

# Synthesis and Complexation of the Metalloligand $\{(\eta^5\text{-C}_5\text{H}_5)[\eta^5\text{-C}_5\text{Me}_3\text{-1,2-(PPh}_2)_2\text{TiCl}_2\}$ (TiPHOS): The First Example of a 1,2-Bis(diphenylphosphanyl)titanocene Derivative

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The reaction of lithium 1,2-bis(diphenylphosphanyl)trimethylcyclopentadienide (**1**) with  $\text{CpTiCl}_3$  leads to the formation of the titanocene diphosphane  $\{(\eta^5\text{-C}_5\text{H}_5)[\eta^5\text{-C}_5\text{Me}_3\text{-1,2-(PPh}_2)_2\text{TiCl}_2\}$  (TiPHOS, **2**). This metalloligand reacts readily with  $(\text{NBD})\text{Cr}(\text{CO})_4$  and  $\text{W}(\text{CO})_5(\text{THF})$  to give, in both cases,

the bimetallic chelate complexes  $(\text{TiPHOS})\text{Cr}(\text{CO})_4$  (**3**) and  $(\text{TiPHOS})\text{W}(\text{CO})_4$  (**4**). The structure of **4** has been determined by X-ray diffraction. The synthesis of a new early-late heterobimetallic complex  $(\text{TiPHOS})\text{Rh}(\text{CO})\text{Cl}$  (**5**) is reported.

## Introduction

Chelating diphosphanes constitute one of the most versatile ligands in organometallic chemistry.<sup>[1]</sup> Within this field, diphosphanes built on a bent metallocene skeleton are of particular interest. Indeed, the structural flexibility of such structures allows the chemist to synthesize a wide range of ligands. Furthermore, coordination of the metalloligand to an electron-rich metal leads to early-late heterobimetallic complexes with a high potential for homogeneous catalysis.<sup>[2]</sup>

Three types of diphosphanes with a bent metallocene framework have already been described (Figure 1).<sup>[3–5]</sup> Both the phosphorous groups can be linked either to each Cp unit (types I, I') or directly to the metal (type II), or one to the Cp and one to the metal (type III).

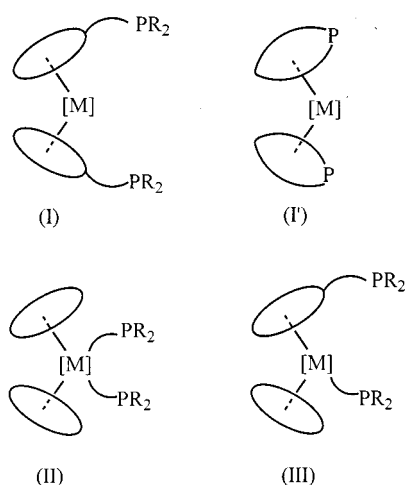


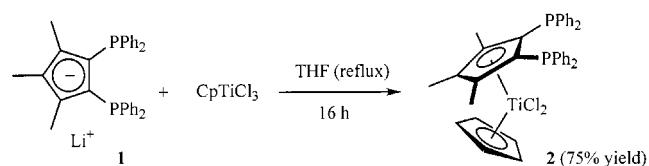
Figure 1. Diphosphanes with a bent metallocene framework

In this paper we describe the synthesis of a new titanocene dichloride metalloligand with the 1,2-bis(diphenylphosphanyl)trimethylcyclopentadienyl moiety. The 1,2-position of the phosphorus atoms on the cyclopentadienyl ring gives the ligand a rigid chelate structure often required for selective catalytic process. This diphosphane complex could, if necessary, be tuned by replacing the two chloride atoms on the titanium atom with other ligands. The synthesis and the characterisation of three new heterobimetallic complexes Ti–Cr, Ti–W and Ti–Rh are also reported.

## Results and Discussion

The ligand **1** was prepared according to an earlier method involving the synthesis of 1,2,3-trimethylcyclopenta-1,3-diene, followed by two repeated sequences of deprotonation-addition to  $\text{PPh}_2\text{Cl}$  and then deprotonation.<sup>[6]</sup>

The reaction of the lithium salt **1** with  $\text{CpTiCl}_3$  was carried out in THF at reflux. The reaction is complete after 16 h and leads to the targeted metalloligand **2** (TiPHOS) in good yield (Scheme 1). The  $^{31}\text{P}$  NMR spectrum of **2** shows a singlet at  $\delta = -21.8$  consistent with a diphosphane not coordinated to a metal atom. The  $^1\text{H}$  NMR spectra of **2** displays two singlets at  $\delta = 1.7$  and  $2.0$  for the  $\text{C}_5\text{Me}_3\text{-1,2-(PPh}_2)_2$  ligand assignable to the central and to both lateral methyl groups, respectively.

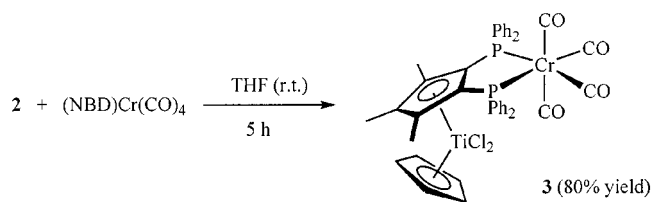


Scheme 1

In order to test the chelating properties of the TiPHOS ligand we coordinated it to the  $\text{Cr}(\text{CO})_4$  fragment. The metalloligand **2** readily displaces NBD from  $(\text{NBD})\text{Cr}(\text{CO})_4$  in THF at room temperature to give the

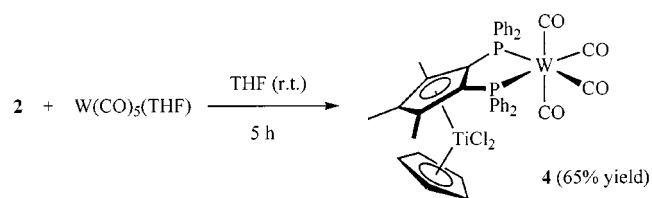
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bimetallic complex **3** (Scheme 2). This complex may be recrystallized from the same solvent. The infrared spectrum in the CO stretching region exhibits four carbonyl stretching bands at 2008, 1922, 1901 and 1881  $\text{cm}^{-1}$  consistent with a *cis*- $\text{L}_2\text{Cr}(\text{CO})_4$  complex. The  $^{31}\text{P}$  NMR spectrum of **3** displays only one singlet at  $\delta = 70.70$  corresponding to a downfield shift of about 93 ppm relative to the original bis(diphenylphosphane) **2**. The  $^1\text{H}$  NMR spectrum of **3** shows one singlet (intensity six) for both lateral methyl groups bonded to the cyclopentadienyl ring. The above data, closely similar to those obtained by Broussier et al. for a ferrocenic diphosphane,<sup>[7]</sup> are consistent with the symmetric chelate structure of **3** (Scheme 2).



Scheme 2

The easy formation of the bimetallic complex **3** prompted us to check the chelating abilities of TiPHOS with  $\text{W}^0$  pentacarbonyl complexes. The reaction of **2** with  $\text{W}(\text{CO})_5(\text{THF})$  in THF leads to the chelate structure **4** (Scheme 3). The  $^{31}\text{P}$  NMR spectrum of **4** shows a singlet at  $\delta = 33.60$  with  $^{183}\text{W}$  satellites. The  $^1\text{H}$  NMR and IR spectra of **4** are similar to those reported for **3** and confirm the structural features. It is worth noting that no trace of the  $-\text{W}(\text{CO})_5$  mono-coordinated intermediate derived from the chelating diphosphane **2** could be detected.



Scheme 3

Suitable crystals of **4** for X-ray measurements were obtained by crystallisation from warm THF. Its molecular structure is shown in Figure 2. The crystallographic study confirms its chelate structure and exhibits the usual tetrahedral (Ti) and distorted octahedral (W) geometries around the metallic fragments. The tungsten–phosphorus distances of 2.528(2) Å are similar to those observed for other tungsten–diphosphane structures (2.52–2.54 Å).<sup>[8]</sup> The P–W–P bite angle of 80.37(8)° corresponds well to that found in a *cis*- $\text{W}(\text{CO})_4$  complex with 1,2-bis(diphenylphosphanyl)ferrocene.<sup>[7]</sup>

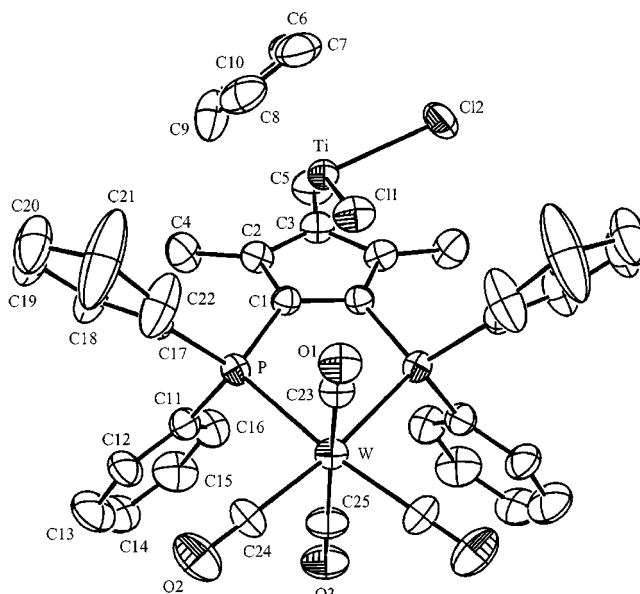
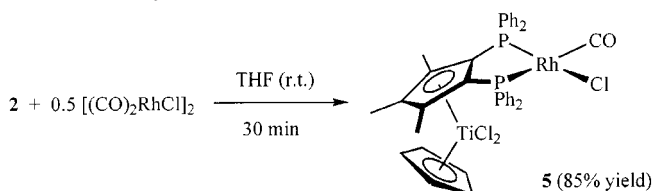


Figure 2. Molecular structure of complex **4**; for clarity the disordered atoms are not represented; selected bond lengths [Å] and angles [°]: Ti–Cp1 2.114, Ti–Cp2 2.072, Ti–C11 2.320(4), Ti–C12 2.364(9), W–P 2.528(2); P–W–P\* 80.37(6), C11–Ti–C12 94.0(3)

Once the chelating abilities of the metalloligand **2** had been revealed, we investigated its complexation with rhodium. Thus, the reaction with 0.5 molar equiv. of  $[(\text{CO})_2\text{RhCl}]_2$  gave the 1:1 adduct **5** which was isolated as a brown yellow powder (Scheme 4). The  $^{31}\text{P}$  NMR spectrum of **5** reveals a single set of eight signals with an AB pattern split by Rh–P coupling ( $^2J_{\text{P-P}} = 36$  Hz;  $^1J_{\text{P-Rh}} = 127$  Hz and 163 Hz) which is in accordance with the *cis*-coordination of both phosphorus atoms of TiPHOS to rhodium. The  $^1\text{H}$  NMR spectrum of **5** differs from that of the precursor phosphane **2** and the other bimetallic systems **3** and **4** in that it displays three methyl resonances instead of two. This anisochrony is indicative of the loss of the plane of symmetry. Finally, the IR spectrum exhibits a single carbonyl stretch at 1984  $\text{cm}^{-1}$  which corresponds to the terminal carbonyl band.



Scheme 4

## Conclusion

We have described the preparation of a new class of titanocene diphosphane complexes. This synthesis involves the preparation of the lithium salt  $[\text{C}_5\text{Me}_3\text{-1,2-(PPh}_2)_2]\text{Li}$  (**1**) followed by its treatment with  $\text{CpTiCl}_3$ . The chelating abilities of the metalloligand **2** (TiPHOS) has been revealed by its reaction with  $(\text{NBD})\text{Cr}(\text{CO})_4$  and  $\text{W}(\text{CO})_5(\text{THF})$  complexes. In both cases the heterobimetallic chelate complexes

have been obtained in good yields. Finally, the reaction of TiPHOS with the dimer  $[(\text{CO})_2\text{RhCl}]_2$  has led to the exclusive formation of a new early late Ti–Rh bimetallic complex. Further studies on bimetallic complexes containing the TiPHOS ligand and their catalytic activity are currently in progress.

## Experimental Section

All manipulations were carried out under an argon atmosphere using vacuum line techniques. Solvents were dried and distilled under argon before use.  $\text{CpTiCl}_3$ ,<sup>[9]</sup>  $(\text{NBD})\text{Cr}(\text{CO})_4$ ,<sup>[10]</sup>  $\text{W}(\text{CO})_5(\text{THF})$ <sup>[11]</sup> and  $[\text{C}_5\text{Me}_3\text{-1,2-(PPh}_2)_2\text{Li}]$ <sup>[6]</sup> (**1**) were prepared by literature methods. NMR spectra were recorded on a BRUKER AC200 (200 MHz for  $^1\text{H}$ , 81 MHz for  $^{31}\text{P}$ , 50 MHz for  $^{13}\text{C}$ ) spectrometer. IR spectra were obtained on a Bruker IFS 66v; abbreviations: m (medium), s (strong), vs (very strong). Elemental analyses were performed on a FISON EA 1108 within the laboratory.

**$\{(\eta^5\text{-C}_5\text{H}_5)[\eta^5\text{-C}_5\text{Me}_3\text{-1,2-(PPh}_2)_2\text{TiCl}_2\}$  (TiPHOS) (**2**):**  $[\text{C}_5\text{Me}_3\text{-1,2-(PPh}_2)_2\text{Li}]$  (0.81 g, 1.7 mmol) in 10 mL of THF was added to  $\text{CpTiCl}_3$  (0.37 g, 1.7 mmol) in 15 mL of THF. After stirring for 16 h at reflux, the solvent was removed in vacuo. The dark red residue was extracted with toluene and filtered through a piece of cotton. The solvent was removed in vacuo, the residue dissolved in  $\text{CH}_2\text{Cl}_2$  and precipitated by the addition of hexane. The mixture was then placed in a freezer ( $-20^\circ\text{C}$ ) for 1 h. The precipitate was collected by filtration and dried in vacuo (0.83 g, 75% yield). –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 1.75$  (s, 3 H,  $\text{CH}_3$ ), 1.97 (s, 6 H,  $\text{CH}_3$ ), 6.03 (s, 5 H,  $\text{C}_5\text{H}_5$ ), 6.95 (m, 10 H, Ph), 7.26 (m, 4 H, Ph), 7.49 (m, 6 H, Ph). –  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , partial):  $\delta = 12.9$  (s,  $\text{CH}_3$ ), 16.2 (s,  $\text{CH}_3$ ), 121.6 (s,  $\text{C}_5\text{H}_5$ ). –  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ ):  $\delta = -21.79$  (s,  $\text{PPh}_2$ ). –  $\text{C}_{37}\text{H}_{34}\text{Cl}_2\text{P}_2\text{Ti}$  (659.41): calcd. C 67.39, H 5.20; found C 67.68, H 5.02.

**(TiPHOS) $\text{Cr}(\text{CO})_4$  (**3**):** A 25 mL flask was charged with **2** (140 mg, 0.2 mmol),  $(\text{NBD})\text{Cr}(\text{CO})_4$  (54 mg, 0.2 mmol) and 10 mL of THF. The solution was stirred at room temperature for 5 h. The solvent was concentrated in vacuo until an abundant precipitate was formed. The brown precipitate was collected by filtration and dried in vacuo (0.13 g, 80% yield). – IR ( $\text{CHCl}_3$ ):  $\nu(\text{CO}) = 2008$  (m), 1922 (s), 1901 (vs), 1881  $\text{cm}^{-1}$  (s). –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 1.88$  (s, 3 H,  $\text{CH}_3$ ), 2.17 (s, 6 H,  $\text{CH}_3$ ), 5.87 (s, 5 H,  $\text{C}_5\text{H}_5$ ), 6.97 (m, 4 H, Ph), 7.29 (m, 6 H, Ph), 7.51 (m, 6 H, Ph), 8.14 (m, 4 H, Ph). –  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , partial):  $\delta = 13.7$  (s,  $\text{CH}_3$ ), 18.1 (s,  $\text{CH}_3$ ), 122.17 (s,  $\text{C}_5\text{H}_5$ ), 216.9 (triplet,  $^2J_{\text{CP}} = 10$  Hz, CO), 223.7 (pseudotriplet,  $^2J_{\text{CP}} = 13$  Hz, CO), 227.9 (triplet,  $^2J_{\text{CP}} = 8$  Hz, CO). –  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 70.70$  (s,  $\text{PPh}_2$ ). –  $\text{C}_{41}\text{H}_{34}\text{Cl}_2\text{CrO}_4\text{P}_2\text{Ti}$  (823.45): calcd. C 59.80, H 4.16; found C 59.29, H 3.96.

**(TiPHOS) $\text{W}(\text{CO})_4$  (**4**):** This compound was obtained following the above procedure but with  $\text{W}(\text{CO})_5(\text{THF})$  (0.12 g, 65% yield). – IR (KBr):  $\nu(\text{CO}) = 2102$  (m), 1905 (s), 1893 (vs), 1865  $\text{cm}^{-1}$  (s). –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 1.86$  (s, 3 H,  $\text{CH}_3$ ), 2.15 (s, 6 H,  $\text{CH}_3$ ), 5.86 (s, 5 H,  $\text{C}_5\text{H}_5$ ), 6.97 (m, 4 H, Ph), 7.28 (m, 6 H, Ph), 7.50 (m, 6 H, Ph), 8.10 (m, 4 H, Ph). –  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , partial):  $\delta = 14.0$  (s,  $\text{CH}_3$ ), 18.0 (s,  $\text{CH}_3$ ), 122.8 (s,  $\text{C}_5\text{H}_5$ ). –  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 33.59$  (s + d,  $^1J_{\text{W-P}} = 244$  Hz,  $\text{PPh}_2$ ). –  $\text{C}_{41}\text{H}_{34}\text{Cl}_2\text{O}_4\text{P}_2\text{TiW}$  (955.30): calcd. C 51.55, H 3.59; found C 51.89, H 4.09.

**(TiPHOS) $\text{Rh}(\text{CO})\text{Cl}$  (**5**):** A 25-mL flask was charged with **2** (42 mg, 0.06 mmol),  $[\text{RhCl}(\text{CO})_2]_2$  (36 mg, 0.03 mmol) and 4 mL

of THF. The mixture was stirred at room temperature for 30 min. during which time a brown yellow precipitate formed. The precipitate was collected by filtration and dried in vacuo (42 mg, 85% yield). – IR (KBr):  $\nu(\text{CO}) = 1984$   $\text{cm}^{-1}$  (vs). –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 1.95$  (s, 3 H,  $\text{CH}_3$ ), 2.09 (s, 3 H,  $\text{CH}_3$ ), 2.10 (s, 3 H,  $\text{CH}_3$ ), 5.93 (s, 5 H,  $\text{C}_5\text{H}_5$ ), 7.21 (m, 10 H, Ph), 7.48 (m, 6 H, Ph), 8.08 (m, 4 H, Ph). –  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , partial):  $\delta = 13.7$  (s,  $\text{CH}_3$ ), 16.8 (s,  $\text{CH}_3$ ), 17.1 (s,  $\text{CH}_3$ ), 122.0 (s,  $\text{C}_5\text{H}_5$ ). –  $^{31}\text{P}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 36.73$  (dd,  $^1J_{\text{Rh-P}} = 127$  Hz,  $^2J_{\text{P-P}} = 36$  Hz,  $\text{PPh}_2$ ), 56.00 (dd,  $^1J_{\text{Rh-P}} = 163$  Hz,  $^2J_{\text{P-P}} = 36$  Hz,  $\text{PPh}_2$ ). –  $\text{C}_{38}\text{H}_{34}\text{Cl}_3\text{OP}_2\text{RhTi}$  (825.78): calcd. C 55.27, H 4.15; found C 54.70, H 4.05.

**X-ray Crystallographic Study of **4**:**  $\text{C}_{41}\text{H}_{31}\text{Cl}_2\text{O}_4\text{P}_2\text{TiW}\cdot 4\text{C}_4\text{H}_8\text{O}$ ,  $M = 1240.66$ , orthorhombic, space group  $Pn2_1m$  (No. 31),  $a = 9.2830(2)$  Å,  $b = 16.6440(4)$  Å,  $c = 19.7850(6)$  Å,  $V = 3056.9(1)$  Å<sup>3</sup>,  $Z = 2$ ,  $\rho_{\text{calcd.}} = 1.348$  g·cm<sup>-3</sup>,  $F(000) = 1258$ ,  $\mu = 2.198$  mm<sup>-1</sup>,  $T = 110(2)$  K, 4720 independent reflections, 4123 observed reflections [ $I \geq 2\sigma(I)$ ], 304 refined parameters, 10 restraints,  $R(F) = 0.0576$ ,  $wR(F^2) = 0.1359$  for all data and  $R(F) = 0.0486$ ,  $wR(F^2) = 0.1276$ ,  $S = 1.073$  for observed reflections. A red crystal of **4** having the approximate dimensions  $0.5 \times 0.2 \times 0.2$  mm<sup>3</sup> and suitable for an X-ray analysis was sealed in a glass capillary in an inert oil and mounted on a Nonius KappaCCD diffractometer with graphite-monochromated Mo- $K_\alpha$  radiation ( $\lambda = 0.71073$  Å). A total of 228 frames of data were collected at 110(2) K with an oscillation range of  $1.5^\circ/\text{frame}$  and an exposure time of 40 s/deg.<sup>[12]</sup> A total of 43869 reflections were indexed, integrated, and corrected for Lorentz and polarization effects using DENZO-SMN and SCALEPACK.<sup>[13]</sup> Data reduction yielded 4720 unique reflections of which 4123 had  $I > 2\sigma(I)$ . Refinement of the unit cell parameters gave  $a = 9.2830(2)$  Å,  $b = 16.6440(4)$  Å,  $c = 19.7850(6)$  Å and  $V = 3056.9(1)$  Å<sup>3</sup>. Systematic absences were consistent with the compound having crystallised in the orthorhombic space group  $Pn2_1m$  (No. 31).

The structure was solved by a Patterson search program<sup>[14]</sup> and refined by full-matrix least-squares on  $F^2$  using SHELXL.<sup>[14]</sup> All non-hydrogen atoms were refined with anisotropic thermal parameters, except for solvent atoms which were refined with a global isotropic temperature factor (the complex crystallised with four THF solvate molecules). The hydrogen atoms of the complex were included in their calculated positions and refined in a riding model with isotropic displacement coefficients (the hydrogen atoms of solvent molecules were not introduced in the model). The nonsubstituted cyclopentadienyl ring and the two chlorine atoms were found to be disordered. The residual peaks on the final difference Fourier map ranged from  $-0.82$  to  $0.88$  e·Å<sup>-3</sup>.

Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-147166. Copies of the data can be obtained free of charge on application to CCDC, 12 union Road, Cambridge CB2 1EZ, UK [Fax: (internat.) + 44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

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