

The synthesis of tungsten imido hydride complexes by the hydrogenolysis of dialkyl complexes

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Received 8 June 1999; accepted 9 August 1999

Abstract

The reaction of the complexes $[(\text{TMS})_2\text{pda}]\text{W}(=\text{NPh})(\text{R})_2$, ($\text{R} = \text{CH}_2\text{CMe}_3, \text{CH}_2\text{CH}_3$) ($[(\text{TMS})_2\text{pda}] = \{\text{o}-[\text{Me}_3\text{SiN}]_2\text{C}_6\text{H}_4\}^{2-}$) with dihydrogen in the presence of trialkyl phosphines results in the formation of a series of W(VI) hydride complexes of the general formula $[(\text{TMS})_2\text{pda}]\text{W}(=\text{NPh})(\text{H})_2(\text{PR}_3)_2$ ($\text{PR}_3 = \text{PMe}_3, \text{PMe}_2\text{Ph}, 1/2\text{Ph}_2\text{PCH}_2\text{CH}_2\text{PPh}_2, 1/2\text{P}(\text{C}_6\text{H}_{11})_3$). The reactions of these complexes with olefins are also described. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Tungsten; Imido; Hydride; Alkyl; Hydrogenolysis

1. Introduction

Recently, we have studied a series of tungsten and molybdenum complexes with the general formula $[(\text{TMS})_2\text{pda}]\text{M}(=\text{NPh})(\text{R})_2$ ($[(\text{TMS})_2\text{pda}] = \{\text{o}-[\text{Me}_3\text{SiN}]_2\text{C}_6\text{H}_4\}^{2-}$; $\text{M} = \text{W}, \text{Mo}$; $\text{R} = \text{alkyl, aryl}$) [1–5]. Although our initial studies were directed toward the synthesis of metal alkylidene complexes for use as olefin metathesis catalysts, the ease with which a wide variety of dialkyl complexes could be synthesized has led us to study the chemistry of these d^0 dialkyl complexes in more detail. We have found that isocyanides readily insert into the metal carbon bonds of these complexes [6] and that the W complexes with β -H containing alkyl groups are stable at and above room temperature (r.t.), despite their electronic and coordinative unsaturation [1].

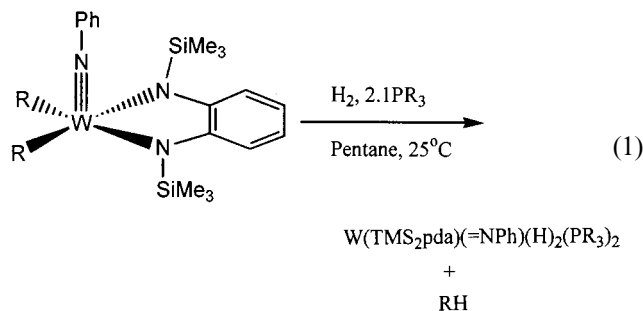
In this paper, we report the reaction of several tungsten dialkyl complexes with H_2 in the presence of trialkyl phosphines. These reactions result in the formation of a series of new W(VI) imido hydride complexes. Despite the fact that the hydride ligand is the simplest anionic ligand in coordination chemistry, there are relatively few examples of transition metal complexes that contain both the imido and hydride ligands [7–16]. The compounds reported herein afford us the opportunity to study the chemistry of the hydride ligand in the presence of the

tungsten imido group. We have found that these hydride complexes react rapidly with olefins to give the corresponding alkane and W(IV) olefin complexes, and they behave as homogeneous olefin hydrogenation catalysts.

2. Results and discussion

2.1. Synthesis

The reaction of the complex $[(\text{TMS})_2\text{pda}]\text{W}(=\text{NPh})(\text{CH}_2\text{CMe}_3)_2$ (**1**) with H_2 (ca. 2 atm) and two equivalents of trialkyl phosphine results in the formation of the seven-coordinate dihydride complexes, $[(\text{TMS})_2\text{pda}]\text{W}(=\text{NPh})(\text{H})_2(\text{PR}_3)_2$ (**2–4**), as shown in Eq. (1). In the case of PCy_3 , only one phosphine can coordinate to the metal for steric reasons and the product is $[(\text{TMS})_2\text{pda}]\text{W}(=\text{NPh})(\text{H})_2(\text{PCy}_3)$ (**5**).



$\text{R} = \text{CH}_2\text{CH}_3, \text{CH}_2\text{CMe}_3$

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Although single crystals of **2** have been obtained, their quality has not resulted in an X-ray diffraction study that is completely refined and therefore publishable. The results of the structural study do however indicate that the phenyl imido group and one amide from the TMS₂pda ligand are *trans* with respect to one another. The other silylamide group is *cis* to the NPh ligand as are the two PMe₃ ligands which are mutually *transoid*. A structure that is derived from a pentagonal bipyramid with the imido group occupying an axial position is consistent with these observations, as shown in Fig. 1.

The r.t. ¹H-NMR spectrum of **2** displays two peaks that are assigned to inequivalent Si(CH₃)₃ groups as well as a hydride resonance at 9.26 ppm that integrates to two protons. The PMe₃ protons appear as a broad singlet at 1.04 ppm. The inequivalence of the Me₃Si groups is consistent with a structure that has the NSiMe₃ groups occupying sites that are *cis* and *trans* to the imido group, while the broadness of the hydride and PMe₃ peaks suggests that the compound is fluxional at r.t.

The low-temperature NMR spectra are consistent with this structural assignment. At –50°C the hydride peak of **2** is a sharp doublet of doublets because the hydrides are the AA' part of an AA'XX' spin system (the ³¹P nuclei being the XX' part.). The PMe₃ protons resolve into a virtual triplet with a peak separation of 3 Hz. This is consistent with the pseudotrans disposition of the PMe₃ groups in the equatorial plane of the pentagonal bipyramid. The ³¹P{¹H} spectra of **2** display a singlet with tungsten satellites (¹J_{W-P} = 188 Hz) from 25 to –50°C.

The NMR spectral parameters of **3** are similar to those of **2** and are also consistent with the proposed structure. The difference is that **3** is more fluxional than **2**. At r.t., the hydride signal is observed as a broad singlet, and at –50°C, this resonance has only decoalesced into a broad triplet that is similar to the one observed for **2** at r.t. It is likely that the fluxionality in **2** and **3** involves dissociation of the PR₃ ligand and the larger steric demands of PMe₂Ph versus PMe₃ lead to a lower activation barrier for dissociation.

The NMR spectra of complex **4** are also consistent with a structure that is derived from a pentagonal bipyramid, as shown in Fig. 1. This structure has an axial phenylimido group with the TMS₂pda ligand amido groups occupying equatorial sites, while the phosphines of the DPPE group occupy one equatorial site and the axial site *trans* to the imido group. The hydrides are symmetrically disposed in the equatorial plane. This structure has a mirror plane that contains the phosphorus atoms of the DPPE group as well as the imido N atom. This mirror plane renders the SiMe₃ protons of the TMS₂pda ligand chemically equivalent as is observed in the proton NMR spectrum. The PPh₂ groups of the DPPE ligand are chemically inequivalent and are observed as a pair of doublets in the ³¹P{¹H}-NMR spectrum of **4**. The hydride ligands give rise to a four line pattern with ¹⁸³W satellites that is the A₂ part of an A₂XY spin system where X and Y are the ³¹P nuclei of the DPPE ligand.

The NMR spectra of **5** are consistent with a structure that is derived from an octahedron in which the PCy₃ group and the phenyl imido ligand are mutually *trans*. This leaves the hydride ligands in *cis* positions as proposed in Fig. 1. The proton NMR spectrum of **5** displays a doublet for the chemically equivalent hydride protons (²J_{P-H} = 83 Hz) and a single peak that is assigned to the Me₃Si protons that are equivalent due to the mirror plane that contains the N, W and P atoms. Furthermore, the value of J_{W-P} (56 Hz) is significantly smaller than that of **2** and **3** as would be expected given the strong *trans* influence of the W=N group.

2.2. Reactivity studies

Although our initial discovery of these complexes occurred when we were examining the interaction of complex **1** with H₂ in the presence of phosphines, a more efficient and economical synthesis involves the reaction of the complex [o-(Me₃SiN)₂C₆H₄]W(=NPh)(CH₂CH₃)₂ (**7**) with H₂ in the presence of the desired phosphine ligand. As part of this procedure, crude **7** was allowed to react with an excess of the phosphine in the presence of ca. 2 atm of

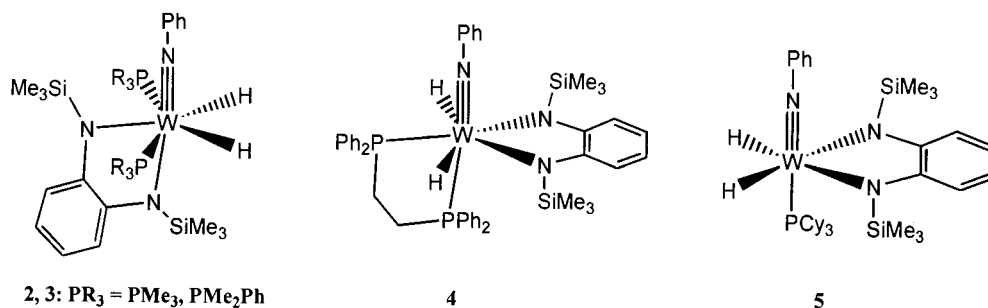
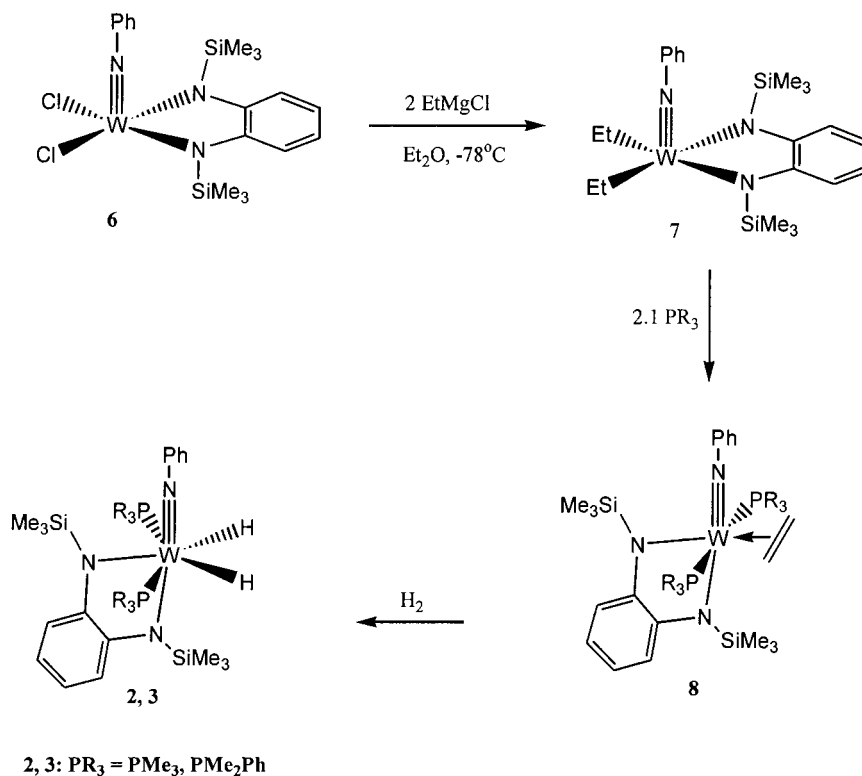


Fig. 1. Proposed structures of compounds **2**–**5**.



Scheme 1.

H₂ over the course of several hours at r.t. This reaction sequence is an efficient synthesis of **2** from [*o*-(Me₃SiN)₂C₆H₄]₂W(=NPh)(Cl)₂ (**6**). It appears that the reaction sequence proceeds as shown in Scheme 1. Addition of PR₃ to the diethyl complex rapidly induces β-H abstraction to generate the ethylene complex **8** [4]. Hydrogenation of **8** in the presence of PR₃ leads to the desired hydride products. We have confirmed that **8** reacts with H₂ under the same conditions as employed here to generate complex **2**.

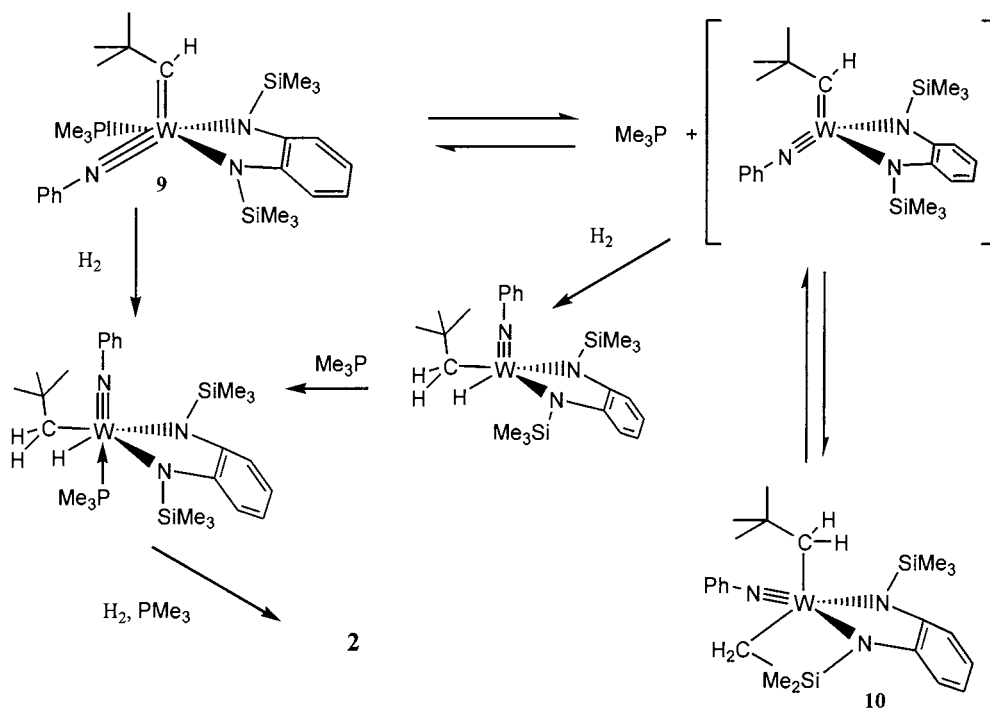
An alternative route to complex **2** involves hydrogenolysis of the alkylidene complex W(NPh)[*o*-(Me₃SiN)₂C₆H₄](CHCMe₃)(PMe₃) (**9**) in the presence of one equivalent of PMe₃. In this reaction, the alkylidene is completely hydrogenated to form neopentane and the metal ends up as **2**. Reactions that were performed in an NMR tube reveal essentially quantitative conversion of **9** to **2**. If the reaction was performed in the absence of added PMe₃, then a 50% yield of **2** was observed. The remaining W containing material is the metallated TMS₂pda complex **10** [3], shown in Scheme 2.

One possible mechanism for the conversion of **9** to **2** involves initial dissociation of PMe₃ from **9**. Hydrogenolysis of the intermediate base-free alkylidene complex would generate a base-free hydrido neopentyl complex that could be trapped by PMe₃ to give **2** by reductive elimination, followed by oxidative addition of H₂. If there is not enough PMe₃ present in the reaction mixture, then **10** is formed along with **2**. Complex **10**

does in fact react with H₂ much more slowly to give a material that has a peak in the hydride region of the ¹H-NMR spectrum that is converted to **2** when it is allowed to react with PMe₃. The direct hydrogenolysis of the alkylidene group of **9** could also lead to **2**.

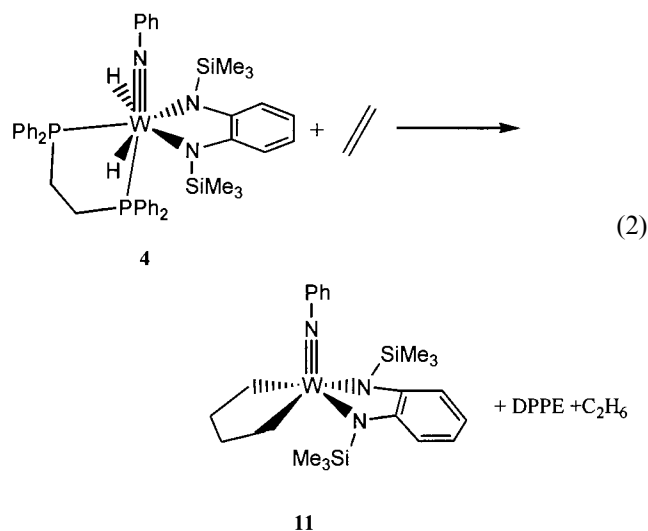
We have explored the reaction of various hydride sources with the dichloride complex **6** as an alternative route to these complexes. Unfortunately, the use of KH, LiAlH₄, LiBEt₃H, LiAl(O-*t*-Bu)₃H and NaBH₄ did not give satisfactory results. While hydride complexes were formed in all cases, there were significant quantities of impurities that proved extremely difficult to separate from the desired products. The most efficient synthesis of these phosphine hydride complexes appears to be generating the diethyl complex **7**, followed by its in situ hydrogenolysis in the presence of excess phosphine ligand.

When a solution of compound **2** was exposed to ethylene at r.t. and 1 atm pressure, the ethylene complex **8** was observed along with one equivalent of ethane. Compound **2** does function as an olefin hydrogenation catalyst though the turnover frequency is very low. Thus, styrene is hydrogenated to form ethylbenzene (TON ~ 3/h) and cyclohexadiene is converted to cyclohexene with 98% selectivity. We believe that the low turnover frequency and the inability to hydrogenate cyclohexene at an appreciable rate is due to the inhibitory effect of the PMe₃ and the formation of a stable cyclohexene complex, respectively. Both of these



factors could inhibit the oxidative addition of H_2 to the metal center and would thereby inhibit the hydrogenation reaction.

When complex **4** was exposed to excess ethylene, a rapid reaction occurred that produced ethane and the metallacyclopentane complex **11** [5] (Eq. (2)). We were surprised by the identity of the W complex formed in this reaction. Evidently, the DPPE ligand in **4** is displaced once the complex is exposed to excess ethylene. Presumably, the formation of a W(VI) ethylene complex occurs as it does with **2** and **3**, but in this case, the DPPE does not bind strongly enough to prevent the formation of the W(VI) metallacyclopentane complex **11**.



Indeed, when **2** is exposed to an excess of ethylene over the period of weeks at r.t., **11** is the final product of the reaction. In all cases, the driving force for the formation of W(VI) leads to the observed thermodynamic product, the W(VI) metallacyclopentane complex.

This work demonstrates the viability of W(VI) imido hydride complexes. One interesting aspect of the reactivity of these compounds is their ability to cycle between the W(IV) and W(VI) formal oxidation states. Unlike typical d^0 complexes that are olefin hydrogenation catalysts [17], the hydrogenation reaction appears to be proceeding via a mechanism that involves both oxidative addition and reductive elimination steps. It is possible that the imido group somehow facilitates the d^2/d^0 couple that is necessary for such reactions. We are actively investigating the oxidative addition and reductive elimination reactivities of this class of compounds.

3. Materials and experimental methods

All syntheses were carried out under a dry argon atmosphere using standard Schlenk techniques. The compounds $W(=NPh)Cl_4(OEt_2)$ (**6**) [18], $[o-(Me_3SiN)_2C_6H_4]W(=NPh)(CH_2CH_3)_2$ (**7**) [1], $W(NPh)[o-(Me_3SiN)_2C_6H_4](C_2H_4)(PMe_3)_2$ (**8**) [5], and $W(NPh)[o-(Me_3SiN)_2C_6H_4](CHCMe_3)(PMe_3)$ (**9**) [2], were synthesized according to literature procedures. Tetrahydrofuran (THF), diethyl ether (Et_2O), and pentane were distilled from sodium benzophenone ketyl. Benzene was distilled from sodium. NMR solvents were

stored over molecular sieves and degassed prior to use. NMR spectra were acquired on either Varian VXR 300 or Gemini 300 spectrometers. ^1H and ^{13}C chemical shifts are referenced to the residual proton peaks of the deuterated solvents and are reported relative to TMS. Elemental analyses were performed by Atlantic Micro-labs, Inc., or by the analytical services of this department.

3.1. $W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{H})_2(\text{PMe}_3)_2$ (**2**)

3.1.1. Method 1

In a glass tube with a Teflon Young's joint, $W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{CH}_2\text{C}(\text{CH}_3)_3)_2$ (**10**) (1.66 g, 2.48 mmol) was dissolved in 25 ml of hexanes. PMe_3 (0.64 ml, 6.20 mmol) was added via syringe. The solution was then placed in liquid nitrogen while a vacuum was applied. Once the solution was frozen solid under vacuum, the flask was sealed. The neck of the flask was then purged with hydrogen gas and the H_2 hose was wired securely to the flask. The flask was opened until the H_2 reached a pressure of 10 PSIG. The flask was then resealed and the H_2 line removed. The reaction was then allowed to warm to r.t. After 4 h of stirring, the color of the solution changed from brown to magenta, and magenta crystals precipitated from solution. The remaining solution was transferred to a Schlenk tube and cooled to -10°C to give additional magenta crystals. The mother liquors were concentrated and cooled to give another crop of crystals. Total yield of **2**: 1.48 g (88%). $^1\text{H-NMR}$ (25°C , C_6D_6): 0.79 (s, 9H, $-\text{Si}(\text{CH}_3)_3$); 0.81 (s, 9H, $-\text{Si}(\text{CH}_3)_3$); 1.04 (s, 18H, $-\text{PMe}_3$); 6.80–7.45 (m, 9H, aromatic); 9.26 (br t, 2H, W-H). $^{13}\text{C}\{^1\text{H}\}\text{-NMR}$ (C_6D_6 , 25°C): 4.92 ($\text{Si}(\text{CH}_3)_3$); 6.43 ($-\text{Si}(\text{CH}_3)_3$); 15.99 (s, $-\text{PMe}_3$); 115.21, 116.53, 118.17, 119.20, 123.31, 126.71, 128.68, 151.13 (aromatic). $^1\text{H-NMR}$ (-50°C , C_7D_8): 0.69 (s, 9H, $-\text{Si}(\text{CH}_3)_3$); 0.75 (s, 9H, $-\text{Si}(\text{CH}_3)_3$); 0.84 (t, separation = 3 Hz, 18H, $-\text{PMe}_3$); 6.74–7.38 (m, 9H, aromatic); 9.0 (d of d, 2H, $^2J_{\text{P-H}} = 37, 41$ Hz, W-H). $^{31}\text{P}\{^1\text{H}\}\text{-NMR}$ (C_6D_6 , 25°C): -24.46 (s, $^1J_{\text{W-P}} = 188$ Hz). Anal. Calc. for $\text{C}_{24}\text{H}_{47}\text{N}_3\text{P}_2\text{Si}_2\text{W}$: C, 42.41; H, 6.97; N, 6.18. Found: C, 42.18; H, 6.79; N, 6.03.

3.1.2. Method 2

$W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{CHCMe}_3)(\text{PMe}_3)$ (**9**) (50 mg, 0.07 mmol) was dissolved in C_6D_6 in an NMR tube fitted with a Teflon Young's joint. One equivalent of PMe_3 (7 μl , 0.07 mmol) was added via a microliter syringe. The NMR tube was then fitted with a Schlenk adapter, frozen in liquid nitrogen, and placed under vacuum. The tube was then sealed while frozen under vacuum. Hydrogen gas was then purged through the Schlenk adapter for 5 min. The Teflon seal was opened to allow the H_2 to fill the vacuum in the NMR tube. The NMR tube was charged with ca. 15 PSIG of H_2 .

Over a period of less than 2 h, complete conversion of **9** to **2** was observed by $^1\text{H-NMR}$.

3.1.3. Method 3

The compound $[(\text{TMS})_2\text{pda}]M(=\text{NPh})(\text{Et})_2$ (**7**) was generated by the addition of 2.1 equivalents of EtMgCl to a diethyl ether solution of $[(\text{TMS})_2\text{pda}]M(=\text{NPh})(\text{Cl})_2$ at -78°C . The solution was allowed to warm to r.t. and was stirred for 30 min. The Et_2O was removed under reduced pressure and the resultant residue was extracted with pentane (3×20 ml) and the combined extracts were then filtered into a tube with a Teflon valve. At this time, 2.1 equivalents of PMe_3 was added to the solution resulting in a change from red to purple. The reaction mixture was stirred for 1 h and then frozen in liquid N_2 and evacuated. The flask was then charged with H_2 (ca. 10–15 PSIG) as described above. The solution was allowed to warm to r.t. and was stirred for 24 h. The solution turned the characteristic magenta color of compound **2**. The crystals that deposited were collected and the mother liquors were cooled to -20°C to harvest further compound from the reaction. The yield of **2** using this procedure is ca. 80% based on the $[(\text{TMS})_2\text{pda}]W(=\text{NPh})(\text{Cl})_2$ starting material. The identity of **2** was confirmed by $^1\text{H-NMR}$ spectroscopy.

3.2. $W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{H})_2(\text{PMe}_2\text{Ph})_2$ (**3**)

This compound was synthesized according to the procedure used for **2** by substituting PMe_2Ph for PMe_3 and following either Method 1 or Method 3, as described above. The yield was ca. 85%. $^1\text{H-NMR}$ (25°C , C_6D_6): 0.53 (s, 18H, $-\text{Si}(\text{CH}_3)_3$); 1.22 (s, 12H, $-\text{PMe}_2\text{Ph}$); 6.72–7.29 (m, 9H, aromatic); 9.63 (br s 2H, W-H). $^1\text{H-NMR}$ (-50°C , C_7D_8): 0.53 (s, 9H, $-\text{Si}(\text{CH}_3)_3$); 0.59 (s, 9H, $-\text{Si}(\text{CH}_3)_3$); 1.19 (t, peak separation = 4 Hz, 12H, $-\text{PMe}_2\text{Ph}$); 6.72–7.29 (m, 9H, aromatic); 9.56 (br t, $^2J_{\text{P-H}} = 40$ Hz, 2H, W-H). $^{13}\text{C}\{^1\text{H}\}\text{-NMR}$ (C_6D_6 , 25°C): 4.92 ($\text{Si}(\text{CH}_3)_3$); 6.43 ($-\text{Si}(\text{CH}_3)_3$); 15.99 (s, $-\text{PMe}_3$); 115.21, 116.53, 118.17, 119.20, 123.31, 126.71, 128.68, 151.13 (aromatic). $^{31}\text{P-NMR}$ (C_6D_6 , 25°C): -17.4 (s, $^1J_{\text{W-P}} = 164$ Hz). Anal. Calc. for $\text{C}_{34}\text{H}_{51}\text{N}_3\text{P}_2\text{Si}_2\text{W}$: C, 50.81; H, 6.39; N, 5.22. Found: C, 50.52; H, 6.19; N, 5.03.

3.3. $W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{H})_2(\text{DPPE})$ (**4**)

In a glass tube with a Teflon Young's joint, $W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{CH}_2\text{C}(\text{CH}_3)_3)_2$ (**10**) (0.65 g, 0.98 mmol) and DPPE (0.39 g, 0.98 mmol) were dissolved in 30 ml of hexanes. The solution was then placed in liquid nitrogen while a vacuum was applied. H_2 gas was introduced as described for **2**. The reaction was then allowed to warm to r.t. After 8 h of stirring, the color of the solution had changed from brown to

purple. Purple solid had also precipitated from solution. The solution was transferred to a Schlenk tube, concentrated to 10 ml, and cooled to -10°C to give additional purple solid. Total yield: 0.71 g (78%). $^1\text{H-NMR}$ (C_6D_6 , 25°C): 0.49 (s, 18H, $-\text{Si}(\text{CH}_3)_3$); 2.23 (m, 2H, $\text{PCH}_2\text{CH}_2\text{P}$); 2.42 (m, 2H, $\text{PCH}_2\text{CH}_2\text{P}$); 6.72–7.69 (aromatic); 10.56 (m, 2H, W-H). $^{31}\text{P}\{^1\text{H}\}$ -NMR (C_6D_6 , 25°C): 30.43 (d, $^2J_{\text{P-P}} = 83$ Hz); -12.70 (d, $^2J_{\text{P-P}} = 83$ Hz). Anal. Calc. for $\text{C}_{44}\text{H}_{53}\text{N}_3\text{Si}_2\text{P}_2\text{W}$: C, 57.08; H, 5.77; N, 4.54. Found: C, 57.33; H, 5.84; N, 4.54.

3.4. $W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{H})_2(\text{PCy}_3)$ (**5**)

In a glass tube with a Teflon Young's joint, $W(\text{NPh})[o-(\text{Me}_3\text{SiN})_2\text{C}_6\text{H}_4](\text{CH}_2\text{C}(\text{CH}_3)_3)_2$ (**10**) (1.06 g, 1.58 mmol), and PCy_3 (0.476 g, 1.70 mmol) were dissolved in ca. 30 ml of pentane. The solution was then placed in liquid nitrogen while a vacuum was applied. Once the solution was frozen solid under vacuum, the flask was sealed. The neck of the flask was then purged with hydrogen gas and the H_2 hose was wired securely to the flask. The flask was opened until the H_2 reached a pressure of 10 PSIG. The flask was then resealed and the H_2 line removed. The reaction was then allowed to warm to r.t. After 12 h of stirring, the color of the solution changed from brown to red. The solution was transferred to a Schlenk tube and concentrated to ca. 10 ml and cooled to -10°C to give red crystals. The mother liquors were concentrated and cooled to give a second crop of crystals. Total yield: 1.04 g (82%). $^1\text{H-NMR}$ (C_6D_6 , 25°C): 0.81 (s, 18H, $-\text{Si}(\text{CH}_3)_3$); 1.04–1.93 (m, 33H, PC_6H_{11}); 6.81 (t, 1H, *p*-Ph H); 6.95 (m, 2H *pda* ring H); 6.99 (t, 2H *m*-Ph-H) 7.18 (d, 2H *o*-Ph-H); 7.47 (m, 2H, *pda* ring H); 11.35 (d, $^2J_{\text{P-H}} = 85$ Hz, $^1J_{\text{W-H}} = 64$ Hz, W-H). $^{31}\text{P}\{^1\text{H}\}$ -NMR (C_6D_6 , 25°C): 66.71 (s, $^1J_{\text{W-P}} = 56$ Hz). Anal. Calc. for $\text{C}_{36}\text{H}_{53}\text{N}_3\text{Si}_2\text{P}_2\text{W}$: C, 54.13; H, 6.69; N, 5.26. Found: C, 53.98; H, 6.64; N, 5.23.

Acknowledgements

We wish to acknowledge the National Science Foundation CHE-9523279 for support of this work.

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