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Transition-metal-substituted phosphanes, arsanes and stibanes, LXIII. Coupling reactions of alkynes with iron coordinated diphenylphosphane: generation of an alkenylphosphane and a chelating bisphosphane ligand[1]

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Abstract

The reaction of $\{Cp(OC)_2[H(Ph)_2P]Fe\}BF_4$ (**1**) with acetylenedicarboxylic acid dimethyl ester (**2**) in the presence of Et_3N followed by addition of HBF_4 leads to the cationic alkenylphosphane complex $[Cp(OC)_2Fe{-}P(Ph)_2{-}C(CO_2Me){=}C(CO_2Me)H]BF_4$ (**5**). In contrast, the reaction of the bis(diphenylphosphane) complex $\{Cp(OC)[H(Ph)_2P]_2Fe\}BF_4$ (**6**) with **2** yields $\{Cp(OC)Fe[P(Ph)_2{-}C(H)(CO_2Me){-}C(H)(CO_2Me){-}PPh_2]\}BF_4$ (**7**), bearing a functionalised dppe-analogous chelate phosphane. The molecular structures of **5** and **7** are determined by single crystal X-ray analysis. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Alkyne; Secondary phosphane complexes; P–H bond; Metal-assisted coupling reaction; Vinylphosphane; Chelate phosphane

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1. Introduction

Due to the activities in the field of the chemistry of complexes and metal catalysis, novel phosphane ligands are of increasing interest, especially those with stereogenic centers [2]. We focus on selective reactions of P–H units facilitated by coordination to a metal fragment [1]. In addition, this approach promises stereospecific reactions at the metal leading to the introduction of stereogenic centres into the phosphorus organic molecule.

Recently we found an easy insertion of diazoacetic ester into the P–H bond of cationic secondary phosphane iron complexes via the terminal nitrogen. In this case even the assistance of a base can be omitted which usually guarantees the intermediate formation of a metal phosphido species responsible for the coupling [1,3].

We now present the results of the reaction between acetylenedicarboxylic acid dimethyl ester and cationic secondary phosphane iron complexes of the type $\{Cp(OC)_2[H(R)_2P]Fe\}^{\oplus}$ and $\{Cp(OC)[H(R_2)P]_2Fe\}^{\oplus}$. Our interest is especially focused on the reaction of the alkyne with the bisphosphane complex **6**, representing access to the formation of chelate phosphane analogues of dppe at the metal.

2. Results

The reaction of the cationic diphenylphosphane iron complex **1** with the alkyne **2** under addition of Et_3N yields the phosphaferracyclopentenone complex **3** described for the first time by Enemark ([4]a). It is

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[1] Part LXII. See [1].

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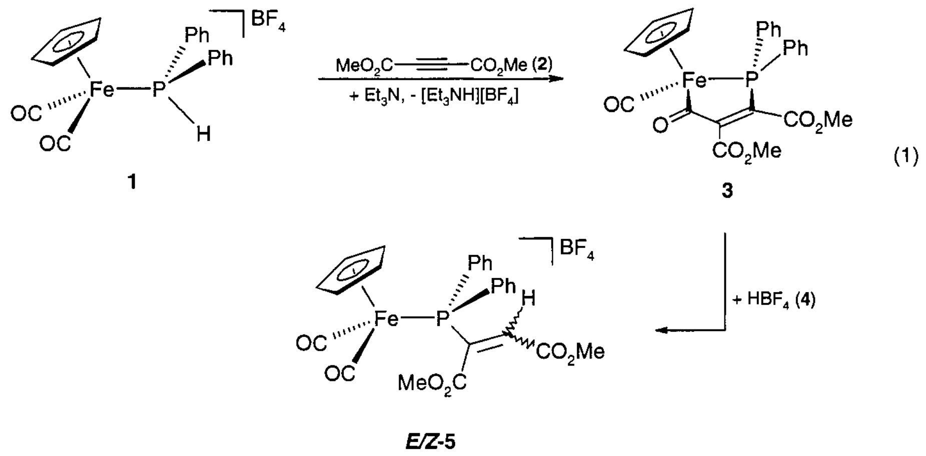


Scheme 1.

obvious that deprotonation of **1** affords the ferrio-phosphane $Cp(OC)_2Fe–PPh_2$ [5] which then couples with **2** in the established [3 + 2]-cycloaddition fashion [4]. The originally expected cationic product deriving from a P–H-insertion of the alkyne, the alkenyldiphenylphosphane complex **5** is not observed. Apparently, the acidity of the ammonium ion $HNEt_3^{\oplus}$ is not sufficient to cleave the C(O)–C=C-bond by protonation of the sp^2-carbon connected to the acyl carbon. However, treatment of **3** with HBF_4 (**4**) leads to an opening of the metallacycle **3** to produce **5** as a mixture of diastereomers (*E*/*Z*, 72:28) (Scheme 1).

In contrast to **1**, the bis(diphenylphosphane) iron complex **6** reacts with one equivalent of **2** in the presence of Et_3N to yield the functionalized chelate diphosphane complex **7** as a yellow microcrystalline powder (86%) (Scheme 2).

These results clearly indicate that the addition of the P–H unit of the secondary phosphane ligand to the C–C triple bond is favoured compared with a [3 + 2]-cycloaddition involving the carbonyl ligand according to the reaction of complex **1** which has only one phosphane coordinated to the metal. Introduction of a second Ph_2PH donor presumably reduces the electrophilicity of the CO carbon atom to such an extent, that C–C-coupling with the alkyne is prevented.

In the coupling reaction according to Scheme 2, **7** is exclusively formed as the *RR*/*SS*-diastereomers having the COOMe-groups in an *anti*-position. Evidence for this fact is given by the two doublet signals of the diastereotopic chelate phosphorus atoms in the ^{31}P-NMR and by the X-ray diffraction study.

It is suggested that **7** is formed by primary deprotonation of **6** to yield the ferrio-phosphane $Cp(OC)[H(Ph_2)P]Fe–PPh_2$ as an intermediate. Coupling with the alkyne and protonation of the resulting adduct leads to the alkenyldiphenylphosphane complex analogue of **5**, $Cp(OC)[H(Ph)_2P]Fe–P(Ph)_2–C(CO_2Me)=C(H)CO_2Me$. This process is repeated with the second diphenylphosphane ligand leading to an intramolecular attack of the metal-bound phosphido function at the activated sp^2-carbon. Protonation of the carbanion terminates the reaction sequence.

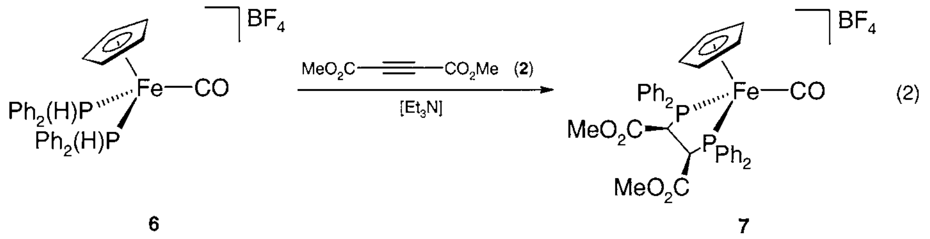
2.1. Molecular structures of *Z*-5 and 7

The central iron atom of the cationic complex salt *Z*-$\{Cp(OC)_2Fe[PPh_2–C(CO_2Me)=C(H)CO_2Me)]\}BF_4$ (***Z*-5**) (Fig. 1) shows an octahedral arrangement for the CO, Cp and phosphane ligands indicated by the corresponding bond angles (C1–Fe1–C2 93.49(18)°, C1–Fe1–P 93.33(11)°, C2–Fe1–P 90.58(12)°).

The bond lengths Fe–CO and Fe–P lie within the expected range (Fe1–C1 1.780(4) Å, Fe1–C2 1.782(4) Å [6], Fe–P 2.2334(10) Å [7]). The substituents at the phosphorus exhibit a staggered conformation with respect to the ligands at the metal, but showing a slight clockwise rotation around the P–Fe1 bond. One of the two phenyl units adopts the *anti*-position with respect to the Cp ligand [Cp(*Z*)Fe1–P–C10 158.90°], while the second one (C20–C25) and the alkenyl group are orientated *syn* to the CO ligands (C20–P–Fe1–C2 144.22(15)°, C30–P–Fe1–C1 171.44(14)°) (Fig. 2).

The substituents at the phosphorus atom show a distorted tetrahedral arrangement with the largest angles including the metal fragment (C10–P–Fe 114.82(8)°, C20–P–Fe 115.41(9)°, C30–P–Fe 113.57(8)°), whereas the angles between the organo groups are diminished (C10–P–C20 101.79(12)°, C10–P–C30 104.70(12)°, C20–P–C30 104.62(12)°).

The nearly planar alkenyl moiety (C41–C30–C31–C51 175.5(3)°) is characterized by a C=C-double bond [8] (C30–C31 1.328(4) Å) with the two ester units adopting an *E*-configuration. Although the two carbons involved in the C=C-double bond are nearly perfectly planar (sum of angles: C30 359.97°, C31 359.9°),



Scheme 2.

the individual angles, especially at C31, are quite different, showing the higher sterical demand of the CO_2Et group compared with the hydrogen atom H31 (C30–C31–C51 131.4(3)°, C30–C31–H31 118.9(1.5)°, C51–C31–H31 109.6(1.5)°).

The X-ray analysis of [Cp(OC)Fe–$PPh_2CH(CO_2Me)CH(CO_2Me)PPh_2$]$BF_4$ (**7**) (Fig. 3) shows an octahedral conformation of the Cp, CO and bisphosphane ligand (C1–Fe1–P2 86.90(11)°, C1–Fe1–P1 96.26(11)°, P2–Fe1–P1 88.09(3)°).

The short Fe1–C1 bond (1.754(4) Å) proves a strong back donation from the metal to the carbonyl group due to the electron-donating bisphosphane chelate ligand [7]. The difference in the Fe–P bond lengths (Fe1–P2 2.1851(10) Å, Fe1–P1 2.2033(10) Å) is evoked by the *anti*-position of the carboxy groups creating different geometries at the carbon and the phosphorus atoms. This different geometry is also indicated by the unequal conformations of the substituents at the phosphorus atoms with respect to the ligands at the iron atom. Although for both phosphorus units a staggered-conformation is observed, the organo groups at P1 show a counter-clockwise rotation of about 35° around the P1–Fe1-axis (Cp(*Z*)–Fe1–P1–C8 143.99°, Cp(*Z*)–Fe1–P1–C10 96.26°, Cp(*Z*)–Fe1–P1–C20 26.96°), whereas at the P2–Fe1-axis only a 15° clockwise rotation is found (Cp(*Z*)–Fe1–P2–C7 165.58°, Cp(*Z*)–Fe1–P2–C30 76.61°, Cp(*Z*)–Fe1–P2–C40 48.03°) (Fig. 4).

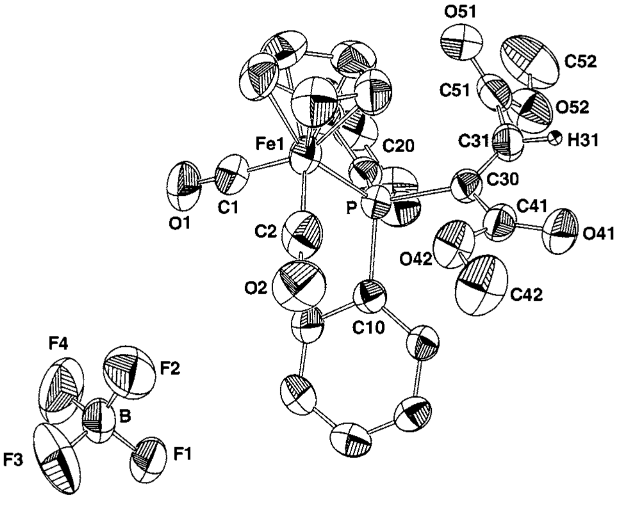


Fig. 1. ORTEP plot of {$Cp(OC)_2Fe[P(Ph)_2$–$C(CO_2Me)$=$C(H)CO_2Me]$}BF_4 (***Z*-5**). Selected bond lengths (Å), bond and torsion angles (°): Fe1–C1 1.780(4), Fe1–C2 1.782(4), Fe1–P 2.2334(10), P–C30 1.838(3), C30–C31 1.328(4); C1–Fe1–C2 93.49(18), C1–Fe1–P 93.33(11), C2–Fe1–P 90.58(12), C20–P–Fe1 115.88(10), C10–P–Fe1 114.89(9), C30–P–Fe1 113.65(9), C31–C30–P 126.7(2), C41–C30–P 119.3(2); C1–Fe1–P–C30 171.88(15), C2–Fe1–P–C30 −94.59(17), C41–C30–C31–C51 −173.8(3), P–C30–C31–C51 3.8(5),C31–C30–C41–O41 −23.7(4).

The substituents at both phosphorus atoms exhibit distorted tetrahedral arrangements with the smallest angles between the phenyl units (C10–P1–C20 102.65(14)°, C30–P2–C40 102.93(14)°) and the greatest ones between the iron centre and the phenyl groups (Fe1–P1–C10 120.04(10)°, Fe1–P1–C20 114.95(9)°, Fe1–P2–C30 123.78(10)°, Fe1–P2–C40 112.50(10)°).

The C–C bond of the ethylene moiety showing a value of 1.521(4) Å lies in the range of normal single bonds [8]. The P1–C8 (1.877(3) Å) and P2–C7 distances (1.865(3) Å) are, due to steric reasons, slightly elongated compared with P–C single bonds [6]. The torsion angle C71–C7–C8–C8 (− 60.2(3)°) proves the staggered-conformation of the carboxy groups with respect to the C–C bond with the phosphorus atoms P1, P2 occupying the '*trans*' positions to the CO_2Me groups (P1–C8–C7–C71 171.62(18)°, P2–C7–

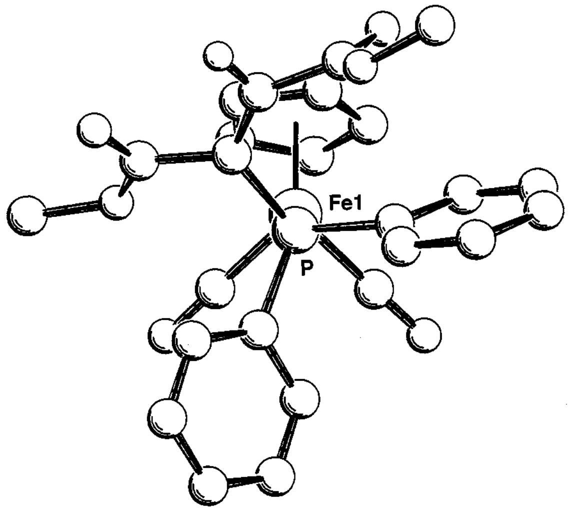


Fig. 2. View along the P–Fe1-axis of ***Z*-5**. The anion is omitted for clarity.

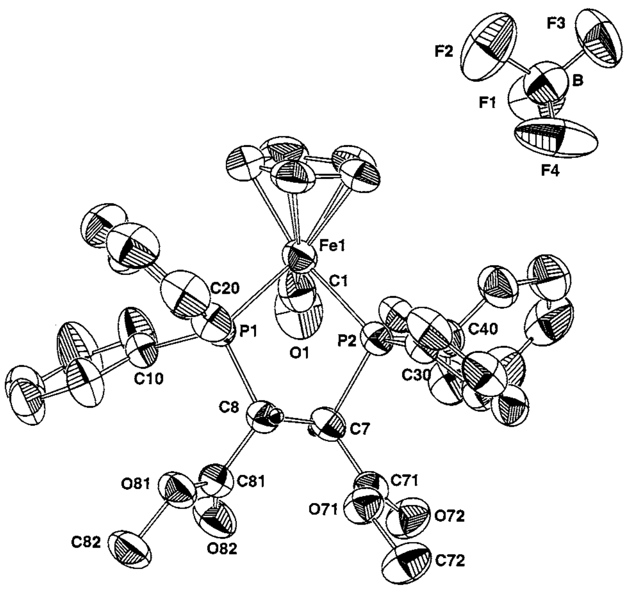


Fig. 3. ORTEP plot of [Cp(OC)Fe–$PPh_2CH(CO_2Me)CH(CO_2Me)PPh_2$]$BF_4$ (**7**). Selected bond lengths (Å), bond and torsion angles (°): Fe1–C1 1.754(4), Fe1–P2 2.1851(10), Fe1–P1 2.2033(10), P1–C8 1.877(3), P2–C7 1.865(3), C7–C8 1.521(4); C1–Fe1–P2 86.90(11), C1–Fe1–P1 96.26(11), P2–Fe1–P1 88.09(3), C8–P1–Fe1 106.99(10), C7–P2–Fe1 104.58(10), C8–C7–P2 106.47(18), C7–C8–P1 110.82(19); P2–Fe1–P1–C8 −7.57(9), P1–Fe1–P2–C7 29.74(10), Fe1–P2–C7–C8 −50.78(19), C71–C7–C8–C81 −60.2(3), P2–C7–C8–P1 46.1(2).

C8–C81 174.33(19)°). The P2–C7–C8–P1 torsion angle (46.1(2)°) verifies a strong distortion of the five-membered cycle due to the two sp^3-hybridized carbon-atoms involved in the ring system.

The same chelate ligand has been generated by Treichel and Wong [9] starting from neutral *fac*-{$Mn(CO)_3[P(Ph)_2H]_2Br$}. But in this case deprotonation to give the reactive phosphido species requires *n*-BuLi. Moreover, this type of metal fragment is not suitable to exert stereocontrol in coupling reactions. Pseudo-tetrahedral half-sandwich fragments of the iron type are known to be efficient in this respect as proved by a series of interesting metal-mediated organic syntheses [10,11]. Therefore, forthcoming experiments will be directed towards the synthesis of chelating diphosphanes with several functional and stereogenic centres.

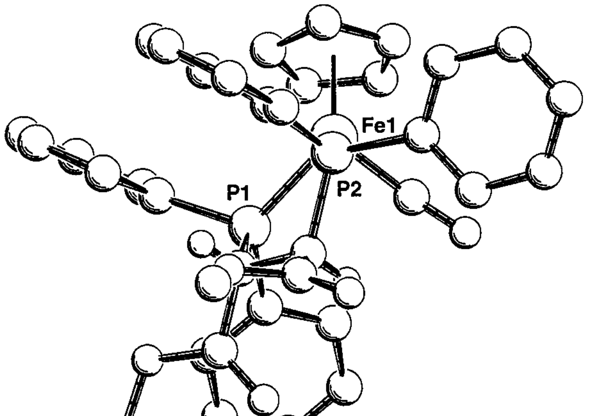


Fig. 4. View along the P2–Fe1-axis of **7**. The anion is omitted for clarity.

3. Experimental section

3.1. General information and instrumentation

All manipulations were carried out under dry N_2 using Schlenk techniques. Solvents were rigorously dried with an appropriate drying agent, distilled before use and saturated with nitrogen. ^1H-, ^{13}C- and ^{31}P-NMR, Bruker AMX 400; IR, Perkin Elmer, Model 283. Melting points, differential thermal analysis (DTA), Du Pont 9000.

3.2. *{(η^5-Cyclopentadienyl)(dicarbonyl)[1,2-di(methylcarboxylato)vinyl-diphenylphosphane]iron(II)} tetrafluoroborate* (**5**)

A solution of 245 mg (0.54 mmol) of {$Cp(OC)_2[Ph_2(H)P]Fe$}BF_4 (**1**) and 85 mg (0.60 mmol) of acetylenedicarboxylic acid dimethyl ester (**2**) in 12 ml of dichloromethane is treated with 61 mg (0.60 mmol) of Et_3N. The reaction mixture is stirred for 2 d at room temperature and the presence of Cp(OC)Fe–PPh_2–$C(CO_2Me)$=$C(CO_2Me)C(=O)$ (3) ([4]a) can be detected by ^{31}P-NMR. 70 mg (0.80 mmol) of HBF_4/H_2O (1:1) (**4**) are added, and the mixture is stirred for another 2 d. Volatiles are removed in vacuo and the residue extracted with 3 ml of acetonitrile. After addition of 20 ml of ether the formed precipitate is separated, washed three times each with 5 ml of ether and dried in vacuo. Yield 233 mg (73%). Yellow-orange crystalline powder. M.p. 195°C (decomp.). Diastereomeric ratio 72:28. ***E*-5**: ^1H-NMR ([D_3]-acetonitrile, 300.4 MHz): $\delta = 7.71–7.42$ (m, 10 H, H_5C_6), 6.65 (s, 1 H, HC=C), 5.58 [d, ^3J(PFeCH) = 1.6 Hz, 5 H, H_5C_5], 3.90 (s, 3 H, H_3C), 3.79 ppm (s, 3 H, H_3C).$^{31}P\{^1H\}$-NMR ([D_3]-acetonitrile, 121.5 MHz): $\delta = 59.8$ ppm (s). IR (acetonitrile): $\nu(CO) = 2047$ (s), 2002 (s); $\nu(C{=}O) = 1738$ (s) cm^{-1}. ***Z*-5**: ^1H-NMR ([D_3]-acetonitrile, 300.4 MHz): $\delta = 7.71–7.42$ (m, 10 H, H_5C_6), 6.22 [d, ^3J(PCCH) = 15.2 Hz, 1 H, HC=C], 5.32 [d, ^3J(PFeCH) = 1.7 Hz, 5 H, H_5C_5], 3.73 (s, 3 H, H_3C), 3.25 ppm (s, 3 H, H_3C). $^{31}P\{^1H\}$-NMR ([D_3]-acetonitrile, 121.5 MHz): $\delta = 65.9$ ppm (s). IR (acetonitrile): $\nu(CO) = 2047$ (s), 2002 (s); $\nu(C{=}O) = 1738$ cm^{-1} (s). $C_{25}H_{22}BF_4FeO_6P$ (592.07): calcd. C 50.72, H 3.75; found C 50.12, H 3.32.

3.3. {[RR/SS1,2-Bis(diphenylphosphino)-1,2-bis(methylcarboxylato)ethane](carbonyl)(η^5-cyclopentadienyl)iron(II)}tetrafluoroborate (**7**)

A solution of 318 mg (0.52 mmol) of $\{Cp(OC)[Ph_2(H)P]_2Fe\}BF_4$ (**6**) in 15 ml of dichloromethane is treated with 74 mg (0.52 mmol) of acetylenedicarboxylic acid dimethyl ester (**2**) and 20 mg (0.20 mmol) of Et_3N and the mixture is stirred for 3 d at room temperature. The volume of the reaction mixture is reduced in vacuo to 2 ml. After the addition of 20 ml of ether, the precipitate is separated, washed three times each with 5 ml of ether and dried in vacuo to give **7**. Yield 338 mg (86%). Yellow crystalline powder. M.p. 204°C (decomp.). ^{1}H-NMR ([D$_3$]-acetonitrile, 400.1 MHz): $\delta = 7.86$–7.24 (m, 20 H, H_5C_6), 4.75 [t, $^3J(\text{PFeCH}) = 1.6$ Hz, 5 H, H_5C_5], 4.35–4.25 (m, 2 H, HCCH), 3.29 (s, 3 H, H_3CO), 3.14 ppm (s, 3 H, H_3CO). $^{13}C\{^1H\}$-NMR ([D$_3$]-acetonitrile, 100.6 MHz): $\delta = 213.5$ (s, CO), 168.2 [dd, $^2J(\text{PCC}) = 19.1$ Hz, $^3J(\text{PCCC}) = 5.0$ Hz, C=O], 167.3 [dd, $^2J(\text{PCC}) = 17.1$ Hz, $^3J(\text{PCCC}) = 5.0$ Hz, C=O], 134.9–128.4 (m, C_6H_5), 52.3 (s, CH_3O), 52.1 (s, CH_3O), 50.6 [dd, $^1J(\text{PC}) = 21.6$ Hz, $^2J(\text{PCC}) = 15.6$ Hz, CH], 49.2 ppm [dd, $^1J(\text{PC}) = 20.1$ Hz, $^2J(\text{PCC}) = 17.1$ Hz, CH]. $^{31}P\{^1H\}$-NMR ([D$_3$]-acetonitrile, 162.0 MHz): $\delta = 100.4$ [d, $^2J(\text{PFeP}) = 43.8$ Hz], 96.7 ppm [d, $^2J(\text{PFeP}) = 43.8$ Hz]. IR (acetonitrile): $\nu(CO) = 1983$ (vs); $\nu(C{=}O) = 1783$ (vs) cm^{-1}. $C_{36}H_{33}BF_4FeO_5P_2$ (750.25): calcd. C 57.63, H 4.43; found C 55.95, H 4.31.

3.4. X-ray analysis of {(η^5-cyclopentadienyl)(dicarbonyl)[(Z-1,2-di(methylcarboxylato)-vinyl)diphenylphosphane]iron(II)}-tetrafluoroborate (**Z-5**) and {[(trans-2,3-bis(diphenylphosphino)butane-1,4-dicarboxylicdimethylester]-(carbonyl)(η^5-cyclopentadienyl)-iron(II)}tetrafluoroborate (**7**)

Suitable orange crystals were obtained by slow diffusion of ether into a saturated solution of ***Z*-5** and **7**, respectively, in dichloromethane. **5**: $C_{25}H_{22}BF_4FeO_6P$; $M = 592.06$; monoclinic; space group, $P2_1/n$ (No. 1014); $a = 13.691(3)$ Å, $b = 14.072(5)$ Å, $c = 14.205(6)$ Å, $\alpha = \gamma = 90.00°$, $\beta = 108.251(13)°$, $V = 2599.1(15)$ Å^3, $Z = 4$; CAD4-diffractometer (Enraf-Nonius); radiation type, Mo–K$_\alpha$; wavelength, $\lambda = 0.71073$ Å; graphite monochromator; $D_{\text{calc.}} = 1.513$ g cm^{-3}; crystal size, $0.40 \times 0.30 \times 0.30$ mm; scale range, $1.80° < \Theta < 24.92°$; $F(000)$, 1208; temperature, 293(2) K; total reflections, 9386; observed reflections, 3389 with $(I > 2.0\sigma I)$; absorption coefficient $\mu = 0.709$ mm^{-1}; semi-empirical absorption correction ($T_{\min}/T_{\max}$, 0.9247/0.9989); structure solution, SHELXS-96 [12] with patterson methods; structure refinement, SHELXL-96 [13] (385 parameters), $R_1 = 0.0389$, $wR_2 = 0.1061$. **7**: $C_{36}H_{33}BF_4FeO_5P_2$, $M = 750.22$, monoclinic; space group, $P2_1/n$ (No. 1014); $a = 12.537(3)$ Å, $b = 16.908(6)$ Å, $c = 16.620(7)$ Å, $\alpha = \gamma = 90.00°$, $\beta = 107.781(14)°$, $V = 3355(2)$ Å^3, $Z = 4$; CAD4-diffractometer (Enraf-Nonius); radiation type, Mo–K$_\alpha$; wavelength, $\lambda = 0.71073$ Å; graphite monochromator; $D_{\text{calc.}} = 1.485$ g cm^{-3}; crystal size, $0.20 \times 0.15 \times 0.15$ mm; scale range, $1.76° < \Theta < 25.06°$; $F(000)$, 1544; temperature, 293(2) K; total reflections, 8856; observed reflections, 4178 with $(I > 2.0\sigma I)$; absorption coefficient $\mu = 0.610$ mm^{-1}; semiempirical absorption correction ($T_{\min}/T_{\max}$, 0.8952/0.9999); structure solution, SHELXS-96 [12] with patterson methods; structure refinement, SHELXL-96 [13] (489 parameters), $R_1 = 0.0385$, $wR_2 = 0.0970$ [14].

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[14] Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as 'supplementary publication no. CCDC-101572 and 101573'. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).