m, 6, Ph), 7.09 (dt, <sup>3</sup>J<sub>H-H</sub> = 4.3 Hz, <sup>1</sup>J<sub>H-P</sub> = 389 Hz, 1, P-H), 5.58 (d, J<sub>P-H</sub> = 0.9 Hz, 10, Cp), -0.60 (dd, <sup>3</sup>J<sub>H-H</sub> = 4.3 Hz, <sup>1</sup>J<sub>P-H</sub> = 71.9 Hz, 2, Ta-H). <sup>13</sup>C NMR: 134.00, 132.88 (d, <sup>1</sup>J<sub>C-P</sub> = 19 Hz), 130.81, 120.68 (Ph), 94.13 (s, Cp). <sup>31</sup>P{<sup>1</sup>H} NMR: 0.17 (s). Anal. Calcd for C<sub>22</sub>TaH<sub>23</sub>PCl: C, 49.41; H, 4.33. Found: C, 49.56; H, 3.97.

Preparation of Cp<sub>2</sub>TaH<sub>2</sub>PPh<sub>2</sub> (5). A 0.555 g (1.04 mmol) amount of 4 was suspended in 35 mL of toluene. A 15 mL volume of 0.25 M aqueous NaOH was added, and the mixture thus obtained was stirred for 1.5 h, until the precipitate was almost completely dissolved. The organic layer turned yellow. Then the toluene solution was decanted and all volatiles were removed in vacuo to give a light-orange oily substance. Recrystallization from  $Et_2O$  gives 5 as yellow crystals in 90.5% yield (0.939 mmol). IR (KBr):  $\nu_{Ta-H} = 1815 \text{ cm}^{-1}$ . <sup>1</sup>H NMR (benzene- $d_6$ ):  $\delta$  7.73 (t, 2, Ph), 7.15 (m, 4, Ph) and 6.95 (m, 4, Ph), 4.66 (s, 10,Cp), -0.10 (d,  $J_{P-H} = 56.9$  Hz, 2, Ta-H). <sup>13</sup>C NMR for 5: 153.48 (d,  ${}^{1}J_{C-P} = 25.3$  Hz), 134.4 (d,  ${}^{2}J_{C-P} =$ 15.2 Hz), 127.54 (d,  ${}^{3}J_{C-P} = 5.3$  Hz), 124.50 (s, Phs), 91.67 (d,  $J_{C-P} = 2.4 \text{ Hz}$ ). <sup>31</sup>P{<sup>1</sup>H} NMR: -48.91 (s). MS (EI): m/e 496  $(Cp_2TaPPh_2), 798 ((Cp_2Ta)_2(C_{10}H_8)PPh_2).$  Anal. Calcd for  $C_{22}$ -TaH<sub>22</sub>P: C, 53.02; H, 4.45. Found: C, 52.75; 4.30.

**Thermolysis of Cp<sub>2</sub>TaH<sub>2</sub>PPh<sub>2</sub>.** A toluene solution of **5** was heated to 100 °C; a light red color appeared, but no reaction product was detected by <sup>1</sup>H NMR. One equivalent of HPPh<sub>2</sub> was added, and the reaction mixture was heated at 100 °C for 3.5 h giving a red solution. NMR spectra indicated the presence of Cp<sub>2</sub>Ta(HPPh<sub>2</sub>)PPh<sub>2</sub>. Spectral data for **8** are as follows. <sup>1</sup>H NMR (benzene-d<sub>6</sub>):  $\delta$  7.60, 7.45, 7.32, 7.18 and 6.90 (m, 20, Phs), 7.34 (d, <sup>1</sup>J<sub>P-H</sub> = 340.1 Hz, 1, P-H), 4.30 (t, J<sub>P-H</sub> = 1.7 Hz, 10, Cp). <sup>13</sup>C NMR: 89.35 (s, Cp). <sup>31</sup>P{<sup>1</sup>H} NMR: -20.97 (d, <sup>2</sup>J<sub>P-P</sub> = 0.09 Hz, 1, PPh<sub>2</sub>), 32.09 (d, <sup>2</sup>J<sub>P-P</sub> = 15.3 Hz, 1, HPPh<sub>2</sub>).

X-ray Diffraction Study of 5. Crystals suitable for X-ray diffraction were grown from an ether solution of 5. Atomic coordinates are given in Table 2, and the crystal data and data collection parameters are summarized in Table 3. Unit cell



Table 2. Positional Parameters and U Values ( $\dot{A}^2$ )and Their Estimated Standard Deviations for $Cp_2TaH_2PPh_2$ 

atom	x	у	z	U(iso)/U(eq)
Ta	0.0004(1)	0.2079(1)	0.7000(1)	0.0213(1)
<b>P</b> (1)	0.0927(3)	0.2945(2)	0.5634(2)	0.0228(7)
C(1)	-0.189(2)	0.3139(9)	0.762(1)	0.045(4)
C(2)	0217(2)	0.2235(9)	0.7965(9)	0.044(4)
C(3)	-0.270(2)	0.1638(9)	0.725(1)	0.045(5)
C(4)	0280(2)	0.219(1)	0.660(1)	0.053(5)
C(5)	0227(2)	0.309(1)	0.675(1)	0.053(5)
C(6)	0.164(2)	0.0687(8)	0.6893(8)	0.035(4)
C(7)	0.264(1)	0.1501(9)	0.7036(6)	0.031(3)
C(8)	0.247(2)	0.1870(9)	0.7842(7)	0.033(4)
C(9)	0.122(2)	0.1334(9)	0.8211(9)	0.042(4)
C(10)	0.072(2)	0.0609(9)	0.7644(8)	0.040(4)
C(11)	0.237(1)	0.3942(7)	0.5837(6)	0.022(3)
C(12)	0.221(1)	0.4747(8)	0.5327(6)	0.024(3)
C(13)	0.323(1)	0.5536(8)	0.5439(7)	0.030(3)
C(14)	0.444(1)	0.5527(9)	0.6065(7)	0.032(3)
C(15)	0.462(1)	0.4730(8)	0.6573(8)	0.030(3)
C(16)	0.363(1)	0.3959(8)	0.6481(7)	0.032(3)
C(21)	0.221(1)	0.2119(8)	0.5020(6)	0.028(3)
C(22)	0.384(1)	0.2237(9)	0.4910(7)	0.029(3)
C(23)	0.475(2)	0.160(1)	0.4439(8)	0.037(4)
C(24)	0.398(2)	0.082(1)	0.4065(9)	0.046(4)
C(25)	0.237(2)	0.067(1)	0.4177(9)	0.051(5)
C(26)	0.145(2)	0.131(1)	0.4631(8)	0.038(4)
H(1)	0.073	0.321	0.695	
H(2)	-0.076	0.191	0.600	

 
 Table 3. Crystal Data and Parameters of the Crystallographic Data Collection

empirical	$C_{22}H_{22}PTa$	monochromator	graphite
color	vellow	abs coef $(cm^{-1})$	6 1 1
0001	needles	$\overline{F}(000)$	968.00
amust sustam	monoclinic	$t_{\rm comp}(\mathbf{V})$	179
ciyst system	monocimic	temp (K)	1/3
space group	$P2_{1}/c$ (No. 14)	$2\theta$ range (deg)	2-54
unit cell		scan mode	$\theta - 2\theta$
params		no. of rflns	3019
a (Å)	8.206 (4)	collcd	
b (Å)	13.970 (6)	no. of obs rflns	2725
c (Å)	15.73 (1)	$(F > 4.0 \ \sigma(F))$	
$\beta$ (deg)	91.34 (6)	weighting	$w^{-1} = [\sigma^2(F) +$
$V(Å^3)$	1803 (3)	scheme	$0.000256(F^2)]^{-1}$
Ζ	4	final R indices	$R = 0.0482, R_{\rm w} =$
radiation (Å)	$\lambda(Mo K_{\alpha}) =$	(obs data)	0.0499
	0.710 69	R indices	$R_{g} = 0.0678, R_{m} =$
diffrac-	Enraf-Nonius	(all data)	0.0678
tometer	CAD-4	goodness of fit	3.05

parameters were determined from least-squares refinement of a set of 25 centered reflections. Two reflections were measured every 2 h as an orientation and intensity check; significant decay of intensity was not observed. The data were corrected for Lorentz, polarization, and absorption (DIFABS<sup>16</sup>). The structure was solved by dirrect methods (SHELX-76<sup>17</sup>) and refined by full-matrix least-squares procedures (SHELX-86<sup>18</sup>) with anisotropic temperature factors for all the non-hydrogen atoms. Calculated idealized positions of all of the hydrogen atoms were included in further refinement. Two hydride hydrogens were found in the difference Fourier synthesis. In

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## Notes

further refinement, the non-hydride hydrogen atoms were refined using the "rider" scheme with fixed isotropic thermal parameters (U = 0.08 Å<sup>2</sup>), and the positions of hydrides were fixed.

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**Supporting Information Available:** Tables of thermal parameters for non-hydrogen atoms, hydrogen atom positional and thermal parameters, and bond distances and angles for **5** (4 pages). Ordering information is given on any current masthead page.

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