Phosphaferrocenes as Phosphorus Ligands. Crystal and Molecular Structure of Tetracarbonyl(3,4-dimethylphosphaferrocene-*P*)iron

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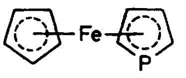
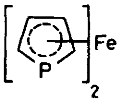
The structure of the title compound shows that the phospholyl ring remains fully aromatic even after phosphorus (*P*-) complexation of phosphaferrocene: the three C–C bond lengths are almost equal with a mean value of 1.411(3) Å and the P–C bonds are very short: 1.739(3) Å. The Fe ··· C_4P and Fe ··· C_5H_5 distances increase upon *P*-complexation and the configuration of the sandwich is staggered whereas it was eclipsed in free phosphaferrocene. The geometry of the P→Fe(CO)$_4$ moiety is a classical trigonal bipyramid with apical phosphorus. The P–Fe distance is short, 2.211(1) Å, indicating that the bond is strong and that phosphaferrocene is a strong ligand even though it is a weak donor on the basis of the i.r. data.

PHOSPHAFERROCENES (1),[1-3] together with 1,1′-diphosphaferrocenes (2)[4,5] and phosphacymantrenes (3),[3,6-8] are the only known phosphorus–carbon heterocycles displaying a classical ' aromatic ' chemistry; indeed it is possible to perform various carbon (*C*-) electrophilic substitutions on these species. This ' aromaticity ' has some striking structural consequences which are clearly evident when comparing the *X*-ray structures of these compounds[1,5,7] with those of free phospholes (4).



(1)



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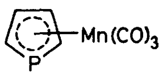

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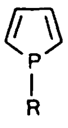
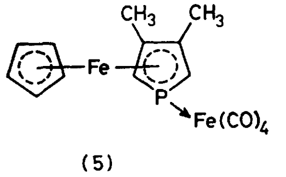


(4)

On the other hand, it has been shown that phosphametallocenes could give *P*-complexes as the more classical phosphines do.[8,9] Thus, it was very interesting to study the structure of one such *P*-complex in order to get an idea of the consequences of complexation on the aromaticity of these species and an idea of the strength and nature of the phosphorus–metal bond. This last aspect can be related to the general controversy on the existence of π back bonding in *P*-complexes[10-12] since, on the basis of chemical and spectroscopic observations, it appears that the phosphorus atom of phosphametallocenes displays strong electron-accepting properties[7] and thus is *a priori* especially adapted to back donation. Hereafter we report our results on an *X*-ray structural study of tetracarbonyl(3,4-dimethylphosphaferrocene-*P*)iron, (5), the synthesis of which has been described previously.[9]



(5)

EXPERIMENTAL

Suitable single-crystal fragments of [Fe(η-C_5H_5){η-Me_2-C_4H_2P–Fe(CO)$_4$}] (5) were extracted from the vessel containing the crude product, since all recrystallization attempts failed.

A systematic search in reciprocal space using a Philips PW1100/16 automatic diffractometer and precession camera photographs show that crystals of (5) belong to the monoclinic system and that the space group is $P2_1/n$.

The unit-cell dimensions, and their standard deviations, were refined at room temperature (20 ± 2 °C) with Mo-K_α radiation (λ = 0.709 30 Å) using the method outlined by Busing and Levy for a four-circle diffractometer.[13] The experimental density was measured by flotation in an aqueous KI solution. Final crystal data are: $C_{15}H_{13}Fe_2O_4P$, M = 400, a = 17.679(3), b = 9.884(2), c = 9.285(2) Å, β = 91.64(2)°, U = 1 622 Å^3, D_m = 1.63 ± 0.02 g cm^{-3}, Z = 4, D_c = 1.63 g cm^{-3}, $F(000)$ = 808, space group $P2_1/n$ (C^5_{2h}), μ = 19.44 cm^{-1}.

A nearly spherical crystal of mean diameter 0.180(5) mm was sealed in a Lindemann glass capillary and mounted on a rotation-free goniometer head.† All quantitative data were obtained from a Picker FACS-1 four-circle diffractometer controlled by a PDP8/A computer using graphite-monochromated Mo-K_α radiation. The monochromator was set at an angle of 11.68°. The refined parameters listed above and the orientation matrix were obtained using standard Picker routines. Intensity data were collected using the θ—2θ flying step-scan technique with a scan rate of 2° min^{-1}, a step-width of 0.05°, and a scan width of 1.7° + (Mo-K_{α_1,α_2} splitting). Each reflection was measured only if $\delta^2(I)/I$ of a prescan was less than 5. Attenuation filters were inserted whenever the scan count exceeded 7 000 counts s^{-1}. In order to improve measurements of weak reflections due to poor diffraction quality, all *hkl*, *hkl* and their equivalent $\bar{h}kl$, and $\bar{h}kl$ reflections within $2 < \theta < 32.5°$ were recorded. The step-scan data were reduced to intensities and standard deviations using the Lehmann and Larsen algorithm.[14] The intensities of three standard reflections were monitored throughout the data collection period at intervals of 2 h; their mean intensities dropped by 6% and a time-dependent linear

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† Currently used following an original design of W. Petter, E.T.H., Zürich, personal communication.

decay correction was therefore applied. Equivalent reflections were averaged using the program DSORTH;[15] factors $R = \Sigma|I_o - \bar{I}|/\Sigma\bar{I}$ and $R' = \Sigma|wI - w\bar{I}|/\Sigma wI$ with $w = 1/\sigma^2(I)$ were 0.012 and 0.015 respectively. After averaging of equivalents, an independent set of 1 975 reflections with $I > 3\sigma(I)$ was corrected for Lorentz and polarization factors. Spherical absorption corrections were applied;[16] the transmission factor was between 0.775 8 and 0.777 8.

For all subsequent computations the Enraf–Nonius SDP/V16 package[17] was used on a PDP 11/60 computer.

The structure was solved using direct methods. The most probable set of Multan[18] was the correct one and the Fourier computed with these phases permitted the location of all non-hydrogen atoms.

A difference map computed at the end of isotropic refinement revealed electron-density concentrations close to the calculated positions for hydrogen atoms (C–H = 0.95 Å). These were included in all subsequent calculations with isotropic temperature factors of 5 Å^2, but not refined. The final difference map shows no maxima greater than 0.04 e Å^{-3}.

Refinement converged to $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $R' = (\Sigma w||F_o| - |F_c||^2/\Sigma w|F_o|^2)^{\frac{1}{2}}$ of 0.028 and 0.039 respectively. The standard deviation of unit weight observation was 1.07 with a fudge factor $p = 0.07$ in $\delta^2(F^2) = \sigma^2(\text{count}) + (pF^2)^2$.

TABLE 1

Positional parameters with their estimated standard deviations in parentheses

Atom	*X*	*Y*	*Z*
Fe(1)	0.137 69(3)	0.355 11(5)	0.132 66(5)
Fe(2)	0.464 34(3)	0.403 08(5)	0.759 45(5)
P	0.048 08(5)	0.197 17(9)	0.123 0(1)
O(1)	0.157 8(2)	0.831 4(4)	−0.054 7(4)
O(2)	0.063 7(2)	0.860 9(3)	0.244 7(4)
O(3)	−0.141 0(2)	−0.065 9(4)	0.420 2(5)
O(4)	−0.012 2(3)	0.299 7(4)	0.486 8(4)
C(1)	0.044 7(2)	0.324 5(4)	−0.006 2(4)
C(2)	0.112 5(2)	0.330 5(4)	−0.083 9(4)
C(3)	0.166 0(2)	0.232 8(4)	−0.036 3(4)
C(4)	0.139 5(2)	0.153 1(3)	0.076 6(4)
C(7)	−0.103 9(2)	0.139 3(4)	0.130 8(5)
C(8)	0.024 5(2)	−0.047 8(4)	0.249 3(4)
C(9)	−0.099 3(2)	−0.000 2(5)	0.360 7(5)
C(10)	−0.021 4(3)	0.220 6(5)	0.398 7(5)
C(5)	0.123 4(3)	0.430 1(5)	−0.204 9(5)
C(6)	0.243 0(3)	0.213 2(6)	−0.098 9(6)
C(11)	0.125 1(3)	0.476 3(6)	0.306 4(6)
C(12)	0.147 8(3)	0.552 7(4)	0.194 9(6)
C(13)	0.215 7(3)	0.503 7(5)	0.153 5(6)
C(14)	0.233 6(3)	0.395 2(5)	0.246 6(7)
C(15)	0.176 7(4)	0.383 5(6)	0.337 2(6)
H(1)	0.002 0(0)	0.384 6(0)	−0.022 7(0)
H(4)	0.168 9(0)	0.082 4(0)	0.122 1(0)
H(5)	0.172 9(0)	0.419 5(0)	−0.244 2(0)
H(52)	0.086 7(0)	0.418 0(0)	−0.290 5(0)
H(53)	0.119 9(0)	0.522 0(0)	−0.170 7(0)
H(61)	0.249 7(0)	0.288 6(0)	−0.176 4(0)
H(62)	0.282 2(0)	0.229 7(0)	−0.029 2(0)
H(63)	0.248 4(0)	0.127 4(0)	−0.140 2(0)
H(11)	0.077 0(0)	0.493 6(0)	0.354 2(0)
H(12)	0.121 7(0)	0.628 2(0)	0.146 3(0)
H(13)	0.244 7(0)	0.538 3(0)	0.075 8(0)
H(14)	0.278 0(0)	0.341 2(0)	0.247 9(0)
H(15)	0.175 7(0)	0.313 4(0)	0.410 2(0)

Table 1 gives the atomic co-ordinates with their standard deviations. Observed and calculated structure factors and thermal parameters are listed in Supplementary Publication No. SUP 22848 (12 pp.).*

DISCUSSION

In the crystal state, (5) consists of discrete molecules in which one iron atom is sandwiched between a cyclopentadienyl ring and a phospholyl ring; the second iron atom is bonded to four carbonyl groups and the phosphorus atom of the phospholyl ring. The individual molecules are linked only by van der Waals type interactions and hydrogen bonds. Table 2 gives the shortest intermolecular distances.

TABLE 2

Intermolecular contacts less than 3.2 Å

$C(1) \cdots H(1)^{I}$	3.01	$C(13) \cdots H(14)^{V}$	2.97
$O(1) \cdots H(12)^{I}$	2.83	$C(14) \cdots H(4)^{V}$	2.78
$O(1) \cdots H(4)^{II}$	2.97	$H(1) \cdots H(4)^{I}$	2.32
$O(1) \cdots H(14)^{III}$	3.03	$H(1) \cdots H(1)^{I}$	2.45
$O(1) \cdots H(14)^{IV}$	3.06	$H(1) \cdots H(12)^{I}$	3.00
$O(2) \cdots H(6)^{V}$	2.80	$H(1) \cdots H(53)^{I}$	3.01
$O(2) \cdots H(14)^{VI}$	3.11	$H(4) \cdots C(1)^{IX}$	2.82
$O(3) \cdots O(4)^{VII}$	3.53	$H(4) \cdots H(14)^{IX}$	3.18
$O(3) \cdots H(13)^{VIII}$	2.98	$H(51) \cdots N(13)^{X}$	2.72
$O(4) \cdots H(15)^{VI}$	2.78	$H(52) \cdots H(11)^{I}$	3.08
$O(4) \cdots H(11)^{VI}$	3.11	$H(53) \cdots H(6)^{X}$	3.13
$C(12) \cdots O(2)^{I}$	3.13	$H(13) \cdots H(15)^{V}$	3.06
$C(12) \cdots H(1)^{V}$	3.17	$H(14) \cdots H(12)^{XI}$	2.90

Roman numeral superscripts refer to the following co-ordinate transformations:

I $-x, -y + 1, -z$
II $-x, -y, -z$
III $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$
IV $x + \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$
V $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$
VI $-x, 1 - y, 1 - z$
VII $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$
VIII $-x, -y, 1 + z$
IX $\frac{3}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$
X $\frac{1}{2} - x, \frac{1}{2} + y, -\frac{1}{2} - z$
XI $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$

Figure 1 shows a molecule of (5) (without the hydrogen atoms) together with the labelling scheme used.† Atoms are represented by their thermal motion ellipsoids scaled to enclose 50% of the electron density. Figure 2 shows the projection of a molecule of (5) on the plane defined by atoms P, C(1), and C(4). Table 3 gives significant

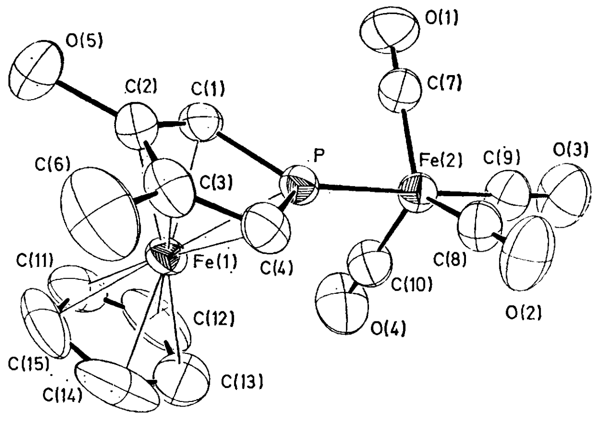

FIGURE 1 Structure of complex (5) showing the atom labelling. Hydrogen atoms are omitted for clarity

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* For details see Notices to Authors No. 7, *J.C.S. Dalton*, 1979, Index issue.

† Drawings are done using ORTEP II, C. K. Johnson, Report ORNL 3794, Oak Ridge, Tennessee, 1965.

bond lengths and angles and Table 4 gives the least-squares planes of interest.

Within experimental error the three C–C bonds of the phospholyl ring in (5) are equal [mean: 1.411(3) Å] and of similar length to the corresponding C–C bonds in the starting phosphaferrocene[1] [mean : 1.408(4) Å]. On the contrary, the C_4P ring is notably less bent around the C(1)–C(4) axis in (5) than in the free phosphaferrocene; the dihedral angle between the mean planes through C(1), C(2), C(3), C(4) and P, C(1), C(4) is 0.9° in (5) [the phosphorus atom lies out of the C(1), C(2), C(3), C(4) mean plane by 0.018(1) Å away from the iron atom] compared to 1.9° in phosphaferrocene.[1] This is partly due to a probable increase of the Fe(1) · · · C_4P ring distance in (5) : 1.635(1) against 1.625(1) Å in phosphaferrocene. Thus the steric bulks of the phosphorus and iron [Fe(1)] atoms cause less distortion in the phospholyl ring. Furthermore, the mean intracyclic P–C bond is shorter in (5) than in phosphaferrocene [1.735(3) against 1.763(3) Å]. Since the Fe(1)–P bond length decreases in the same way [2.224(1) Å in (5) against 2.276(1) Å in phosphaferrocene] this probably indicates a change in the covalent radius of phosphorus upon complexation rather than an increase of the cyclic delocalization within the phospholyl ring. However all these data clearly show that the phospholyl ring remains fully aromatic in (5) and this has two interesting consequences: (*i*) the lone pair on phosphorus does not participate significantly to the cyclic delocalization within the C_4P ring of free phosphaferrocenes; and (*ii*) it remains probably possible to perform *C*-electrophilic substitutions on the phospholyl ring of phosphaferrocenes even after *P*-complexation.

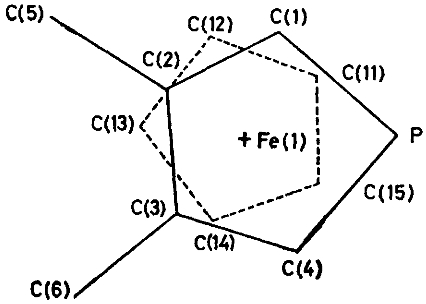

FIGURE 2 Projection of (5) on the plane defined by P, C(1), and C(4)

Nevertheless, *P*-complexation causes several distortions in the structure of the sandwich itself. It has already been noted that the Fe(1) · · · C_4P distance increases upon complexation and so does the Fe(1) · · · C_5H_5 distance : 1.664(1) Å in (5) against 1.655(1) Å in free phosphaferrocene.[1] In the same way, the dihedral angle between the planar cyclopentadienyl ring and the planar four-membered carbon moiety of the phospholyl group increases from 3.18° in phosphaferrocene[1] to 5.3° in (5). Finally, the conformation of the phosphaferrocene system is staggered in (5) (see Figure 2) whereas it is eclipsed in free phosphaferrocene. These rather striking changes are not easily explained by simple electronic or steric reasons.

TABLE 3

Bond lengths (Å) and angles (°)

Fe(1)–P	2.224(1)	
Fe(1)–C(2)	2.082(3)	
Fe(1)–C(2)	2.062(4)	Mean: 2.065(2)
Fe(1)–C(3)	2.053(4)	
Fe(1)–C(4)	2.064(3)	
Fe(1)–C(11)	2.027(5)	
Fe(1)–C(12)	2.043(4)	
Fe(1)–C(13)	2.020(4)	Mean: 2.024(2)
Fe(1)–C(14)	2.012(5)	
Fe(1)–C(15)	2.021(5)	
C(1)–C(2)	1.417(5)	Mean: 1.411(3)
C(2)–C(3)	1.414(6)	
C(3)–C(4)	1.402(5)	
C(2)–C(5)	1.510(6)	Mean: 1.509(4)
C(3)–C(6)	1.509(6)	
P–C(1)	1.738(4)	Mean: 1.739(3)
P–C(4)	1.741(4)	
C(11)–C(12)	1.352(10)	
C(12)–C(13)	1.361(9)	Mean: 1.354(4)
C(13)–C(14)	1.408(10)	
C(14)–C(15)	1.334(11)	
C(15)–C(11)	1.319(10)	
Fe(2)–C(7)	1.786(4)	
Fe(2)–C(8)	1.786(4)	Mean: 1.783(2)
Fe(2)–C(9)	1.770(5)	
Fe(2)–C(10)	1.792(4)	
Fe(2)–P	2.211(1)	
C(7)–O(1)	1.138(5)	
C(8)–O(2)	1.141(5)	Mean: 1.139(2)
C(9)–O(3)	1.137(5)	
C(10)–O(4)	1.140(5)	
C(1)–P–C(4)	91.4(2)	
P–C(1)–C(2)	111.6(2)	
P–C(4)–C(3)	112.0(3)	
C(1)–C(2)–C(3)	112.3(3)	
C(4)–C(3)–C(2)	112.5(3)	
C(1)–C(2)–C(5)	122.2(4)	
C(4)–C(3)–C(6)	122.6(4)	
C(2)–C(3)–C(6)	124.7(4)	
C(3)–C(2)–C(5)	125.4(4)	
Fe(2)–P–C(1)	125.2(1)	
Fe(2)–P–C(4)	132.2(2)	
C(11)–C(12)–C(13)	107.5(5)	
C(12)–C(13)–C(14)	106.3(5)	
C(13)–C(14)–C(15)	107.0(5)	
C(14)–C(15)–C(11)	109.4(6)	
C(15)–C(11)–C(12)	109.6(6)	
Fe(2)–C(7)–O(1)	176.4(4)	
Fe(2)–C(8)–O(2)	178.7(4)	
Fe(2)–C(9)–O(3)	176.8(5)	
Fe(2)–C(10)–O(4)	179.6(4)	
P–Fe(2)–C(13)	173.7(7)	

The last point of interest deals with the structure of the $P \rightarrow Fe(CO)_4$ moiety. As in classical $[Fe(CO)_4L]$ com-

TABLE 4

Least-squares planes

Plane (1): C(1), C(2), C(3), C(4)
$-0.3617x - 0.6542y - 0.6642z = 2.3467$ ($\chi^2 = 0$)*
[P 0.018(1), Fe(1) −1.635(1)]
Plane (2): P, C(1), C(4)
$-0.3518x - 0.6492y - 0.6743z = 2.3223$ ($\chi^2 = 0$)
[Fe(1) −1.631(1), Fe(2) 0.323(1)]
Plane (3): Cyclopentadienyl ring
$-0.4466x - 0.6294y - 0.6359z = 5.7279$ ($\chi^2 = 2$)
[Fe(1) 1.664(1)]
Dihedral angles (°) (1)—(2) 0.9, (1)—(3) 5.3

* $\chi^2 = \sum_i \left[\frac{d_i^2}{\sigma^2(d_i)}\right]$, d_i is the distance of the *i*th atom to the mean plane.

plexes (L = phosphine group, PR_3) the co-ordination polyhedron of atom Fe(2) is a trigonal bipyramid with three carbonyl groups in the equatorial plane; the fourth carbonyl is axial as is the phosphorus atom. The mean value of the Fe(2)–C distances is 1.783(2) Å and the Fe–P distance is 2.211(1) Å. Other data are given in Table 3.

In conclusion, the Fe(2) atom lies practically in the P, C(1), C(4) mean plane as indicated by mean plane (2) in Table 4 and by the sum of the C(1)–P–C(4), Fe(2)–P–C(1), and Fe(2)–P–C(4) angles (358.8°). Among all these data the most significant is the value of the Fe(2)–P distance: 2.211(1) Å. A comparison with the known P–$Fe(CO)_4$ bond lengths shows that this bond is rather short in (5): $HPh_2P\rightarrow Fe(CO)_4$ 2.237(2) Å;[19] $Bu^t{}_3P\rightarrow Fe(CO)_4$ 2.364(1) Å;[20] $\overline{\mathrm{CF_2CF_2C(AsMe_2)=C}}$-$(PPh_2)\rightarrow Fe(CO)_4$ 2.224(3);[21] $Me_4P_2\rightarrow Fe(CO)_4$ 2.260 (5) Å.[22] Thus, it seems that phosphaferrocene is a rather stronger ligand for the $Fe(CO)_4$ moiety than more classical phosphines. Nevertheless Table 5 clearly indicates that phosphaferrocene is a poorer donor than ordinary phosphines. It seems difficult to explain these two results without using the π back-bonding concept.

TABLE 5

Infrared data of various $[Fe(CO)_4L]$ complexes

L	ν(CO)/cm^{-1}			Solvent	Ref.
PPh_2H	2 056	1 984	1 953—1 946	Hexane	*a*
Phosphaferrocene [complex (5)]	2 060	1 985	1 957—1 951	Decalin	*b*
$PBu^t{}_3$	2 045	1 968	1 931	Pentane	*c*
PEt_3	2 048.6	1 974.5	1 935.7	Hexadecane	*d*
PPh_3	2 050.7	1 977.8	1 944.5	Hexadecane	*d*
$P(OMe)_3$	2 062.5	1 991.0	1 961.0—1 949.7	Hexadecane	*d*

[a] J. G. Smith and D. T. Thompson, *J. Chem. Soc.* (*A*), 1967, 1964. [b] Ref. 9. [c] H. Schumann, L. Rösch, H. J. Kroth, H. Neumann, and B. Neudert, *Chem. Ber.*, 1975, **108**, 2487. [d] A. Reckziegel and M. Bigorgne, *J. Organometallic Chem.*, 1965, **3**, 341.

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