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Dicarbonyl(η^5-pentamethylcyclopentadienyl)(triphenylphosphine)iron(II) hexafluorophosphate

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.176
Data-to-parameter ratio = 18.0

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

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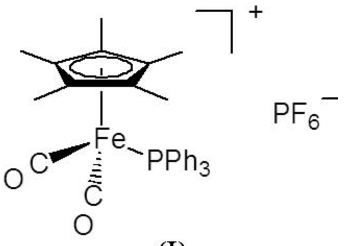
In the title compound, [Fe($C_{10}H_{15}$)($C_{18}H_{15}P$)(CO)$_2$]PF_6, the Fe^{II} atom is coordinated by two carbonyl groups, a triphenylphosphine ligand and a pentamethylcyclopentadienyl ligand in a typical piano-stool structure.

Comment

The crystal structure of [FeCp*(PPh$_3$)(CO)$_2$]PF$_6$ (Cp* is pentamethylcyclopentadienyl), (I), shows that the molecule adopts a three-legged piano-stool geometry: the Fe^{II} atom possesses one Cp* with an η^5-bonding fashion, two carbonyls and one PPh$_3$ ligand (Fig. 1). An analogous crystal structure, *viz*. [FeCp(PPh$_3$)(CO)$_2$]PF$_6$ (Cp is cyclopentadienyl), has been reported by Janik *et al.* (1995). The Cp* ring is bonded to the Fe^{II} atom through five C atoms, with Fe—C distances in the normal range [2.109 (4)–2.154 (4) Å]. The Fe—P length [2.2519 (11) Å] is slightly longer than that of [FeCp(PPh$_3$)(CO)$_2$]PF$_6$ [2.243 (2) Å], while the others [Fe—CO = 1.773 (4) and 1.776 (4) Å; P—F = 1.528 (4)–1.575 (4) Å] are quite similar. The P—Fe—C angles [90.84 (13) and 92.64 (14)°] are smaller than those of [FeCp(PPh$_3$)(CO)$_2$]PF$_6$ [92.1 (2) and 92.8 (2)°] and the C—Fe—C angle [97.38 (18)°] is larger than that of [FeCp(PPh$_3$)(CO)$_2$]PF$_6$ [95.6 (3)°]. This may stem from steric repulsion between the methyl groups of the Cp* ring and the phenyl groups of the PPh$_3$ ligand.



(I)

Experimental

[FeCp*(PPh$_3$)(CO)$_2$]PF$_6$, (I), was prepared according to the literature method of Catheline & Astruc (1984). Single crystals were obtained by solvent diffusion at 253 K over a few days from a CH$_2$Cl$_2$ layer containing (I) and an overlayer of hexane.

Crystal data

[Fe($C_{10}H_{15}$)($C_{18}H_{15}P$)(CO)$_2$]·PF$_6$
M_r = 654.35
Orthorhombic, *Pbca*
a = 17.534 (1) Å
b = 15.616 (1) Å
c = 21.394 (2) Å
V = 5858.0 (6) Å^3
Z = 8
D_x = 1.484 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 11285 reflections
θ = 3.0–27.5°
μ = 0.69 mm^{-1}
T = 153.2 K
Block, yellow
0.30 × 0.13 × 0.08 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min} = 0.781$, $T_{\max} = 0.946$
45741 measured reflections
6674 independent reflections
4973 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.065$
$\theta_{\max} = 27.5°$
$h = -17 \rightarrow 22$
$k = -20 \rightarrow 16$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.176$
$S = 1.11$
6674 reflections
370 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0831P)^2 + 2.6059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.90$ e Å^{-3}
$\Delta\rho_{\min} = -0.53$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—C1	2.109 (4)	P2—F1	1.559 (4)
Fe1—C2	2.118 (4)	P2—F2	1.575 (4)
Fe1—C3	2.130 (4)	P2—F3	1.528 (4)
Fe1—C4	2.154 (4)	P2—F4	1.564 (5)
Fe1—C5	2.147 (4)	P2—F5	1.560 (4)
Fe1—C11	1.776 (4)	P2—F6	1.541 (4)
Fe1—C12	1.773 (4)	O1—C11	1.145 (5)
Fe1—P1	2.2519 (11)	O2—C12	1.146 (5)
C12—Fe1—C11	97.38 (18)	C12—Fe1—P1	92.64 (14)
C11—Fe1—P1	90.84 (13)		

H atoms were refined using a riding model, with C—H = 0.95 Å and with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = U_{\text{eq}}(\text{C})$].

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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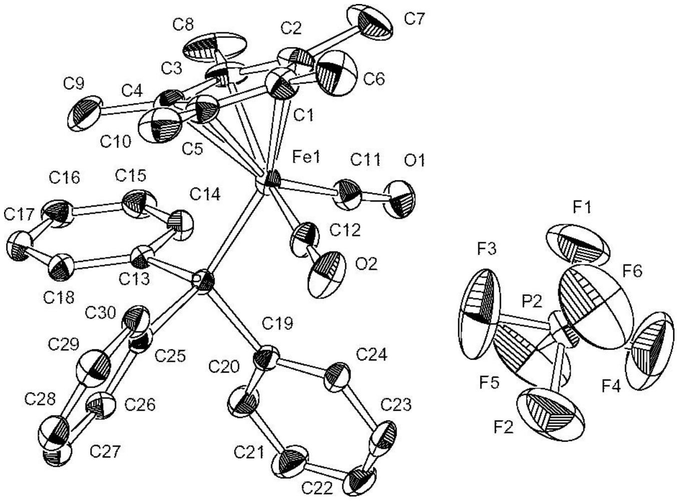

Figure 1
ORTEPII (Johson, 1976) drawing of the cation and anion of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

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