metal-organic papers

Pentacarbonyl(3,3′,4,4′-tetramethyl-1,1′-diphosphaferrocene-κP)tungsten(0)

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In the title compound, [WFe(C_6H_8P)$_2$(CO)$_5$], the pholpholyl rings in the diphosphaferrocene moiety are in a skew conformation. The intramolecular distance between the P atoms of 3.553 (4) Å indicates a secondary bonding interaction.

Comment

This work forms part of our studies of substituted diphosphaferrocenes, their stereochemistry and structural analyses (Sieroń *et al.*, 1998; Zakrzewski *et al.*, 1998, 2002). The title compound, (I), represents the first structurally characterized complex with a P-monosubstituted diphosphaferrocene. The crystal structure of (I) is shown in Fig. 1. The phospholyl rings are planar, with a dihedral angle of 2.65 (9)° between the rings. The corresponding bond distances and angles are equal within experimental error, except for slight differences in the P1—C5/P11—C15 bond lengths and C2—P1—C5/C12—P11—C15 angles (see Table 1).

(I)
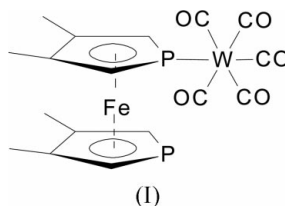
The conformation of the 1,1′-diphosphaferrocene moiety is usually described as a function of the dihedral angle θ, defined as the angle between the planes normal to each phospholyl ring that contain both Fe and P atoms (Ashe & Al-Ahmad, 1996). Two P atoms are eclipsed at $\theta = 0°$, whereas in the antiperiplanar conformation $\theta = 180°$. The reported structure shows a skew conformation of the phospholyl rings with $\theta = 38.3$ (1)°. The intramolecular distance between the P atoms is 3.553 (4) Å, which indicates a secondary bonding interaction (Alcock, 1972, 1990).

The W atom is not coplanar with the attached cyclopentadienyl ring and deviates from its plane, with a C3—C2—P1—W torsion angle of 164.0 (2)°. The W—P1 distance is 2.464 (2) Å, and the W—C21 bond length of 2.002 (5) Å *trans* to W—P1 is slightly shorter than the other four W—C bond lengths, which range from 2.031 (5) to 2.050 (5) Å.

The absence of hydrogen bonds and π-stacking interactions suggests that the crystal packing is determined only by van der Waals forces.

Experimental

Compound (I) was synthesized according to the procedure reported by Deschamps *et al.* (1984). Crystals suitable for X-ray analysis were

obtained by slow diffusion of pentane into a solution of compound (I) in dichloromethane.

Crystal data

[WFe(C_6H_8P)$_2$(CO)$_5$]	$D_x = 2.009$ Mg m^{-3}
$M_r = 601.93$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 48
$a = 13.279$ (13) Å	reflections
$b = 10.857$ (9) Å	$\theta = 3.6–14.0°$
$c = 14.061$ (12) Å	$\mu = 6.69$ mm^{-1}
$\beta = 101.02$ (7)°	$T = 293$ K
$V = 1990$ (3) Å^3	Prism, red
$Z = 4$	$0.46 \times 0.13 \times 0.11$ mm

Data collection

Siemens $P3$ diffractometer	$R_{int} = 0.018$
ω–2θ scans	$\theta_{max} = 27.5°$
Absorption correction: ψ scan	$h = -17 \rightarrow 16$
(North et al., 1968)	$k = 0 \rightarrow 14$
$T_{min} = 0.236$, $T_{max} = 0.480$	$l = 0 \rightarrow 18$
4752 measured reflections	3 standard reflections
4567 independent reflections	every 100 reflections
3802 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2$
$R[F^2 >2\sigma(F^2)] = 0.026$	$+ 1.1109P]$
$wR(F^2) = 0.059$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
4567 reflections	$\Delta\rho_{max} = 0.71$ e Å^{-3}
240 parameters	$\Delta\rho_{min} = -0.67$ e Å^{-3}
H-atom parameters constrained	Extinction correction: $SHELXL$97
	Extinction coefficient: 0.00038 (9)

Table 1
Selected geometric parameters (Å, °).

P1···P11	3.553 (4)	P1—C5	1.746 (4)
W—P1	2.464 (2)	P11—C12	1.765 (5)
W—C21	2.002 (5)	P11—C15	1.766 (5)
W—C22	2.040 (5)	O1—C21	1.137 (5)
W—C23	2.031 (5)	O2—C22	1.134 (6)
W—C24	2.048 (5)	O3—C23	1.142 (6)
W—C25	2.050 (5)	O4—C24	1.134 (6)
P1—C2	1.755 (4)	O5—C25	1.125 (5)
P1—W—C21	177.58 (13)	C22—W—C23	90.6 (2)
P1—W—C22	89.34 (13)	C22—W—C24	179.15 (17)
P1—W—C23	88.68 (14)	C22—W—C25	90.4 (2)
P1—W—C24	91.48 (13)	C23—W—C24	89.2 (2)
P1—W—C25	85.55 (14)	C23—W—C25	174.12 (17)
C21—W—C22	88.36 (18)	C24—W—C25	89.9 (2)
C21—W—C23	92.10 (19)	C2—P1—C5	90.61 (19)
C21—W—C24	90.82 (19)	C12—P11—C15	88.2 (2)
C21—W—C25	93.72 (18)		

All H atoms were located in difference Fourier syntheses but were positioned with ideal geometry (C—H = 0.93 Å) and refined with fixed isotropic displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)] using the riding model. The positions of the methyl H atoms were idealized

Figure 1
View of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 40% probability level.

(C—H = 0.96 Å), then refined with fixed isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(C)] as rigid groups allowed to rotate but not tip.

Data collection: $P3$ Software (Siemens, 1993); cell refinement: $P3$ Software; data reduction: $XDISK$ in $SHELXTL/PC$ (Sheldrick, 1990); program(s) used to solve structure: $SHELXS$97 (Sheldrick, 1997); program(s) used to refine structure: $SHELXL$97 (Sheldrick, 1997); molecular graphics: XP in $SHELXTL/PC$; software used to prepare material for publication: $PLATON$ (Spek, 2003).

References

Alcock, N. W. (1972). *Adv. Inorg. Chem. Radiochem.* **15**, 1–58.
Alcock, N. W. (1990). *Bonding and Structure: Structural Principles in Inorganic and Organic Chemistry.* New York: Ellis Horwood.
Ashe, A. J. III & Al-Ahmad, S. (1996). *Adv. Organomet. Chem.* **39**, 325–353.
Deschamps, B., Mathey, F. & Fisher, J. H. (1984). *Inorg. Chem.* **23**, 3455–3462.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Siemens (1993). *P3 Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sieroń, L., Tosik, A. & Bukowska-Strzyżewska, M. (1998). *J. Chem. Crystallogr.* **28**, 621–628.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Zakrzewski, J., Kłys, A., Bukowska-Strzyżewska, M. & Tosik, A. (1998). *Organometallics*, **17**, 5880–5886.
Zakrzewski, J., Kłys, A., Bukowska-Strzyżewska, M. & Tosik, A. (2002). *J. Organomet. Chem.* **645**, 268–273.