

# Synthesis and Electrochemistry of a Bis- $\eta^2$ -Coordinated Tetrametallic Transition-Metal Complex. Crystal Structure of $[(\eta^5\text{-C}_5\text{H}_4\text{SiMe}_3)_2\text{Ti}(\text{C}\equiv\text{Cfc})_2]\text{Pd}(\text{PPh}_3)^\dagger$

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The reaction of  $[\text{Ti}](\text{C}\equiv\text{Cfc})_2$  (**1**;  $[\text{Ti}] = (\eta^5\text{-C}_5\text{H}_4\text{SiMe}_3)_2\text{Ti}$ ,  $\text{Fc} = (\eta^5\text{-C}_5\text{H}_4)\text{Fe}(\eta^5\text{-C}_5\text{H}_5)$ ) with  $\text{Pd}(\text{PPh}_3)_4$  leads to the formation of tetrametallic  $\{[\text{Ti}](\text{C}\equiv\text{Cfc})_2\}\text{Pd}(\text{PPh}_3)$  (**2**). The X-ray structure determination shows that both  $\text{C}\equiv\text{Cfc}$  units are  $\eta^2$ -coordinated to the Pd center, causing a planar  $\text{Ti}(\text{C}\equiv\text{C})_2\text{Pd}$  arrangement. The  $\text{PPh}_3$  ligand is located outside of this plane. Cyclic voltammetry studies of **2** reveal significant differences with respect to the electrochemical behavior of **1**.

## Introduction

The last decade has seen an increasing interest in the preparation of homo- and heterometallic complexes, due to their possible application in the design of materials with new and/or interesting bulk properties.<sup>1–3</sup> In this respect, mono- or bis(alkynyl) metallocenes with group IV metal atoms have attracted considerable interest.<sup>4</sup> Their use in the stabilization of low-valent transition-metal (TM) complex fragments in  $\{[\text{Ti}](\text{C}\equiv\text{CR})_2\}\text{ML}$  type compounds ( $[\text{Ti}] = (\eta^5\text{-C}_5\text{H}_4\text{SiMe}_3)_2\text{Ti}$ ,  $\text{ML} = 10\text{–}14\text{-valence-electron TM building block}$ ) was examined.<sup>4–9</sup> Also, a number of main-group-metal ions could be imbedded into a bis( $\eta^2$ -alkynyl) metallocene frame-

work.<sup>10</sup> Nevertheless, the mutual influences of the  $\eta^2$ -coordinated TM complex fragment and the corresponding metallocene unit on their individual redox properties have not been the focus of interest. To the best of our knowledge, only the redox behavior of  $\{[\text{Ti}](\text{C}\equiv\text{Cfc})_2\}\text{Ni}(\text{CO})$  has been reported ( $\text{Fc} = (\eta^5\text{-C}_5\text{H}_4)\text{Fe}(\eta^5\text{-C}_5\text{H}_5)$ ).<sup>11</sup> In that compound, a shift of the Ti(IV)/Ti(III) redox couple to a more negative potential was detected in comparison to the respective value of  $[\text{Ti}](\text{C}\equiv\text{Cfc})_2$  (**1**).<sup>11</sup> Of interest was the introduction of a heavier congener of Ni in order to study the electrochemical properties of  $\{[\text{Ti}](\text{C}\equiv\text{Cfc})_2\}\text{ML}$  complexes. In this respect, we here describe the preparation and properties of  $\{[\text{Ti}](\text{C}\equiv\text{Cfc})_2\}\text{Pd}(\text{PPh}_3)$ .

## Results and Discussion

$[\text{Ti}](\text{C}\equiv\text{Cfc})_2$  (**1**) was reacted with 1 equiv of  $\text{Pd}(\text{PPh}_3)_4$  in a 1:1 *n*-pentane/toluene mixture, producing heterotetranuclear  $\{[\text{Ti}](\text{C}\equiv\text{Cfc})_2\}\text{Pd}(\text{PPh}_3)$  (**2**) (eq 1). The reaction was accompanied by a color change from violet to brown.

The coordination of both Ti-bound acetylide ligands to the  $\text{Pd}(\text{PPh}_3)$  moiety is shown by the diagnostic shift of the  $\text{C}\equiv\text{C}$  stretching vibration in the IR spectrum from  $2056\text{ cm}^{-1}$  in **1** to  $1841\text{ cm}^{-1}$  in **2**.

In the  $^1\text{H}$  NMR spectrum of **2** the signals of the protons of the  $\text{SiMe}_3$  groups and the  $\text{C}_5\text{H}_4$  ligands of the Fc moieties are observed at virtually the same frequen-

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<sup>†</sup> Dedicated to Prof. Dr. Dietmar Seyferth on the occasion of his 70th birthday.

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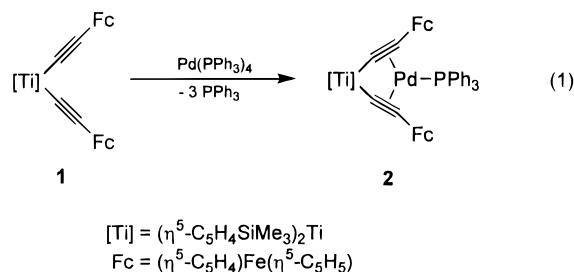
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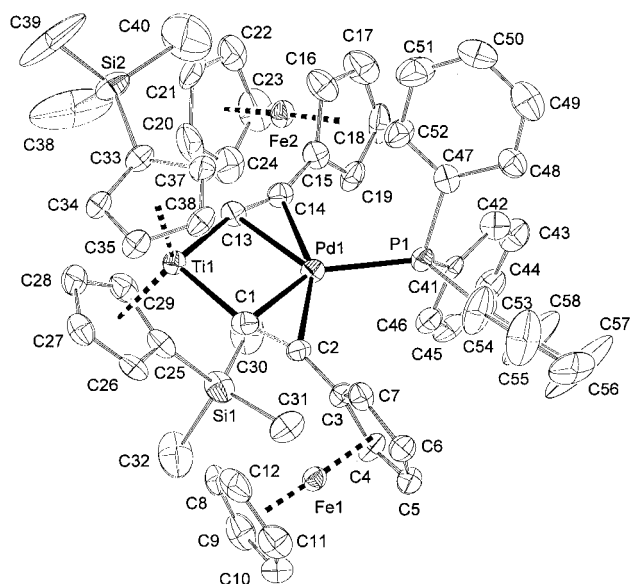
cies as reported for **1**.<sup>11</sup> In contrast, the signal of the C<sub>5</sub>H<sub>5</sub> protons at 4.03 ppm in **2** is shifted slightly to higher field in comparison to that of **1** (4.19 ppm). The respective resonance signals of the Ti-bound cyclopentadienyl ligands are found at 5.61 and 5.72 ppm, respectively, with an AA'XX' spin system ( $J_{\text{HH}} = 2.1$  Hz). In comparison to **1**, these resonance signals are shifted significantly to higher field (cf. **1**: 6.19, 6.69 ppm).<sup>11</sup>

The most informative features of the <sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **2** are the carbon resonance signals of the acetylenic units (Ti–C<sub>α</sub>≡C<sub>β</sub>). Due to the presence of a spin-active phosphorus nucleus in Pd(PPh<sub>3</sub>), the respective signal for C<sub>β</sub> is found as a doublet at 115.0 ppm (<sup>2</sup>J<sub>CP</sub> = 9.0 Hz), while for C<sub>α</sub> a singlet is observed at 179.0 ppm. Thus, in comparison to the respective signal set of **1**, the C<sub>β</sub> resonance frequency has shifted by 15 ppm to higher field and the C<sub>α</sub> signal is displaced by 25 ppm to lower field. The resonance signals of the cyclopentadienyl ligands of the titanocene as well as the ferrocenyl fragments are practically unaffected by the addition of the Pd(PPh<sub>3</sub>) moiety.<sup>11</sup>

The <sup>31</sup>P{<sup>1</sup>H} NMR spectrum of **2** contains the signal of the PPh<sub>3</sub> group at 36.8 ppm, which is typical for a Pd-bound phosphine ligand.<sup>7</sup>

The result of the X-ray structural determination of **2** is depicted in Figure 1. Table 1 lists selected bond lengths and angles, and Table 2 contains crystal and refinement data.

Heterometallic **2** crystallizes in the monoclinic space group *Cc* with four independent molecules per unit cell. Both FcC≡C units are η<sup>2</sup>-coordinated to a low-valent Pd(PPh<sub>3</sub>) entity. This leads to a planar arrangement for Ti(1)–C(1)–C(2)–C(13)–C(14)–Pd(1). Within this array, Pd(1) possess a somewhat distorted trigonal-planar environment. The P(1) atom is positioned by 0.4808(40) Å out of the Ti(C≡C)<sub>2</sub>Pd-containing plane. The Pd(1)–P(1) distance (2.3075(13) Å) mirrors that of other palladium–phosphorus distances,<sup>12</sup> e.g., in the complex [Ti](C≡CC≡CEt)<sub>2</sub>Pd(PPh<sub>3</sub>) (2.276(4) Å).<sup>7b</sup> The Ti–C<sub>C≡C</sub> σ-bond lengths (Ti(1)–C(1) = 2.092(5) Å, Ti(1)–C(13) = 2.084(5) Å) resemble separations known from, for example, the parent molecule **1** (2.104(10), 2.088(13) Å)<sup>11</sup> or other heterometallic tweezer-type molecules.<sup>4</sup> Compared with the C≡C triple-bond separations found for **1** (1.227(13), 1.21(2) Å),<sup>11</sup> the respective distances observed for **2** (C(1)–C(2) = 1.260(6) Å, C(13)–C(14) = 1.259(6) Å) are somewhat elongated. This is most common for η<sup>2</sup>-coordinated organic or organometallic alkynes and is consistent with the shift of the C≡C stretching vibration to lower wavenumbers in the IR spectrum (vide supra).<sup>4</sup> Due to the η<sup>2</sup>-coordination of both FcC≡C units to Pd(1), a typical *trans* deformation



**Figure 1.** ORTEP drawing (50% probability level) and atom-numbering scheme of **2**.

**Table 1. Important Interatomic Distances (Å) and Angles (deg) of Complex 2**

Distances			
Ti(1)–Pd(1)	2.9330(9)	C(14)–C(15)	1.459(6)
Ti(1)–C(1)	2.092(5)	Pd(1)–C(1)	2.182(5)
Ti(1)–C(13)	2.084(5)	Pd(1)–C(2)	2.276(5)
C(1)–C(2)	1.260(6)	Pd(1)–C(13)	2.203(4)
C(13)–C(14)	1.259(6)	Pd(1)–C(14)	2.311(4)
C(2)–C(3)	1.467(6)	Pd(1)–P(1)	2.3075(13)
Angles			
Ti(1)–Pd(1)–P(1)	169.54(6)	C(1)–C(2)–C(3)	157.3(5)
C(1)–Ti(1)–C(13)	96.42(18)	C(13)–C(14)–C(15)	158.7(5)
C(1)–Pd(1)–C(13)	90.47(18)	D(1)–Fe(1)–D(2) <sup>a</sup>	177.44(28)
C(2)–Pd(1)–C(14)	155.18(16)	D(3)–Fe(2)–D(4) <sup>a</sup>	175.74(50)
C(2)–C(1)–Ti(1)	164.3(4)	D(5)–Ti(1)–D(6) <sup>a</sup>	130.34(21)
C(14)–C(13)–Ti(1)	164.9(4)		
Torsion Angles			
Ti(1)–C(1)–C(2)–C(3)			–179.5(9)
Ti(1)–C(13)–C(14)–C(15)			–174.3(9)

<sup>a</sup> D(1)–D(6) = centroids of the cyclopentadienyl ligands.

of the Ti–C≡C–C<sub>Fc</sub> fragment is observed (Ti(1)–C(1)–C(2) = 164.3(4)°, C(1)–C(2)–C(3) = 157.3(5)°, Ti(1)–C(13)–C(14) = 164.9(4)°, C(13)–C(14)–C(15) = 158.7(5)°).<sup>4</sup> At 169.54(6)°, the angle Ti(1)–Pd(1)–P(1) deviates from linearity. The torsion angles Ti(1)–C(1)–C(2)–C(3) (–179.5(9)°) and Ti(1)–C(13)–C(14)–C(15) (–174.3(9)°) mirror the planarity of the Ti(C≡C)<sub>2</sub>Pd unit. In this context, it must be noted that the C(3)–C(4)–C(5)–C(6)–C(7) and C(15)–C(16)–C(17)–C(18)–C(19) planes are bent out of the Ti(C≡C)<sub>2</sub>Pd plane (C(3)–C(4)–C(5)–C(6)–C(7) = 67.09(12)°; C(15)–C(16)–C(17)–C(18)–C(19) = 60.61(17)°). Thus, both Fc groups are tilted to the same side of the Ti(C≡C)<sub>2</sub>Pd plane, pointing away from the PPh<sub>3</sub> ligand (Figure 2).

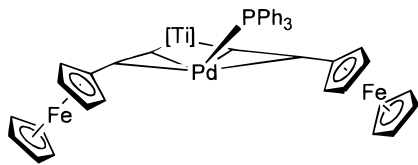
In contrast, in {[Ti](C≡CSiMe<sub>3</sub>)<sub>2</sub>}MR complexes (M = Cu, Ag, R = C<sub>6</sub>H<sub>2</sub>Ph<sub>3</sub>-2,4,6; M = Au, R = C<sub>6</sub>H<sub>2</sub>(CF<sub>3</sub>)<sub>3</sub>-2,4,6) the SiMe<sub>3</sub> groups are twisted above and below the Ti(C≡C)<sub>2</sub>M plane and the angles of the Ti–M–C<sub>ipso</sub> array are linear (M = Cu, 178.0(3)°; M = Ag, 178.41(9)°; M = Au, 179.4(2)°).<sup>13</sup> Most likely, this contrary behavior in **2** is due to the steric demand of the bulky PPh<sub>3</sub> ligand as well as the Fc groups. The color change observed in the course of the reaction of **1** with Pd-

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**Table 2. Crystal and Intensity Collection Data for Complex 2**

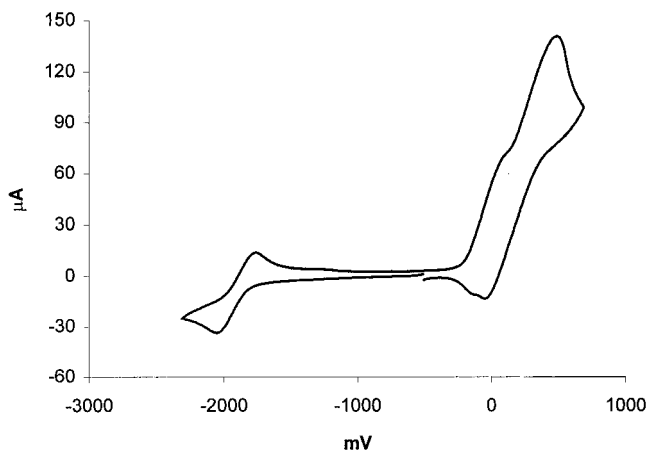
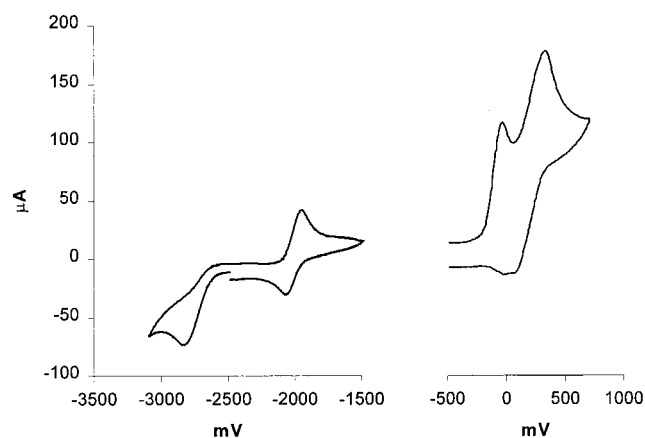
empirical formula	C <sub>58</sub> H <sub>59</sub> Fe <sub>2</sub> PPdSi <sub>2</sub> Ti
mol mass (amu)	1109.20
cryst syst	monoclinic
space group	Cc
a (Å)	21.4277(2)
b (Å)	22.6762(3)
c (Å)	11.8324(1)
V (Å <sup>3</sup> )	5692.51(10)
$\beta$ (deg)	98.063(1)
$\rho_{\text{calcd}}$ (g cm <sup>-3</sup> )	1.24(4)
F(000)	2280
Z	4
cryst dimens (mm)	0.2 × 0.2 × 0.15
diffractometer	Bruker SMART CCD
radiation ( $\lambda$ , Å)	Mo K $\alpha$ (0.710 73)
max and min transmissn	0.865, 0.628
abs coeff ( $\mu$ , mm <sup>-1</sup> )	1.0149
temp (K)	293(2)
scan mode	$\omega$ -scan
scan range (deg)	2.62 $\leq 2\theta \leq$ 60.28
index ranges	-22 $\leq h \leq$ 29 -30 $\leq k \leq$ 31 -11 $\leq l \leq$ 16
total no. of rflns	20 359
no. of unique rflns	10 107
no. of obsd rflns ( $I \geq 2\sigma(I)$ )	7781
no. of refined params	592
R1, <sup>a</sup> wR2 <sup>a,b</sup> ( $I > 2\sigma(I)$ )	0.0411, 0.0760
R1, <sup>a</sup> wR2 <sup>a,b</sup> (all data)	0.0607, 0.0829
S	1.036
max, min peak in final Fourier map (e Å <sup>-3</sup> )	0.438, -0.524

<sup>a</sup>  $R1 = \sum(|F_o| - |F_c|)/\sum|F_o|$ ;  $wR2 = [\sum(w(F_o^2 - F_c^2)^2)/\sum(wF_o^4)]^{1/2}$ .  
<sup>b</sup>  $w = 1/[\sigma^2(F_o^2) + (0.0261P)^2 + 0.0000P]$  with  $P = [F_o^2 + 2F_c^2]/3$   
<sup>c</sup>  $S = [\sum w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ ;  $n$  = number of reflections,  $p$  = parameters used.

**Figure 2.** Schematic representation of the Ti(C≡CFc)<sub>2</sub>Pd(PPh<sub>3</sub>) array.

(PPh<sub>3</sub>)<sub>4</sub> seemed to indicate a change in the electronic framework of the Ti-containing entity upon  $\eta^2$ -coordination of the Pd(PPh<sub>3</sub>) fragment. Therefore, complexes **1** and **2** have been studied with cyclic voltammetry in THF solutions in order to determine such an influence (Figures 3 and 4).

In THF, complex **1** generally exhibits electrochemical behavior similar to that reported with MeCN as solvent (Ti(IV)/Ti(III),  $E_{1/2} = -1.99$  V,  $\Delta E = 150$  mV; Fe(II)/Fe(III),  $E_{\text{ox}} = 0.00$  V; Fe(II)/Fe(III):  $E_{1/2} = 0.22$  V,  $\Delta E = 260$  mV).<sup>11,14</sup> This behavior persists even when the measurement is carried out at low temperature ( $-30$  °C). However, it has been established earlier that the electrochemically generated FcC≡CC≡CFc exhibits two well-resolved reversible one-electron oxidations in MeCN,<sup>11</sup> which is not the case in THF (Figure 3). In

**Figure 3.** Cyclic voltammogram of **1** in THF solution in the presence of [n-Bu<sub>4</sub>N][PF<sub>6</sub>] ( $c = 0.1$  mol dm<sup>-3</sup>) at 25 °C under N<sub>2</sub> (scan rate 100 mV s<sup>-1</sup>; potentials are referenced to FcH/FcH<sup>+</sup> as internal standard ( $E_{1/2} = 0.00$  V)).**Figure 4.** Cyclic voltammograms in the reductive (a, left) and oxidative regions (b, right) of **2** in THF solution in the presence of [n-Bu<sub>4</sub>N][PF<sub>6</sub>] ( $c = 0.1$  mol dm<sup>-3</sup>) at 25 °C under N<sub>2</sub> (scan rate 100 mV s<sup>-1</sup>; potentials are referenced to FcH/FcH<sup>+</sup> as internal standard ( $E_{1/2} = 0.00$  V)).

comparison, in the potential region from  $-500$  to  $1000$  mV the cyclic voltammogram of **2** displays synchronous irreversible one-electron oxidations of the two Fc entities at  $E_{\text{ox}} = -0.06$  V (Figure 4b). Thus, the electrochemical response of the Fc units connected to Ti(IV) and Pd(0) via an organic  $\pi$ -system is shifted to a more negative potential in comparison to **1**. This indicates a facilitation of the oxidation of Fe(II) to Fe(III). As in the case of **1**, the immediate Ti-C $\equiv$ C  $\sigma$ -bond cleavage takes place after oxidation of the Fc units.<sup>11,14</sup> The reversible electrochemical response of the all-carbon butadiynyl-bridged FcC≡CC≡CFc ( $E_{1/2} = 0.22$  V,  $\Delta E = 210$  mV) molecule can be observed at the same potential as in the case of **1**.

In contrast to the reversible Ti(IV)/Ti(III) reduction wave observed for **1** ( $E_{1/2} = -1.99$  V), the cyclic voltammogram of complex **2** exhibits an irreversible one-electron reduction ( $E_{\text{red}} = -2.72$  V; Figure 4a). The irreversibility of this wave has been demonstrated by multicyclic experiments in the region from  $-1500$  to  $-3000$  mV. In the course of these experiments a new and subsequently reversible wave at  $E_{1/2} = -1.99$  V ( $\Delta E = 120$  mV) appears, which can be assigned to the Ti(IV)/Ti(III) redox couple of **1**. This is indicative for the

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loss of the "Pd(PPh<sub>3</sub>)" building block initialized by the one-electron reduction of **2** and subsequent formation of **1**. In order to gain a deeper insight into this finding, the heterobimetallic complex {[Ti](C≡CSiMe<sub>3</sub>)<sub>2</sub>}Pd(PPh<sub>3</sub>)<sup>16</sup> has been postulated in an electrochemical study, which revealed that identical behavior is observed with an irreversible one-electron reduction process at  $E_{\text{red}} = -2.71$  V and development of a reversible wave at  $E_{1/2} = -1.95$  V ( $\Delta E = 100$  mV).

### Experimental Section

**General Methods.** All reactions were carried out under an atmosphere of nitrogen using standard Schlenk techniques. THF and Et<sub>2</sub>O were purified by distillation from sodium/benzophenone ketyl; *n*-pentane was purified by distillation from calcium hydride. Infrared spectra were obtained with a Perkin Elmer FT-IR Spectrum 1000 spectrometer. <sup>1</sup>H NMR spectra were recorded on a Bruker Avance 250 spectrometer operating at 250.130 MHz in the Fourier transform mode; <sup>13</sup>C-{<sup>1</sup>H} NMR spectra were recorded at 62.895 MHz. Chemical shifts are reported in  $\delta$  units (parts per million) downfield from tetramethylsilane with the solvent as the reference signal (CDCl<sub>3</sub>: <sup>1</sup>H NMR,  $\delta$  7.27; <sup>13</sup>C-{<sup>1</sup>H} NMR,  $\delta$  77.0). <sup>31</sup>P-{<sup>1</sup>H} NMR spectra were recorded at 101.255 MHz in CDCl<sub>3</sub> with P(OMe)<sub>3</sub> as external standard ( $\delta$  139.0, relative to H<sub>3</sub>PO<sub>4</sub> 85%). Melting points were determined on a Gallenkamp MFB 595 010 M melting point apparatus. Microanalyses were performed by the Department of Organic Chemistry, Technical University of Chemnitz. Cyclic voltammetry was carried out in a solution of [*n*-Bu<sub>4</sub>N][PF<sub>6</sub>] (0.1 mol dm<sup>-3</sup>) in THF at 25 °C on a Radiometer DEA 101 digital electrochemical analyzer, using a standard three electrode cell arrangement (Pt, Pt, SCE) and were referenced to the ferrocene/ferrocenium couple FcH/FcH<sup>+</sup> ( $E_{1/2} = 0.00$  V) as internal standard.

**General Remarks:** [Ti](C≡CFc)<sub>2</sub><sup>11</sup> and Pd(PPh<sub>3</sub>)<sub>4</sub><sup>15</sup> were prepared according to published procedures. All other chemicals were purchased from commercial providers and were used as received.

**Synthesis of {[Ti](C≡CFc)<sub>2</sub>}Pd(PPh<sub>3</sub>) (**2**).** Complex **1**<sup>11</sup> (150 mg, 0.2 mmol) was dissolved in a 1:1 *n*-pentane/toluene mixture (30 mL, 25 °C). Then, Pd(PPh<sub>3</sub>)<sub>4</sub> (240 mg, 0.2 mmol) was added in one portion. In the course of the reaction the

color changed from violet to brown. After the mixture was stirred for 10 h at 25 °C, all volatiles were evaporated in vacuo. Then, the brown residue was extracted with *n*-pentane (4 × 15 mL) and filtered through a pad of Celite. The filtrate was concentrated to 5 mL, and 5 mL of toluene was added. Cooling to -40 °C yielded **2** (150 mg, 70% based on **1**) as brown single crystals. Mp (°C): 104 dec. IR (KBr; cm<sup>-1</sup>): 1841 (s) ( $\nu_{\text{C}=\text{C}}$ ). <sup>1</sup>H NMR (CDCl<sub>3</sub>;  $\delta$ ): 0.37 (s, 18 H, SiMe<sub>3</sub>), 4.03 (s, 10 H, C<sub>5</sub>H<sub>5</sub>), 4.17 (pt,  $J_{\text{HH}} = 1.4$  Hz, 4 H, C<sub>5</sub>H<sub>4</sub>), 4.22 (pt,  $J_{\text{HH}} = 1.4$  Hz, 4 H, C<sub>5</sub>H<sub>4</sub>), 5.61 (pt,  $J_{\text{HH}} = 2.1$  Hz, 4 H, C<sub>5</sub>H<sub>4</sub>), 5.72 (pt,  $J_{\text{HH}} = 2.1$  Hz, 4 H, C<sub>5</sub>H<sub>4</sub>), 7.1–7.4 (m, 15 H, C<sub>6</sub>H<sub>5</sub>). <sup>13</sup>C-{<sup>1</sup>H} NMR (CDCl<sub>3</sub>;  $\delta$ ): 1.1 (SiMe<sub>3</sub>), 66.9 (CH/C<sub>5</sub>H<sub>4</sub>), 69.3 (C<sub>5</sub>H<sub>5</sub>), 70.5 (CH/C<sub>5</sub>H<sub>4</sub>), 71.6 (<sup>1</sup>C/C<sub>5</sub>H<sub>4</sub>), 107.0 (CH/C<sub>5</sub>H<sub>4</sub>), 110.7 (<sup>1</sup>C/C<sub>5</sub>H<sub>4</sub>), 111.7 (CH/C<sub>5</sub>H<sub>4</sub>), 115.0 (d,  $^2J_{\text{CP}} = 9.0$  Hz, TiC≡C), 128.5 (d,  $J_{\text{CP}} = 7.0$  Hz, CH/C<sub>6</sub>H<sub>5</sub>), 128.6 (CH/C<sub>6</sub>H<sub>5</sub>), 133.6 (d,  $J_{\text{CP}} = 19.0$  Hz, CH/C<sub>6</sub>H<sub>5</sub>), 137.2 (d,  $J_{\text{CP}} = 62.0$  Hz, <sup>1</sup>C/C<sub>6</sub>H<sub>5</sub>), 179.0 (TiC≡C). <sup>31</sup>P-{<sup>1</sup>H} NMR (CDCl<sub>3</sub>;  $\delta$ ): 36.8 (PPh<sub>3</sub>). Anal. Calcd for C<sub>58</sub>H<sub>59</sub>Fe<sub>2</sub>-PPdSi<sub>2</sub>Ti (1140.64): C, 61.07; H, 5.21. Found: C, 60.97; H, 5.29.

**X-ray Structure Determination of **2**.** The solid-state structure of **2** was determined from single-crystal X-ray diffraction. Data collection was performed on a Bruker SMART CCD diffractometer using Mo K $\alpha$  radiation. Crystallographic data of **2** are given in Table 2. The structure was solved by direct methods (SHELX 97 by G. M. Sheldrick, University of Göttingen, Göttingen, Germany, 1997). An empirical absorption correction was applied. The structure was refined by the least-squares method based on  $F^2$  with all reflections. All non-hydrogen atoms were refined anisotropically; the hydrogen atoms were placed in calculated positions. Compound **2** contains disordered *n*-pentane, for which no satisfactory model could be refined. The SQUEEZE<sup>17</sup> procedure from PLATON<sup>18</sup> was used to take this electron density into account.

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**Supporting Information Available:** Tables of crystal data collection and refinement details, positional and thermal parameters, and bond distances and angles and a figure giving an additional view of **2**. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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