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# Synthesis of the tertiaryphosphine derivatives of iron carbonyl phosphines: preparation of tetracarbonyl[tris(2-furyl)phosphine]iron(0), pentacarbonylbis[μ-bis(2-furyl)phosphido]-[tris(2-furyl)phosphine]diiron(0), tetracarbonyl[tris(2-benzofuryl)phosphine]iron(0) and pentacarbonylbis[μ-bis(2-benzofuryl)phosphido]-[tris(2-benzofuryl)phosphine]diiron-(0)

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## Abstract

Pentacarbonyl iron reacts with tris(2-furyl)phosphine (or tris(2-benzofuryl)phosphine) in the presence of sodium borohydride in refluxing *n*-butanol to give the title compounds. The crystal and molecular structure of pentacarbonylbis[μ-bis(2-furyl)phosphido][tris(2-furyl)phosphine]diiron(0) were determined by X-ray crystallography.

*Keywords:* Iron; Dinuclear; Carbonyls; X-ray structure; Benzoferrylphosphine complex

## 1. Introduction

Mononuclear carbonylmetallate anions and dianions, such as carbonylferrates, are well-established species [1], and have been valuable stoichiometric reagents in organic chemistry [2], metal cluster synthesis and the preparation of novel mononuclear organometallic compound [3]. Carbonylferrates are strong nucleophiles that readily undergo alkylation and acylation reactions [2].

Controlling the degree of substitution of iron pentacarbonyl by neutral ligands has usually been difficult. Coordinatively saturated iron pentacarbonyl undergoes substitution only under thermal or photochemical conditions which promote the loss of carbon monoxide. Functionalized metal carbonyl anions $[(CO)_xMX]^{y-}$ offer an extra dimension of reactivity in that transformations can follow metal–carbon σ-bond formation. A variety of iron carbonyl complexes such as $[Fe(CO)_xL_y]^{z-}$ and $[Fe(CO)_xL^1L^2]^{y-}$ (L,L$^1$ or $L^2 = R_3Si$ [4], $R_3P$ or $(RO)_3P$ [5], $R_3Si$, $R_3Sn$ or $R_3P$[6] have been synthesized previously.

With the aim of using iron carbonyl complexes as catalysts, we decided to prepare iron species of the type $[Fe(CO)_x\ P(2\text{-furyl})_{3y}]^{z-}$ using the relatively electron-poor tris(2-furyl)phosphine (**1a**). Among triarylphosphines, the 2-furyl group appears to act as most strongly electron-withdrawing group [7]. It is thought that, in complexes of this kind, the high electron charge on the metal is stabilized by the low donor effect of the ligand [8]. We have also synthesized tris(2-benzofuryl)phosphine (**1b**) for the first time and have undertaken the study of its reactivity towards iron pentacarbonyl:

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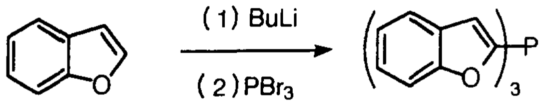


**1b**

## 2. Results

Three approaches were used to prepare complexes. First, the method previously employed for $[Fe(CO)_3(PPh_3)_2]$ was used. Thus P(2-furyl)$_3$ was added to an ethanolic solution of KH $[Fe(CO)_4]$ (prepared from $[Fe(CO)_5]$ and KOH in ethanol) at 70°C for 24 h [5m]. Despite the use of exactly 2 mol equivalents of phosphine, the only product obtained with a low yield, was the previously unknown complex $[Fe(CO)_4\{P(2\text{-furyl})_3\}]$ (**2a**) (20%). The structure was assigned on the basis of spectral data. Secondly, **2a** was obtained with a better yield (42%) by treating $[Fe(CO)_5]$ with $LiAlH_4$ in refluxing tetrahydrofuran (THF) in the presence of P(2-furyl)$_3$, using Siegl's procedure [5a]. Thirdly, we have found that $[Fe(CO)_5]$ reacts with P(2-furyl)$_3$ and $NaBH_4$ in refluxing *n*-butanol [5k] to give $[Fe(CO)_4\{P(2\text{-furyl})_3\}]$ (**2a**) (43%) and a new crystalline, air-stable complex **3a** (11%). High resolution mass spectra of **3a** are consis-

Table 1
Selected bond distances (Å) and angles (°) for $[Fe_2(CO)_5\{P(2\text{-furyl})_3\}(\mu\text{-}P(2\text{-furyl})_2)_2]$

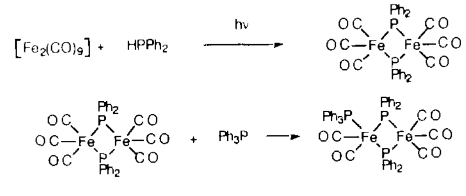
| | | | | | |
|---|---|---|---|---|---|
| *Bond distances* | | | | | |
| Fe(1)–P(1) | 2.203(2) | Fe(2)–P(2) | 2.248(3) | P(1)–C(6) | 1.815(9) |
| Fe(1)–P(2) | 2.191(2) | Fe(2)–P(3) | 2.241(3) | P(1)–C(10) | 1.81(1) |
| Fe(1)–P(3) | 2.197(2) | Fe(2)–C(3) | 1.76(1) | P(1)–C(14) | 1.800(9) |
| Fe(1)–C(1) | 1.82(1) | Fe(2)–C(4) | 1.75(1) | P(2)–C(26) | 1.83(1) |
| Fe(1)–C(2) | 1.757(9) | Fe(2)–C(5) | 1.74(1) | P(2)–C(30) | 1.83(1) |
| | | | | P(3)–C(18) | 1.799(9) |
| | | | | P(3)–C(22) | 1.819(9) |
| *Bond angles* | | | | | |
| Fe(2)–Fe(1)–P(1) | 118.57(8) | Fe(1)–Fe(2)–P(2) | 51.34(7) | | |
| Fe(2)–Fe(1)–P(2) | 53.26(8) | Fe(1)–Fe(2)–P(3) | 51.54(7) | | |
| Fe(2)–Fe(1)–C(1) | 140.1(3) | Fe(1)–Fe(2)–C(3) | 97.3(3) | | |
| Fe(2)–Fe(1)–C(2) | 99.8(3) | Fe(1)–Fe(2)–C(4) | 140.9(4) | | |
| P(1)–Fe(1)–P(2) | 171.7(1) | Fe(1)–Fe(2)–C(5) | 112.8(3) | | |
| P(1)–Fe(1)–P(3) | 95.06(9) | P(2)–Fe(2)–P(3) | 78.6(1) | | |
| P(1)–Fe(1)–C(1) | 92.9(3) | P(2)–Fe(2)–C(3) | 91.9(3) | | |
| P(1)–Fe(1)–C(2) | 89.3(3) | P(2)–Fe(2)–C(4) | 97.4(4) | | |
| P(2)–Fe(1)–P(3) | 80.83(9) | P(2)–Fe(2)–C(5) | 164.2(3) | | |
| P(2)–Fe(1)–C(1) | 95.1(3) | P(3)–Fe(2)–C(3) | 146.0(4) | | |
| P(2)–Fe(1)–C(2) | 90.9(3) | P(3)–Fe(2)–C(4) | 105.7(4) | | |
| P(3)–Fe(1)–C(1) | 103.8(3) | P(3)–Fe(2)–C(5) | 90.7(3) | | |
| P(3)–Fe(1)–C(2) | 150.7(4) | C(3)–Fe(2)–C(4) | 107.9(5) | | |
| C(1)–Fe(1)–C(2) | 104.9(5) | C(3)–Fe(2)–C(5) | 90.5(5) | | |
| | | C(4)–Fe(2)–C(5) | 96.7(5) | | |

Table 2
Comparison of mean geometric parameters for $[(^iPr_2NP)(^iPr_2NPCl)PFe_2(CO)_6]$ (**4**), $[Fe_2(\mu\text{-}PPh)_2(CO)_6]$, $[Fe_2(CO)_5\{P(2\text{-furyl})_3\}\{\mu\text{-}P(2\text{-furyl})_2\}_2]$ (**3a**) and $[Fe_2\{\mu\text{-}P(CF_3)_2\}_2(CO)_6]$

| Compound | Fe...Fe (Å) | Fe–P (Å) | Fe–P–Fe (°) | P–F–P (°) | Dihedral angle [a] (°) | Reference |
|---|---|---|---|---|---|---|
| $[(^iPr_2NP)(^iPr_2NPCl)PFe_2(CO)_6]$ **4** | 2.593(2) | 2.193(3)<br>2.332(3)<br>2.198(3)<br>2.303(3) | 72.4(9)<br>68.0(1) | 76.1(1)<br>76.6(1) | – | 12 |
| $[Fe_2(\mu\text{-}PPh)_2(CO)_6]$ | 2.623(2) | 2.233(2) | 72.0(1) | 79.9(1) | 100 | 13 [b] |
| $[Fe_2(CO)_5\{P(2\text{-furyl})_3\}\{\mu\text{-}P(2\text{-furyl})_2\}_2]$ **3a** | 2.715(2) | 2.191(2)<br>2.197(2)<br>2.248(3)<br>2.241(3) | 75.40(8)<br>75.45(8) | 80.83(9)<br>78.6(1) | 105.5(0.6) | this work |
| $[Fe_2\{\mu\text{-}P(CF_3)_2\}_2(CO)_6]$ | 2.819(1) | 2.193(1)<br>2.194(2) | 80.0(1)<br>79.9(1) | 83.5(1) | 118.9 | 14 |

[a] Dihedral angle between the planes containing $P_2$, $Fe_1$, $P_3$ and $P_2$, $Fe_2$, $P_3$ providing a quantitative measure of the folding of the $Fe_2P_2$ ring along the $P_2 \ldots P_3$ vector.

[b] For features of $Fe_2(\mu\text{-}PRR')_2(CO)_6$ complexes, see [14,15].



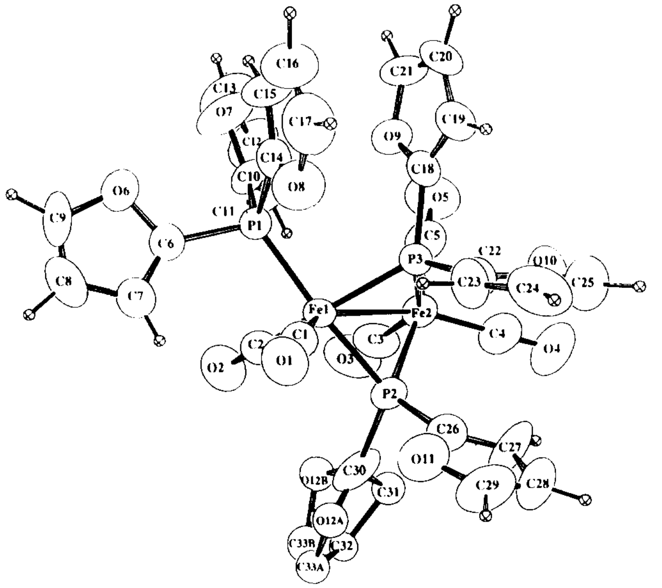
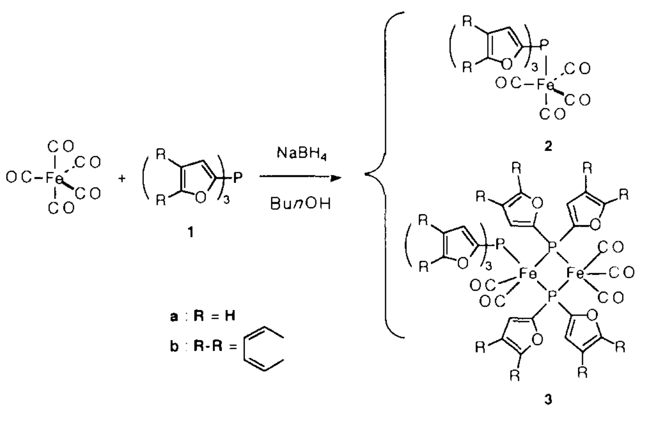


Fig. 1. ORTEP plot of **3a**.

tent with the formulation $[Fe_2(CO)_5\{P(2\text{-furyl})_3\}\{\mu\text{-}P(2\text{-furyl})_2\}_2]$:



Although the spectroscopic data are compatible with this dinuclear formulation for **3a**, X-ray crystallographic characterization was carried out. An ORTEP drawing is shown in Fig. 1 [9].

Thermally initiated displacement of carbon monoxide from $[(CO)_3Fe(\mu\text{-}PPh_2)_2Fe(CO)_3]$ by phosphines and phosphites has been shown to give derivatives similar to those reported here [10,11].

Selected bond distances and angles for $[Fe_2(CO)_5\{P(2\text{-furyl})_3\}\{\mu\text{-}P(2\text{-furyl})_2\}_2]$ are given in Table 1.

The data presented in Table 2 show clearly that the detailed geometry of the central $Fe_2P_2$ ring varies considerably with the electronegativity of R in the bridging ligand $PR_2$. The observed molecular structures of $[(^{i}Pr_2NP)(^{i}Pr_2NPCl)PFe_2(CO)_6]$ (**4**), $[Fe_2(\mu\text{-}PPh)_2(CO)_6]$, $[Fe_2(CO)_5\{P(2\text{-furyl})_3\}\{\mu\text{-}P(2\text{-furyl})_2\}_2]$ (**3a**) and $[Fe_2\{\mu\text{-}P(CF_3\}_2)_2(CO)_6]$ demonstrate the electron-withdrawing property of the 2-furyl substituents on the phosphorus atoms in **3a**. In particular, the Fe–Fe bond distance (2.715 Å) is considerably longer than in hexacarbonyl $[Fe_2(\mu\text{-}PPh)_2(CO)_6]$(2.623 Å). In contrast, the Fe–P distances fall within a small overall range (2.191–2.303 Å).

Treatment of $[Fe(CO)_5]$ with tris(2-benzofuryl)phosphine (**1b**) in the presence of $NaBH_4$ in refluxing *n*-butanol [5k] gave two air-stable products, tetracarbonyl[tris(2-benzofuryl)phosphine]iron(0) (**2b**) (20%) and pentacarbonylbis[μ-bis(2-benzofuryl)-phosphido]-[tri(2-benzofuryl)phosphine]diiron(0) (**3b**) (15%). Thus similar behaviour was observed for both **1a** and **1b** towards iron pentacarbonyl under reducing conditions.

Binuclear transition metal complexes have been the subject of intense research activity [16]. We report here two novel and stable phosphido-bridged complexes bearing unusual ligands, together with two new mononuclear complexes.

## 3. Experimental details

### 3.1. General

All reactions were run under argon in oven-dried glassware. Thin layer chromatography (TLC) was performed on silica gel 60 $F_{254}$ (Merck). $^1H$ and $^{13}C$ NMR spectra were recorded in $CDCl_3$ solutions at 200 and 50 MHz using a Bruker AC-200 spectrometer or at 400 and 100 MHz using a Bruker AM-400 spectrometer. $^{31}P$ NMR spectra were recorded in $CDCl_3$ solutions at 80 MHz using a Bruker AC-200 spectrometer. Carbon–proton couplings were determined by DEPT sequence experiments [17].

### 3.2. Tris(2-furyl)phosphine (1a)

A 21 round-bottomed flask equipped with a mechanical stirrer was charged with furan (152 g, 2.25 mol) in anhydrous ether (300 ml) and cooled to −30°C. Butyllithium (1.5 mol) prepared from lithium (22 g, 3.14 atg), butyl bromide (205 g, 1.5 mol) in anhydrous ether (500 ml) was added. The temperature was maintained at −30°C for 4 h. Then phosphorus tribromide (73 g, 0.27 mol) in anhydrous ether (200 ml) was added at 0°C. After stirring for 2 h, the solution was warmed to room temperature and stirred for 12 h. After hydrolysis and extraction with ether, the organic layer was washed with hydrochloric acid (0.1 N) and dried over $MgSO_4$. Concentration in vacuo gave the crude product. Complex **1a** was purified by chromatography on silica gel (pentane : ether, 90 : 10) or by crystallization from petroleum ether (60–80°C) (yield, 87%); 63°C melting point (m.p.), (63°C [18]). $^1$H NMR: $\delta$ 7.64 (3, m), 6.79 (3, m), 6.38 (3, m) ppm $^{13}$C{$^1$H} NMR: $\delta$ 148.8 (s, $J_{CP} = 3.04$ Hz), 147.5 (d, $J_{CP} = 2.77$ Hz), 121.2 (d, $J_{CP} = 25.5$ Hz), 110.8 (d, $J_{CP} = 6.5$ Hz) ppm [19]. $^{31}$P{$^1$H} NMR: $\delta$ −78.4 ppm. Anal. Found: C, 62.09, H, 4.13. $C_{12}H_9O_3P$ Calc.: C, 62.06; H, 3.9%.

### 3.3. *Tris(2-benzofuryl)phosphine (**1b**)*

The same procedure was used, furan being replaced by benzofuran. The crude product was purified by chromatography on silica gel, eluting with pentane: ether (95 : 5) or by crystallization from petroleum ether (80–100°C) yield, (43%; m.p. 152–153°C). IR ($CCl_4$): 1535, 1442, 1258 cm$^{-1}$. $^1$H NMR: $\delta$ 7.35 (m), 7.65 (m) ppm. $^{13}$ C{$^1$H} NMR; $\delta$ 158.1 (s, $J_{CP} = 3.3$ Hz), 150.8 (s, $J_{CP} = 5.18$ Hz), 127.8 (d, $J_{CP} = 6.24$ Hz), 125.6 (d), 123.0 (d), 121.4 (d), 118.4 (d, $J_{CP} = 22.48$ Hz), 111.7 (d) ppm. $^{31}$P {$^1$H} NMR: $\delta$ −68.7 ppm. Mass spectroscopy (MS): *m/z* 384 (3), 383 (28), 382 ($M^+$)(100), 289 (6), 264 (10), 234 (75), 205 (11), 189 (11), 148 (57), 120 (20). HRMS Found: 382.0754 $C_{24}H_{15}O_3P$ calc.: 382.0758. Anal. Found: C, 75.22; H, 3.99. $C_{24}H_{15}O_3P$ Calc.: C, 75.39; H, 3.95%.

### 3.4. *Preparation of **2a**, **3a**, **2b** and **3b***

A 250 ml two-necked round-bottomed flask equipped with a magnetic stirrer and a reflux condenser was charged with tris(2-furyl)phosphine (2.78 g, 12 mmol), butanol (100 ml) and $NaBH_4$ (0.19 g, 5 mmol). The mixture was stirred under argon, and iron pentacarbonyl (0.66 ml, 5 mmol) was added dropwise with a syringe. When gas evolution ceased, the mixture was heated under reflux for 4 h. After filtration, the cake was washed with $CH_2Cl_2$. The solvent was removed in vacuo. The crude product in solution in $CH_2Cl_2$ was chromatographed on silica gel (pentane : ether, 90 : 10): (yields, 43% **2a**, 11% **3a**, 20% **2b**, 15% **3b**).

### 3.5. *Tetracarbonyl[tris(2-furyl)phosphine]iron(0) (**2a**)*

(M.P. 119–120°C (light petroleum). IR ($CCl_4$): $\nu$ 2060 (m), 1980 (m), 1950 (s) cm$^{-1}$. $^1$H NMR: $\delta$ 7.72 (s, br), 7.03 (s, br), 6.49 (s, br) ppm. $^{13}$C{$^1$H} NMR: $\delta$ 212.3 (s, $J_{CP} = 20.48$ Hz), 149.0 (d, $J_{CP} = 5.63$ Hz), 145.9 (s, $J_{CP} = 79.16$ Hz), 123.6 (d, $J_{CP} = 21.43$ Hz), 111.4 (d, $J_{CP} = 8.35$ Hz) ppm. MS: *m/z* 400 (12), 372 (24), 316 (76), 288 (97), 232 (21), 190 (32), 154 (100). HRMS Found: 399.9447 $C_{16}H_9O_7P^{56}Fe$ Calc.: 399.9435. $^{31}$P{$^1$H} NMR: $\delta$ 21.82 (coordination chemical shift, 100.22 ppm [20]). Anal. Found: C, 48.00; H, 2.25. $C_{16}H_9FeO_7P$ Calc.: 48.04; H, 2.27%.

### 3.6. *Pentacarbonylbis[μ-bis(2-furyl)phosphido][tris(2-furyl)phosphine]diiron(0) (**3a**)*

IR ($CCl_4$); $\nu$ 2040 (m), 1980 (vs), 1945 (m) cm$^{-1}$. $^1$H NMR: $\delta$ 7.59 (3, s), 7.33 (2, s), 7.27 (2, s), 6.88 (3, s), 6.36 (5, br s), 6.10 (2, br s), 5.98 (2, s), 5.80 (2 br s) ppm. $^{13}$C{$^1$H} NMR: $\delta$ 212.63 (s), 212.60 (s), 212.56 (s), 148.8 (s,$J_{CP} = 70.0$ Hz), 147.3 (d, $J_{CP} = 5.0$ Hz), 146.4 (d), 145.4 (d), 121.1 (d, $J_{CP} = 18.0$ Hz), 119.8 (d, $J_{CP} = 8.0$ Hz), 119.7 (d, $J_{CP} = 8.0$ Hz), 119.5 (d), 111.1 (d, $J_{CP} = 15.0$ Hz), 111.0 (d, $J_{CP} = 6$ Hz); ppm. $^{31}$P{$^1$H} NMR: $\delta$ 78.2 (s, br)(2P), 20.5 (t, $J_{PP} = 10.2$ Hz)(1P). MS: *m/z* 814 (2), 786 (3), 758 (5), 730 (6), 674 (11), 498 (3), 442 (14), 337 (3), 232 (20), 165 (8), 152 (9), 134 (9), 109 (9), 98 (7), 78 (9), 70 (16), 69 (10), 57 (12), 56 (15), 43 (18), 28 (100). HRMS, Found, 813.9010. $C_{33}H_{21}O_{12}P_3^{56}Fe_2$ Calc.: 813.8944. Anal. Found: C, 48.67; H, 2.65. $C_{33}H_{21}O_{12}P_3Fe_2$ Calc. C, 48.69; H, 2.60%.

### 3.7. *Tetracarbonyl[tris(2-benzofuryl)phosphine]iron(0) (**2b**)*

M.p. 165–166°C (petroleum ether); IR ($CCl_4$): $\nu$ 2060 (m), 2010 (vw), 1992 (m), 1960 (vs), 1920 (m), 1910 (w), 1255, 1115, 1080, 925 cm$^{-1}$. $^1$H NMR: $\delta$ 7.80–7.20 (m) ppm. $^{13}$C{$^1$H} NMR: $\delta$ 211.9 (s), 127.1 (d), 123.7 (d), 122.4 (d), 122.3 (s, $J_{CP} = 77.0$ Hz), 120.6 (d, $J_{CP} = 22.0$ Hz), 112.3 (d); ppm. $^{31}$P{$^1$H} NMR: $\delta$ 28.4 (coordination chemical shift, 97.1 ppm [20]). Anal. Found: C, 61.04; H, 2.78. $C_{28}H_{15}FeO_7P$ Calc.: C, 61.12; H, 2.75%.

### 3.8. *Pentacarbonylbis[μ-bis(2-benzofuryl)phosphido]-[tris(2-benzofuryl)phosphine]diiron(0) (**3b**)*

M.p. 215–216°C (petroleum ether). IR ($CCl_4$): $\nu$ 2040 (m), 2000 (vs), 1970 (s), 1950 (m), 1255, 915 cm$^{-1}$ $^1$H NMR: $\delta$ 7.8–6.7 (m) ppm. $^{13}$C{$^1$H} NMR $\delta$ 214.1 (s), 212.0 (s), 158.1 (s), 157.7 (s, $J_{CP} = 6.0$ Hz), 157.2 (s), 156.3 (s), 151.2 (br s, $J_{CP} = 68.0$ Hz), 150.2 (s, $J_{CP} = 68.5$ Hz,), 127.9 (s), 127.3 (s, $J_{CP} = 7.0$ Hz), 126.2 (d), 125.6 (d), 125.25 (d), 124.0 (d), 123.3 (d), 123.1 (d), 122.95 (d), 122.9 (d), 122.0 (d), 121.5 (d), 121.0 (d), 120.3 (d), 118.4 (d, m), 116.5 (d, m), 111.8 (d), 111.7 (d), 111.2 (s), 110.6 (d) ppm. $^{31}$P{$^1$H} NMR: $\delta$ 73.5, 28.9, 27.3. MS (fast atom bombardment mode, matrix, *m*-nitrobenzyl alcohol): 1167 (6), 1166 (18), 1165 (42), 1164 (34) ($C_{61}H_{35}O_{12}P_3^{56}Fe_2$), 1137 (11), 1136 (9)($M^+ - CO$), 1110 (13), 1109 (40), 1108 (54)($M^+ - 2CO$), 1082 (18), 1081 (42), 1080 (64)($M^+ - 3CO$), 1026 (23), 1025 (60), 1024 (100)[($M^+ - 5CO$) or ($M^+ - Fe(CO)_3$)], 907 (10)[($M^+$ −(benzofuryl)-

Fe(CO)$_3$)], 827 (22), 759 (12)(M$^+$ – [(benzofuryl)$_2$PFe(CO)$_3$], 643 (43), 642 (100)(M$^+$ – [(benzofuryl)$_3$PFe(CO)$_3$], 525 (2)(M$^+$ – [(benzofuryl)$_4$PFe(CO)$_3$], 494 (4)[((benzofuryl)$_3$PFe(CO)$_2$)$^+$], 391 (13), 307 (28), 265 (12)[((benzofuryl)$_2$P)$^+$]. Anal. Found: C, 63.01; H, 3.04. $C_{61}H_{35}Fe_2O_{12}P_3$ Calc.: C, 62.91; H, 3.03%.

### 3.9. X-ray crystallography of $C_{33}H_{21}Fe_2O_{12}P_3$ (3a)

$C_{33}H_{21}Fe_2O_{12}P_3$: $M_1 = 814.1$; orthorhombic: $Pna2_1$; $a = 22.553(6)$, $b = 16.552(6)$ and $c = 9.250(1)$ Å; $V = 3452(1)$ Å$^3$; $Z = 4$; $\rho_{calc} = 1.566$ g cm$^{-3}$; $\lambda$(Mo K$\alpha$) = 0.709 26 Å; $\mu = 10.36$ cm$^{-1}$; $F(000) = 1648$; $T = 294$ K; final $R = 0.039$ for 1952 observations. A crystal of **3a** of dimensions 0.25 × 0.25 × 0.35 mm was placed on an Enraf–Nonius CAD4 automatic diffractometer with graphite monochromatized Mo K$\alpha$ radiation. The cell parameters were obtained by fitting a set of 25 high-$\Theta$ reflections. The data collection ($2\Theta_{max} = 50°$; scan $\omega$–$2\Theta = 1$; $t_{max} = 60$ s; $hkl$ range $h = 0$–$26$, $k = 0$–$19$, $l = 0$–$11$; intensity controls without appreciable decay (0.1%)) gave 3081 reflections of which 1952 were independent with $I > 3\sigma(I)$. After Lorentz and polarization corrections, the structure was solved by direct methods which revealed many non-hydrogen atoms. The remaining non-hydrogen atoms of the structure were found after successive scale-factor refinements and Fourier differences. After isotropic refinement ($R = 0.065$) and then anisotropic refinement ($R = 0.041$), the hydrogen atoms were set in theoretical positions. The whole structure was refined by the full-matrix least-squares techniques (use of $F$ magnitude; $x$, $y$, $z$, $\beta_{ij}$ for Fe, P, O and C atoms and $x, y, z$ fixed for H atoms; 439 variables and 1952 observations; $w = 1/\sigma(F_o)^2 = [\sigma^2(I) + (0.04F_o^2)^2]^{-1/2})$ with the resulting $R = 0.039$, $R_w = 0.038$ and $S_w = 1.236$ (residual $\Delta\rho \leq 0.38$ electrons Å$^{-3}$; $\Delta/\sigma = 0.14$). Atomic scattering factors were taken from [21]. All the calculations were performed on a Digital Micro VAX 3100 computer with the MOLEN package [22].

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