Di-μ-diphenylphosphido-bis[tricarbonyliron(II)] dichloromethane solvate

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.132
Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $[Fe_2(C_{12}H_{10}P)_2(CO)_6]\cdot CH_2Cl_2$, the complex molecule lies across a mirror plane. Two diphosphide ligands bridge two $(CO)_3Fe$ fragments to form a dinuclear complex, in which the Fe—Fe distance is 2.6701 (6) Å. Two aromatic rings within the same molecule interact by face-to-face π–π stacking, with a centroid-to-centroid distance of 3.601 (2) Å.
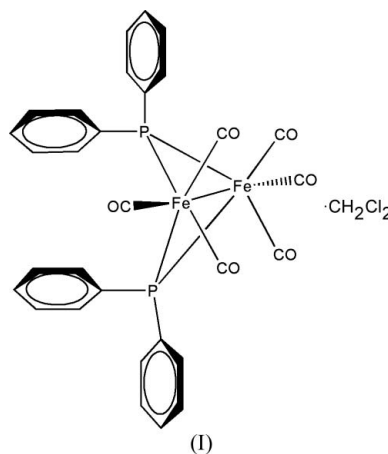
Comment

Di-μ-diphenylphosphido-bis(tricarbonyliron) was first prepared by Job *et al.* (1966) from pentacarbonyl iron and tetraphenylphosphine at 493 K. Collman *et al.* (1977) developed an alternative synthesis, which entails the addition of diphenylchlorophosphine to disodium octacarbonyldiferrate. We report here the crystal structure of the dichloromethane solvate, (I).

(I)

The molecular structure of (I) is shown in Fig. 1. The complex molecule lies across a mirror plane. Two diphosphide ligands bridge two $(CO)_3Fe$ fragments to form a dinuclear complex, in which the Fe—Fe distance is 2.6701 (6) Å. The P2—Fe—Fe^i—P1 ring adopts a butterfly arrangement with the P-donor in the wingtip positions [symmetry code: (i) $x, \frac{1}{2} - y, z$]. The dihedral angle between the two P—Fe—Fe^i planes is 104.8 (4)°. The Fe—P bond distances and C—P—C bond angles are comparable to those found in similar molecules (Ginsburg *et al.*, 1979).

Two aromatic rings within the same molecule interact by face-to-face π–π stacking, the centroid-to-centroid distance being 3.601 (2) Å. The supramolecular structure of (I) (Fig. 2) is achieved by three edge-to-face interactions per complex molecule: H4···Ph^{ii} = 2.65 Å, C4···Ph^{ii} = 3.521 (6) Å and C4—H4···Ph^{ii} = 157°; H30A···Ph^{iii} = 2.73 Å, C30···Ph^{iii} =
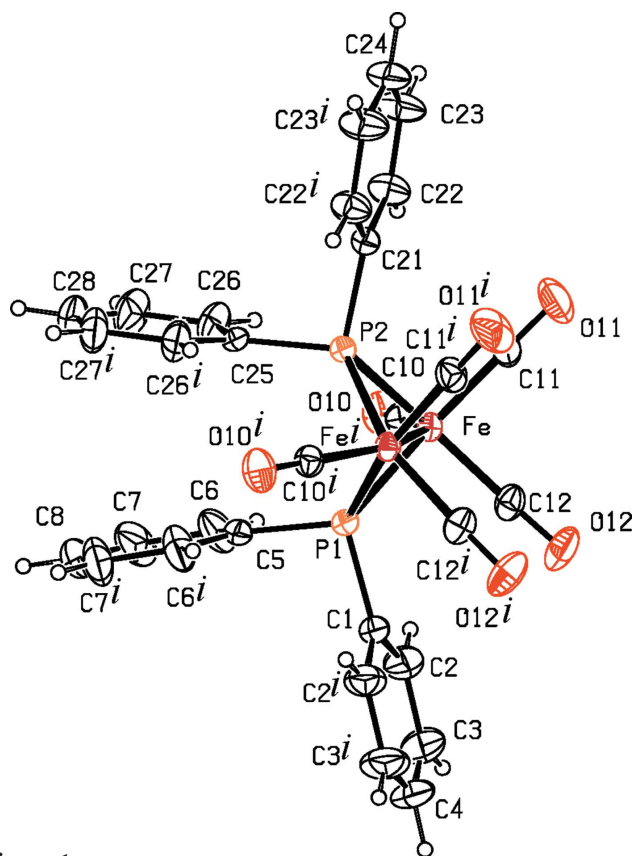
Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids. The dichloromethane solvent has been omitted. [Symmetry code: (i) $x, \frac{1}{2} - y, z$.]
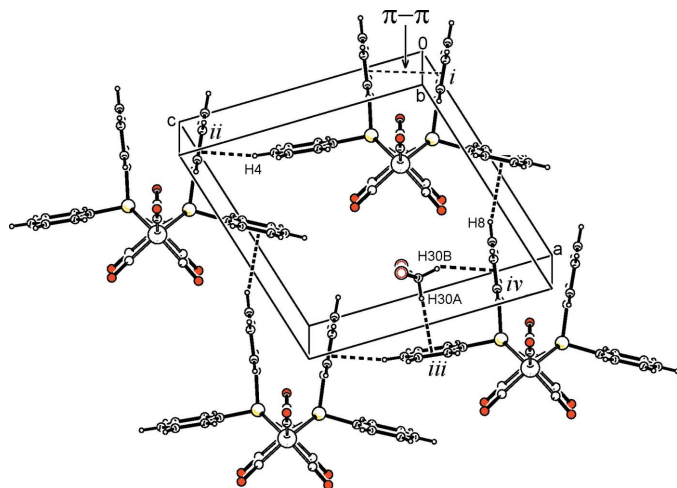

Figure 2
The supramolecular structure of (I), showing face-to-face and edge-to-face π–π stackings (dashed lines). Only one solvent molecule is shown. [Symmetry codes: (i) $x, \frac{1}{2} - y, z$; (ii) $x, y, 1 + z$; (iii) $1 + x, y, z$; (iv) $1 + x, \frac{1}{2} - y, z$.]

3.521 (6) Å and C30—H30A···Ph^{iii} = 157°; H30B···Ph^{iv} = 2.57 Å, C30···Ph^{iv} = 3.610 (10) Å and C30—H30B···Ph^{iv} = 176° [symmetry codes: (ii) $x, y, 1 + z$; (iii) $1 + x, y, z$; (iv) $1 + x, \frac{1}{2} - y, z$] (Ph1, Ph2 and Ph3 are rings C1–C4/C2^{i}/C3^{i}, C5–C8/C6^{i}/C7^{i} and C25–C28/C26^{i}/C27^{i}, respectively).

Experimental

Crystal data

[Fe$_2$(C$_{12}$H$_{10}$P)$_2$(CO)$_6$]·CH$_2$Cl$_2$	$D_x = 1.494$ Mg m^{-3}
$M_r = 735.03$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/m$	Cell parameters from 600
$a = 11.3218 (11)$ Å	reflections
$b = 12.7214 (13)$ Å	$\theta = 20$–$25°$
$c = 11.8011 (12)$ Å	$\mu = 1.19$ mm^{-1}
$\beta = 105.976 (2)°$	$T = 293 (2)$ K
$V = 1634.1 (3)$ Å^3	Rectangular block, yellow
$Z = 2$	$0.22 \times 0.12 \times 0.09$ mm

Data collection

Bruker SMART area-detector diffractometer	2896 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.022$
Absorption correction: none	$\theta_{max} = 27.5°$
8071 measured reflections	$h = -14 \rightarrow 12$
3826 independent reflections	$k = -15 \rightarrow 16$
	$l = -7 \rightarrow 15$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0878P)^2]$
$wR(F^2) = 0.132$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3826 reflections	$\Delta\rho_{max} = 1.17$ e Å^{-3}
211 parameters	$\Delta\rho_{min} = -0.76$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe—P1	2.2250 (9)	P1—C1	1.821 (4)
Fe—P2	2.2200 (8)	P1—C5	1.825 (4)
Fe—C10	1.775 (3)	P2—C21	1.837 (4)
Fe—C11	1.792 (4)	P2—C25	1.825 (4)
Fe—C12	1.798 (3)	O10—C10	1.131 (3)
Fe—Fe^i	2.6071 (6)	O11—C11	1.128 (3)
Cl1—C30	1.627 (7)	O12—C12	1.134 (4)
P1—Fe—P2	79.84 (3)	Fe^i—Fe—C12	97.90 (8)
P1—Fe—C10	102.55 (8)	Fe—P1—C1	119.60 (11)
P1—Fe—C11	156.48 (12)	Fe—P1—C5	124.09 (8)
P1—Fe—C12	89.11 (11)	Fe—P1—Fe^i	71.73 (3)
Fe^i—Fe—P1	54.14 (2)	C1—P1—C5	98.69 (18)
Fe^i—Fe—P2	54.04 (2)	Fe^i—P1—C1	119.60 (11)
P2—Fe—C10	105.59 (8)	Fe^i—P1—C5	124.09 (8)
P2—Fe—C11	90.81 (12)	Fe—P2—C21	120.96 (9)
P2—Fe—C12	151.02 (9)	Fe—P2—C25	122.51 (9)
C10—Fe—C11	100.73 (14)	Fe—P2—Fe^i	71.91 (3)
C10—Fe—C12	102.93 (12)	C21—P2—C25	98.94 (16)
Fe^i—Fe—C10	148.53 (8)	Fe^i—P2—C21	120.96 (9)
C11—Fe—C12	88.87 (16)	Fe^i—P2—C25	122.51 (9)
Fe^i—Fe—C11	103.02 (12)		

Symmetry code: (i) $x, -y + \frac{1}{2}, z$.

All H atoms were positioned geometrically and refined using a riding model [C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C)]. The highest peak in the difference Fourier map is 0.93 Å from atom Cl1.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97* and *WinGX2003* (Farrugia, 1999).

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