

## Notes

# Thermal [2 + 2] Cycloadditions between the Metallodiphosphene ( $\eta^5\text{-C}_5\text{Me}_5$ )(CO)<sub>2</sub>Fe–P=P–Mes\* and a Phosphaalkyne<sup>†</sup>

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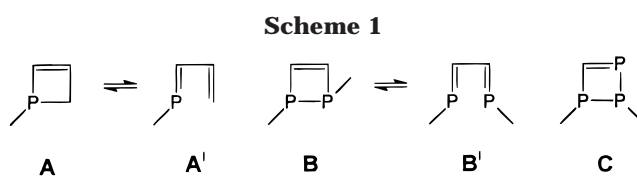
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**Summary:** The synthesis of *trans*-1,2-dihydro-1-(2,4,6-tri-*tert*-butylphenyl)-2-(dicarbonyl- $\eta^5$ -pentamethylcyclopentadienylferrio)-4-diisopropylamino-1,2,3-triphosphete (**9**) was conducted by treating 1-(dicarbonyl- $\eta^5$ -pentamethylcyclopentadienylferrio)-2-(2,4,6-tri-*tert*-butylphenyl)diphosphene (**7**) with an equimolar amount of 2-diisopropylamino-1-phosphaethyne (**2b**). Monitoring the reaction by <sup>31</sup>P{<sup>1</sup>H} NMR evidenced the formation of *trans*-1,2-dihydro-1-(dicarbonyl- $\eta^5$ -pentamethylcyclopentadienylferrio)-2-(2,4,6-tri-*tert*-butylphenyl)-4-diisopropylamino-1,2,3-triphosphete (**8**) as an intermediate, which in solution slowly isomerized to the final product **9**.

## Introduction

The chemistry of 1,2-dihydrophosphetes **A**<sup>2</sup> and 1,2-dihydrodiphosphetes **B**<sup>2</sup> is well-developed. These ring systems have attracted considerable interest as they can be regarded as masked 1-phosphabutadienes<sup>3</sup> and 1,4-diphosphabutadienes<sup>4</sup> via ring opening (Scheme 1). Accordingly, at elevated temperatures, these elusive heterobutadienes or their [W(CO)<sub>5</sub>] adducts were trapped by suitable dienophiles to give six-membered rings or alternatively coordinated as  $\eta^2$ -ligands to platinum centers via the P=C  $\pi$  bond.<sup>5</sup>

In contrast, information on 1,2-dihydro-1,2,3-triphosphetes **C** is scarce. The first representative was obtained as a [W(CO)<sub>5</sub>] complex **4** by the thermolysis of



2*H*-azaphosphirene **1** in the presence of *t*BuC≡P (**2a**) (Scheme 2).<sup>6</sup>

Recently, we described a more general access to metal-free 1,2-dihydro-1,2,3-triphosphetes **5** by reacting phosphaalkynes **2a–d** with (PCF<sub>3</sub>)<sub>4</sub> in THF at 25 °C (Scheme 3).<sup>7</sup> Heterocycle **5b** was also synthesized by treatment of *i*Pr<sub>2</sub>NC≡P (**2b**) with Me<sub>3</sub>P=PCF<sub>3</sub>.<sup>7</sup>

Invariably, the formation of all the 1,2-dihydro-1,2,3-triphosphetes has been rationalized by a stepwise addition of 2 equiv of a reactive phosphinidene (PCF<sub>3</sub>) or phosphinidene complex [(Me<sub>3</sub>Si)<sub>2</sub>CHP–W(CO)<sub>5</sub>] to the phosphaalkyne. Consequently, 2*H*-diphosphirenes are postulated as intermediates. Principally, it is also conceivable that 1,2-dihydro-1,2,3-triphosphetes are the result of a thermal [2 + 2] cycloaddition between reactive diphosphenes and suitable phosphaalkynes.<sup>8</sup>

Substantiation of this idea is the subject of the present report.

## Results and Discussion

Reaction of the metallodiphosphene ( $\eta^5\text{-C}_5\text{Me}_5$ )(CO)<sub>2</sub>Fe–P=P–Mes\* (Mes\* = 2,4,6-*t*Bu<sub>3</sub>C<sub>6</sub>H<sub>2</sub>) (**7**) with an equimolar amount of phosphaalkyne *i*Pr<sub>2</sub>NC≡P (**2b**) in *n*-pentane at temperatures slowly rising from –78 to +25 °C led to the formation of a dark red oil. This oil was dissolved in toluene-*d*<sub>8</sub>, and the subsequent reaction was monitored by <sup>31</sup>P{<sup>1</sup>H} NMR spectroscopy.

<sup>†</sup> Transition Metal Substituted Diphosphenes. 38.<sup>1a</sup> Reactive E=C-(p-p) $\pi$  Systems. 47.<sup>1b</sup>

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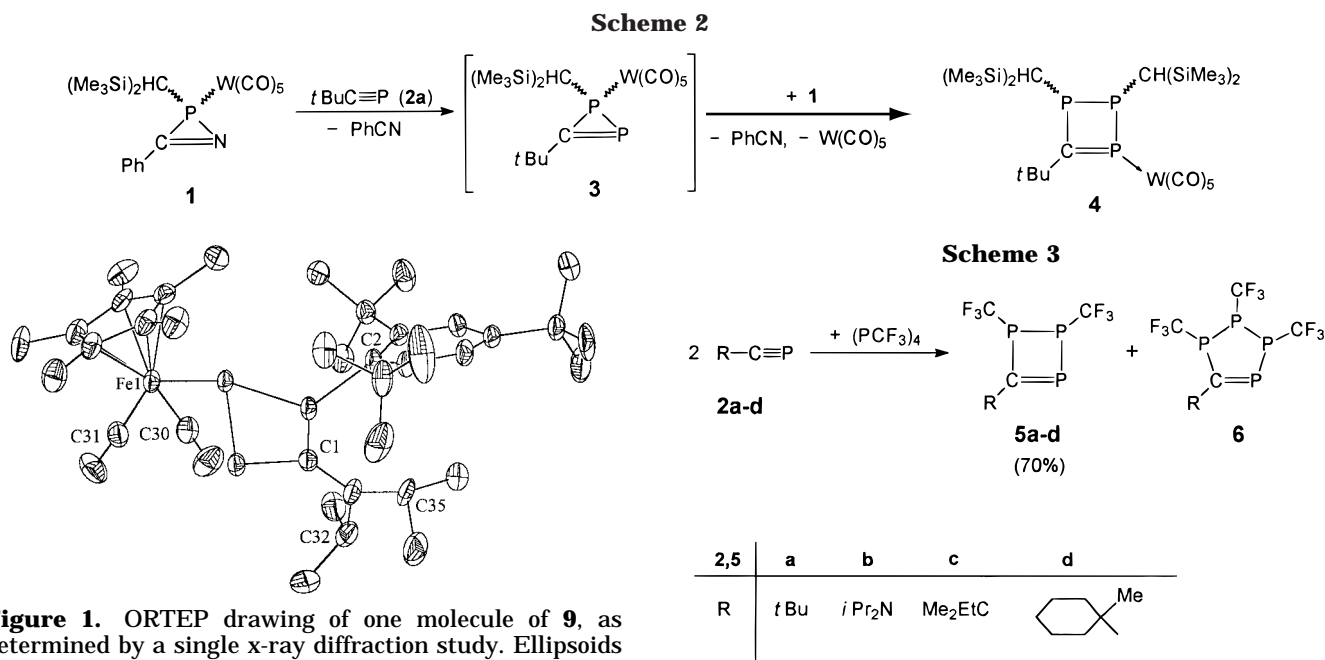
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**Figure 1.** ORTEP drawing of one molecule of **9**, as determined by a single x-ray diffraction study. Ellipsoids are scaled to enclose 60% of the electron density. Hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg): Fe(1)–P(3) 2.3220(13), P(1)–P(3) 2.190(2), P(2)–P(3) 2.239(2), P(1)–C(1) 1.749(3), P(2)–C(1) 1.816(3), P(2)–C(2) 1.891(3), C(1)–N(1) 1.352(3), C(32)–N(1) 1.475(4), C(35)–N(1) 1.486(4), Fe(1)–C(30) 1.750(3), Fe(1)–C(31) 1.761(3); Fe(1)–P(3)–P(1) 111.38(6), Fe(1)–P(3)–P(2) 114.03(5), P(1)–P(3)–P(2) 77.91(6), P(3)–P(2)–C(1) 84.98(11), P(3)–P(1)–C(1) 88.09(11), P(1)–C(1)–P(2) 102.67(15), P(1)–C(1)–N(1) 129.9(2), P(2)–C(1)–N(1) 127.0(2), C(32)–N(1)–C(35) 116.8(2), C(2)–P(2)–P(3) 122.15(11), C(1)–P(2)–C(2) 112.66(12).

Two sets of three doublets of doublets were attributed to compounds **8** and **9**, which after 2 h were present in a ratio of 3:1. During the course of the reaction, the resonance of **8** decreased in favor of the signals of **9**. After 3 days, the rearrangement of intermediate **8** to the final product **9** reached completion. The latter was isolated as red crystals in 95% yield.

The <sup>31</sup>P{<sup>1</sup>H} NMR spectrum of **9** displays a doublet of doublets at δ 90.7 for the dicoordinate atom P(1) with couplings to the metalated phosphorus P(3) of 260.6 Hz and to the arylated atom P(2) of 94.3 Hz. A doublet of doublets at δ 52.6 [<sup>1</sup>J<sub>P(3)P(1)</sub> = 260.4 Hz, <sup>1</sup>J<sub>P(3)P(2)</sub> = 249.3 Hz] is assigned to P(3). The doublet at higher field [δ –30.8 ppm, <sup>1</sup>J<sub>P(2)P(3)</sub> = 249.7 Hz, <sup>2</sup>J<sub>P(1)P(2)</sub> = 94.3 Hz] is due to the arylated phosphorus P(2). (The numbering is in accordance with Figure 1, however, not with the systematic nomenclature given in the summary).

In keeping with this, the isomeric 1,2-dihydro-1,2,3-triphosphete **8** features a doublet of doublets at δ 188.4 [<sup>1</sup>J<sub>P(1)P(2)</sub> = 241.2 Hz, <sup>2</sup>J<sub>P(1)P(3)</sub> = 50.3 Hz] for the dicoordinate phosphorus P(1). Doublets of doublets at δ 106.4 [<sup>1</sup>J<sub>P(2)P(3)</sub> = 163.5 Hz, <sup>2</sup>J<sub>P(3)P(1)</sub> = 50.3 Hz] and –52.2 [<sup>1</sup>J<sub>P(2)P(1)</sub> = 240.5 Hz, <sup>1</sup>J<sub>P(2)P(3)</sub> = 163.5 Hz] are attributed to the metal-functionalized phosphorus atom P(3) and the arylated atom P(2), respectively. In comparison with the final product **9** [<sup>1</sup>J<sub>P(2)P(3)</sub> = 249.7 Hz], the <sup>1</sup>J coupling in the former diphosphene unit of **8** is significantly smaller (163.5 Hz). The high-field shift of P(1) in **9** (δ 90.7) as compared to δ(P1) in **8** (δ 188.4) may be the result of an effective π-conjugation of the

lone pair of electrons at the planar nitrogen atom N(1) with the adjacent C=P bond. It is conceivable that an effective π-conjugation in **8** is prevented for steric reasons.

In a separate experiment, <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR data of the intermediate **8** were collected. These data mainly resemble those of **9** and underline the structural similarity of **8** and **9**. It is obvious, however, that the resonance of the dicoordinate carbon atom in **8** (δ = 208.0) is significantly deshielded when compared with the respective signal in **9** (δ = 203.1). Moreover, the CH resonance of the isopropyl groups in **8** (δ = 3.37 m) occurs at higher field than in product **9** (δ = 3.90 m). These observations may be rationalized by the magnetic influence of the aryl ring onto the CNR<sub>2</sub> function in **9**.

The IR spectrum of **9** shows two strong bands at ν = 1991 and 1943 cm<sup>-1</sup> in the ν(CO) region. In precursor **7**, the corresponding bands were observed at ν = 2005 and 1956 cm<sup>-1</sup>, indicating that the diphosphene ligand in **7** has a lower σ-donor/π-acceptor capacity than the heterocyclic ligand in **9**.

The topology of product **9** was ascertained by a single-crystal X-ray diffraction study (Figure 1, Table 1). Dark red single crystals were grown from a *n*-pentane solution at –30 °C. The analysis shows the presence of a 1,2-dihydro-1,2,3-triphosphete ring linked to the metal through a Fe–P single bond [2.322(1) Å]. Usually, Fe–P bond lengths in low-valent carbonyliron complexes fall in the range of 2.12–2.39 Å.<sup>11</sup> The ligand can be regarded as one leg in a distorted three-legged piano-stool arrangement. The three angles the ligands form with the metal center are C(30)–Fe(1)–C(31) 93.8(2)°, P(3)–Fe(1)–C(30) 91.64(11)°, and P(3)–Fe(1)–C(31) 88.94(10)°. Two legs of the piano stool are represented by terminal carbonyl groups. The most interesting feature of **9** is the geometry of the 1,2-

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Scheme 4

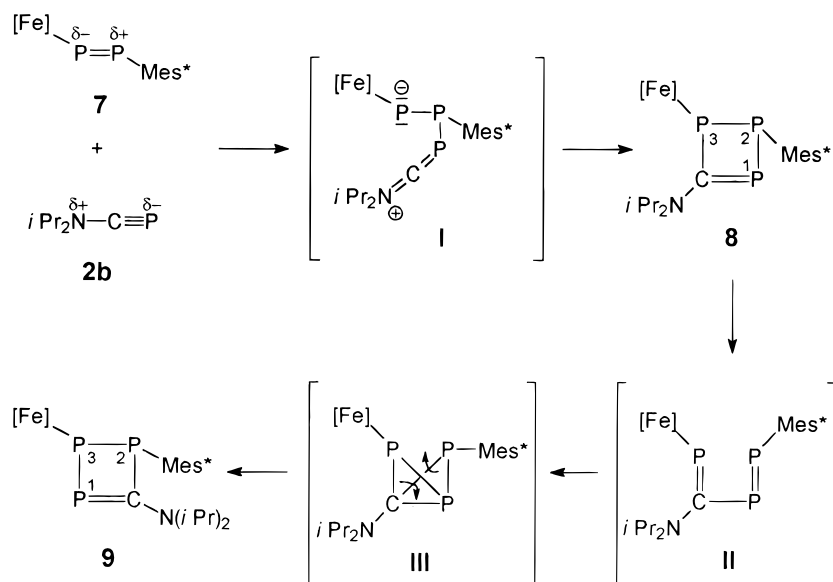


Table 1. Crystallographic Data

|   |   |
|---|---|
| formula   | C <sub>37</sub> H <sub>58</sub> FeNO <sub>2</sub> P <sub>3</sub> ·0.5C <sub>5</sub> H <sub>12</sub> |
| space group   | <i>P</i> 1  |
| data collection temp (K)                                | 173   |
| <i>a</i> (Å)  | 12.693(13)  |
| <i>b</i> (Å)  | 13.732(9)   |
| <i>c</i> (Å)  | 14.522(10)  |
| α (deg)   | 104.63(5)   |
| β (deg)   | 115.27(6)   |
| γ (deg)   | 103.40(6)   |
| <i>V</i> (Å <sup>3</sup> )                              | 2035(3)   |
| <i>Z</i>  | 2   |
| <i>d</i> <sub>calcd</sub> (g cm <sup>-3</sup> )         | 1.198   |
| μ (mm <sup>-1</sup> )                                   | 0.521   |
| max 2θ  | 54.00   |
| no. of reflns measd                                     | 9171  |
| no. of indep reflns                                     | 8771  |
| no. of reflns obsd [ <i>I</i> > 2σ( <i>I</i> )]         | 6125  |
| no. of params refined                                   | 417   |
| <i>R</i> <sub>value</sub> [ <i>I</i> > 2σ( <i>I</i> )]  | 0.0486  |
| <i>R</i> <sub>value</sub> (all data)                    | 0.0752  |
| GOF   | 0.941   |
| residual electron density (max/min) (e/Å <sup>3</sup> ) | 0.688 and -0.558  |

dihydro-1,2,3-triphosphete ligand, which is attached to the iron atom via the tricoordinate atom P(3). The P=P bond of metallo-diphosphene **7** [2.027(3) Å] is lengthened to 2.239(2) Å [P(2)–P(3)], in **9**. This P–P single bond is markedly longer than P(1)–P(3) [2.190(2) Å], which can be rationalized by the decreased covalent radius of a dicoordinate phosphorus atom. In **5b**, however, both P–P bonds are of identical length [2.201(2) and 2.204(2) Å]. The P=C bond [1.749(3) Å] falls in the typical range encountered for C-amino-functionalized PC double bonds [1.70–1.76 Å],<sup>12</sup> indicating a π-conjugation with the nitrogen lone pair. In keeping with this, atom N(1) is trigonal planar (sum of angles = 359.2°) and the atomic distance C(1)–N(1) [1.352(3) Å] is shorter than the standard value of a C<sub>sp<sup>2</sup></sub>–N<sub>sp<sup>2</sup></sub> single bond (1.45 Å). The endocyclic angle at the planar carbon atom C(1) [sum of angles 359.6°; P(1)–C(1)–P(2)

= 102.68°] is comparable with the situation in **5b** [104.69(8)°]. The angle P(1)–P(3)–P(2) of 77.91(6)° at P(3) in a trans-annular position to C(1) is the most acute endocyclic angle within the ring. The four-membered ring is slightly puckered, featuring dihedral angles φ<sub>1</sub> = 152.37° or φ<sub>2</sub> = 153.90° between the planes defined by the atoms P(1)–P(3)–P(2) and P(1)–C(1)–P(2) or the atoms P(3)–P(1)–C(1) and P(3)–P(2)–C(1). As in precursor **7**, the [Cp\*(CO)<sub>2</sub>Fe] unit and the supermesityl substituent remain in a trans-disposition at the ring.

For the formation of **9** from **7** and **2b**, the following mechanism is conceivable. The thermal [2 + 2] cycloaddition between **7** and **2b** proceeds charge-controlled and in two steps via adduct **I**. The interesting rearrangement **8** → **9** is initiated by an electrocyclic ring opening to give transient 1,2,4-triphosphabutadiene **II**. Similar processes are described by Mathey et al.<sup>3,4</sup> and by Weber et al.<sup>13</sup> with 1,2-dihydrophosphetes and 1,2-dihydrodi-phosphetes. To date, only one heavily substituted 1,2,4-triphosphabutadiene has been described in the literature [Mes\*–P=P–C(OSiMe<sub>3</sub>)=P–Mes\*].<sup>14</sup> In line with the reactivity of sterically less-congested 1,2,3-triphosphabutadienes,<sup>15</sup> cyclization to the bicyclic phosphabutane is obvious. Ring opening eventually afforded product **9** (Scheme 4).

## Experimental Section

All reactions were performed under nitrogen; the solvents were purified, dried, and degassed by standard techniques. <sup>1</sup>H, <sup>13</sup>C, and <sup>31</sup>P NMR spectra were recorded on a Bruker AC 200F spectrometer [200.13 (<sup>1</sup>H), 50.32 (<sup>13</sup>C), and 81.01 (<sup>31</sup>P) MHz] and a Varian Unity 600 instrument [150 MHz (<sup>13</sup>C)]. All chemical shifts are reported in ppm downfield from internal TMS (<sup>1</sup>H and <sup>13</sup>C) and external 85% H<sub>3</sub>PO<sub>4</sub> (<sup>31</sup>P). Mass spectra (EI) were obtained at 70 eV on a Varian MAT CH5 instrument. The compounds *i*Pr<sub>2</sub>NC≡P (**2b**)<sup>9</sup> and (η<sup>5</sup>-C<sub>5</sub>Me<sub>5</sub>)(CO)<sub>2</sub>Fe–P=Mes\* (**7**)<sup>10</sup> were prepared according to the literature.

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1-(2',4',6'-tri-*tert*-butylphenyl)-2-[dicarbonyl( $\eta^5$ -pentamethylcyclopentadienyl)ferrio]-4-(diisopropylamino)-1,2-dihydro-1,2,3-triphosphete (**9**). A glass ampule with two smash valves, magnetic stirring bar, and side-fixed NMR tube was equipped with the solution of ferriodiphosphene (**7**) (0.180 g, 0.32 mmol) in 5 mL of *n*-pentane. A sample of *i*Pr<sub>2</sub>NC≡P (0.050 g, 0.34 mmol) was condensed onto this solution at -196 °C. The stirred reaction mixture was allowed to warm from -78 °C to ambient temperature in a period of 2 h. The dark red solution was freed from *n*-pentane in vacuo. The dark red residue was dissolved in 0.5 mL of toluene-*d*<sub>8</sub>, and the solution was transferred to the NMR tube fixed to the reaction vessel. The tube was melted off, and the course of the reaction was monitored by <sup>31</sup>P NMR spectroscopy at room temperature. After 2 h, compounds **8** and **9** were detected in a molar ratio of 3:1. Under these conditions, complex **8** was slowly converted into **9**. After 24 h, the ratio of **8**:**9** decreased to 1:3, whereas after 3 d, only product **9** can be observed in the spectrum. Triphosphete **9** was isolated by crystallization from *n*-pentane at -30 °C (0.220 g, 95% yield). IR (*n*-pentane solution): 1991 s [ $\nu$ (CO)], 1943 s cm<sup>-1</sup> [ $\nu$ (CO)]. <sup>1</sup>H NMR (toluene-*d*<sub>8</sub>):  $\delta$  0.9 [d, <sup>3</sup>J<sub>HH</sub> = 6.5 Hz, 3H, CH(CH<sub>3</sub>)], 1.0 [d, <sup>3</sup>J<sub>HH</sub> = 6.5 Hz, 3H, CH(CH<sub>3</sub>)], 1.1 [d, <sup>3</sup>J<sub>HH</sub> = 6.5 Hz, 6H, CH(CH<sub>3</sub>)], 1.28 (s, 15H, C<sub>5</sub>Me<sub>5</sub>), 1.68 [s, 18H, *o*-C(CH<sub>3</sub>)<sub>3</sub>], 1.85 [s, 9H, *p*-C(CH<sub>3</sub>)<sub>3</sub>], 3.90 [m, 2H, CH(CH<sub>3</sub>)], 7.66 (s, 2H, *m*-arylH). <sup>13</sup>C{<sup>1</sup>H} NMR (toluene-*d*<sub>8</sub>):  $\delta$  9.6 [m, C<sub>5</sub>(CH<sub>3</sub>)<sub>5</sub>], 19.5–21.5 [m, CH(CH<sub>3</sub>)<sub>2</sub>], 31.4 [s, *p*-C(CH<sub>3</sub>)<sub>3</sub>], 34.3 [s, *o*-C(CH<sub>3</sub>)<sub>3</sub>], 34.6 [s, *o*-C(CH<sub>3</sub>)<sub>3</sub>], 36.6 [s, *p*-C(CH<sub>3</sub>)<sub>3</sub>], 39.7 [s, *o*-C(CH<sub>3</sub>)<sub>3</sub>], 52.5 [d, <sup>3</sup>J<sub>PC</sub> = 4.7 Hz, CH(CH<sub>3</sub>)<sub>2</sub>], 95.8 [s, C<sub>5</sub>(CH<sub>3</sub>)<sub>5</sub>], 139.1 (ddd, <sup>1</sup>J<sub>PC</sub> = 109.4 Hz, <sup>2</sup>J<sub>CP</sub> = <sup>3</sup>J<sub>CP</sub> ≈ 5 Hz, *i*-aryl-C), 158.0 (s) and 158.6 (s, *o*-aryl-C), 203.1 (ddd, <sup>1</sup>J<sub>CP(1)}</sub> = 76.6 Hz, <sup>1</sup>J<sub>CP(2)}</sub> = 57.8 Hz, <sup>2</sup>J<sub>CP(3)}</sub> = 14.0 Hz, C=P), 217.4 (m, CO). <sup>31</sup>P{<sup>1</sup>H} NMR (toluene-*d*<sub>8</sub>):  $\delta$  90.7 [dd, <sup>1</sup>J<sub>P(1)P(3)}</sub> = 260.6 Hz, <sup>2</sup>J<sub>P(1)P(2)}</sub> = 94.3 Hz, P(1)], 52.6 [dd, <sup>1</sup>J<sub>P(3)P(1)}</sub> = 260.4 Hz, <sup>1</sup>J<sub>P(3)P(2)}</sub> = 249.3 Hz, P(3)], -30.8 [dd, <sup>1</sup>J<sub>P(2)P(3)}</sub> = 249.7 Hz, <sup>2</sup>J<sub>P(2)P(1)}</sub> = 94.3 Hz, P(2)]. (The numbering is in accord with the result of the X-ray structure analysis depicted in Figure 1). MS: *m/z* (relative intensity) 697 (M<sup>+</sup>, 0.05), 669 (M<sup>+</sup> - CO, 0.1), 641 (M<sup>+</sup> - 2CO, 0.15), 554 (7<sup>+</sup>, 0.2), 450 [M<sup>+</sup> - Fe(CO)<sub>2</sub>Cp\*, 0.5], 424 (M<sup>+</sup> - CO - Mes\*, 1), 278 [Cp\*(CO)<sub>2</sub>-

FeP<sup>+</sup>, 18], 277 [Cp\*(CO)<sub>2</sub>FeP - H<sup>+</sup>, 53], 231 (Mes<sup>++</sup> - CH<sub>3</sub>, 26), 86 (C<sub>5</sub>H<sub>12</sub>N<sup>+</sup>, 21.5), 57 (*t*Bu<sup>+</sup>, 100), 44 (*i*PrH<sup>+</sup>, 50), 43 (*i*Pr<sup>+</sup>, 31), 28 (CO<sup>+</sup>, 59). Due to inclusions of varying amounts of *n*-pentane, no reliable C,H analyses were obtained.

The intermediate **8** was characterized by its <sup>1</sup>H, <sup>13</sup>C{<sup>1</sup>H}, and <sup>31</sup>P{<sup>1</sup>H} NMR spectra in toluene-*d*<sub>8</sub>. <sup>1</sup>H NMR:  $\delta$  0.87 [d, <sup>3</sup>J<sub>HH</sub> = 6.8 Hz, 6H, CH(CH<sub>3</sub>)], 0.88 [d, <sup>3</sup>J<sub>HH</sub> = 6.8 Hz, 6H, CH(CH<sub>3</sub>)], 1.31 (s, 15H, C<sub>5</sub>Me<sub>5</sub>), 1.41 [s, 18H, *o*-C(CH<sub>3</sub>)<sub>3</sub>], 1.88 [s, 9H, *p*-C(CH<sub>3</sub>)<sub>3</sub>], 3.37 [m, 2H, CH(CH<sub>3</sub>)], 7.43 (s, 2H, *m*-aryl-H). <sup>13</sup>C{<sup>1</sup>H} NMR:  $\delta$  9.0 [m, C<sub>5</sub>(CH<sub>3</sub>)<sub>5</sub>], 19.1–21.4 [m, CH(CH<sub>3</sub>)<sub>2</sub>], 31.1 [s, *p*-C(CH<sub>3</sub>)<sub>3</sub>], 34.1 [s, *o*-C(CH<sub>3</sub>)<sub>3</sub>], 38.4 [s, *p*-C(CH<sub>3</sub>)<sub>3</sub>], 39.4 [s, *o*-C(CH<sub>3</sub>)<sub>3</sub>], 51.4 [s, CH(CH<sub>3</sub>)<sub>2</sub>], 95.5 [s, C<sub>5</sub>(CH<sub>3</sub>)<sub>5</sub>], 139.1 (d, <sup>1</sup>J<sub>PC</sub> = 102.8 Hz, *i*-aryl-C), 147.2 (s), 156.8 (s), and 156.9 (s, aryl-C), 208.0 (ddd, <sup>1</sup>J<sub>CP(1)}</sub> = 75.1 Hz, <sup>1</sup>J<sub>CP(3)}</sub> = 56.8 Hz, <sup>2</sup>J<sub>CP(2)}</sub> = 14.0 Hz, C=P), 215.8 (s) and 216.2 (s, CO). <sup>31</sup>P{<sup>1</sup>H} NMR:  $\delta$  188.4 (dd, <sup>1</sup>J<sub>PP</sub> = 241.2 Hz, <sup>2</sup>J<sub>PP</sub> = 50.3 Hz, C=P), 106.4 (dd, <sup>1</sup>J<sub>PP</sub> = 163.5 Hz, <sup>2</sup>J<sub>PP</sub> = 50.3 Hz, P-Fe), -52.2 (dd, <sup>1</sup>J<sub>PP</sub> = 240.5, 163.5 Hz, PMes\*).

**X-ray Structure Determinations.** All data sets were collected on a Syntex P21 diffractometer using Mo K $\alpha$  ( $\lambda$  = 0.710 73 Å) radiation and a graphite monochromator. The crystal structure was solved by Patterson methods and refined with the SHELXL93 program using all symmetrically independent reflections in the range 4° ≤ 2 $\Theta$  ≤ 54°. The hydrogen atoms were included as constrained contributions in the final stages of least-squares refinement while using anisotropic temperature factors for all other atoms. Crystal data are assembled in Table 1.

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**Supporting Information Available:** Tables giving atomic coordinates, thermal parameters, and bond distances and angles for **9** (13 pages). Ordering information is given on any current masthead page.

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