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Tetracarbonyl(tricyclohexylphosphine)iron(0)

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Key indicators

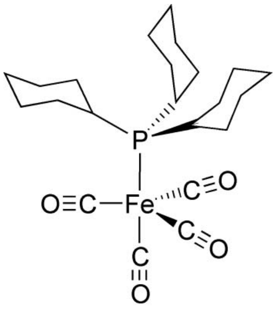
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.099
Data-to-parameter ratio = 12.4

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

The coordination of the Fe atom in the title compound, $[Fe(P(C_6H_{11})_3(CO)_4]$, is trigonal biypramidal, with the phosphine ligand adopting one of the axial positions.

Comment

In the course of our attempts to synthesize complexes of the general formula $[Fe(CO)_{3-x}(PR_3)_x(\eta^4\text{-1-azadiene})]$ (Berger *et al.*, 2000), we have produced several complexes $[Fe(CO)_{5-x}(PR_3)_x]$ (x = 1, 2) from $Fe_2(CO)_9$ and the corresponding phosphine ligands. One of these is the title compound, (I) (Fig. 1).



(I)

The Fe atom in (I) is coordinated in a trigonal–bipyramidal fashion with the phosphine ligand situated in one of the axial positions. Taking into account the standard uncertainties, the Fe—C bonds are virtually identical, with an average value of 1.776 (7) Å. The P—C bond lengths show expected values. The bond angles correspond to a nearly ideal trigonal–bipyramidal coordination mode for iron and to a nearly perfect tetrahedral environment for the P atom.

A search of the Cambridge Structural Database (Version 5.26, November 2004; Allen, 2002) yielded 38 compounds in which an Fe atom is coordinated by four terminal carbon monoxide ligands and by one phosphine ligand. All of these compounds show similar molecular structures to (I), with a trigonal–bipyramidal ligand arrangement and the phosphine adopting one of the axial positions. Nevertheless, there is one compound with a P—Fe—C(*trans*) bond angle of only 143.9° (Howell *et al.*, 1998), presumably due to a very bulky phosphine ligand with *ortho*-isopropylphenyl substituents. It is remarkable that the Fe—P bond length in (I) is almost the longest that has been reported until now. Only the above-mentioned derivative with a tris(*ortho*-isopropylphenyl)-phosphine (2.289 Å; Howell *et al.*, 1998), the complex with a tri-*tert*-butylphosphine ligand (2.363 Å; Pickardt *et al.*, 1976), the compound exhibiting a tris(*ortho*-methylphenyl)phos-

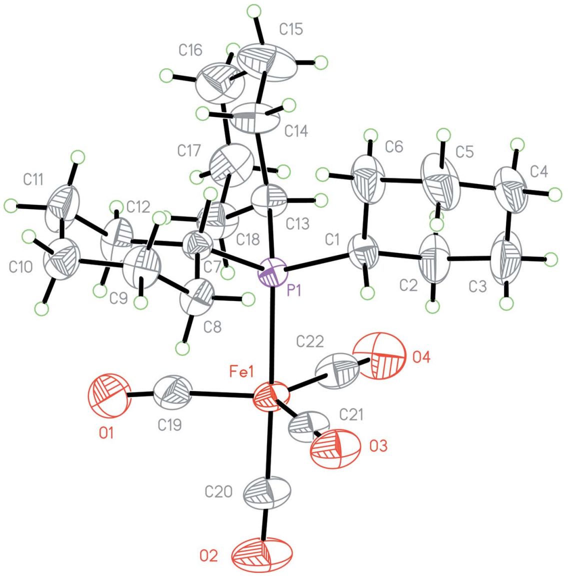


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

phine ligand (2.306 Å; Howell *et al.*, 1991, 1993) and a trinuclear iron–carbonyl compound in which a diphenylphosphine ligand bearing a coordinated methoxycarbonylethenyl group is attached to an $Fe(CO)_4$ group (2.283 Å; Montio *et al.*, 1990) show similar or even slightly longer Fe—P bonds.

Experimental

A 500 mg sample of $Fe_2(CO)_9$ (1.37 mmol) was stirred together with 770 mg (2.75 mmol) tricyclohexylphosphine in anhydrous *n*-heptane (20 ml) at 323 K. At the outset, the reaction mixture was a yellow suspension which slowly turned into a red solution. After all the material had dissolved, the *n*-heptane was evaporated *in vacuo*, the residue was dissolved in anhydrous dichloromethane and then filtered over a short silica column. With a mixture of light petroleum (b.p. 313–333 K) and dichloromethane (10:1), the title compound was eluted as a yellow band. Crystallization from light petroleum (b.p. 313–333 K) and dichloromethane (15:1) yielded crystalline material suitable for X-ray diffraction experiments.

Crystal data

$[Fe(C_{18}H_{33}P)(CO)_4]$
$M_r = 448.30$
Monoclinic, $P2_1/c$
$a = 13.8477$ (6) Å
$b = 9.9505$ (4) Å
$c = 17.0330$ (3) Å
$\beta = 94.432$ (3)°
$V = 2339.99$ (14) Å^3
$Z = 4$
$D_x = 1.273$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.74$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.3 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.826$, $T_{\max} = 0.910$ (expected range = 0.783–0.863)
6062 measured reflections
3142 independent reflections
2771 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\max} = 23.3°$
3 standard reflections
frequency: 60 min
intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.099$
$S = 1.03$
3142 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.9945P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—C19	1.769 (4)	P1—C13	1.863 (2)
Fe1—C20	1.774 (3)	P1—C1	1.871 (2)
Fe1—C22	1.776 (3)	C19—O1	1.147 (4)
Fe1—C21	1.785 (3)	C20—O2	1.148 (4)
Fe1—P1	2.2922 (7)	C21—O3	1.155 (3)
P1—C7	1.855 (2)	C22—O4	1.152 (4)
C19—Fe1—C20	86.14 (15)	C7—P1—C13	106.66 (11)
C19—Fe1—C22	118.09 (17)	C7—P1—C1	103.17 (11)
C20—Fe1—C22	92.69 (15)	C13—P1—C1	105.18 (12)
C19—Fe1—C21	124.03 (17)	C7—P1—Fe1	116.22 (8)
C20—Fe1—C21	90.18 (13)	C13—P1—Fe1	113.98 (8)
C22—Fe1—C21	117.86 (15)	C1—P1—Fe1	110.55 (9)
C19—Fe1—P1	92.29 (10)	O1—C19—Fe1	173.6 (3)
C20—Fe1—P1	178.14 (12)	O2—C20—Fe1	177.9 (3)
C22—Fe1—P1	88.93 (9)	O3—C21—Fe1	176.5 (3)
C21—Fe1—P1	89.88 (8)	O4—C22—Fe1	178.4 (3)

All H atoms were positioned with an idealized geometry (C—H = 0.97 and 0.98 Å), with $U_{\rm iso}(H) = 1.5U_{\rm eq}(C)$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *SET4* (de Boer *et al.*, 1984); data reduction: *MolEN* (Enraf–Nonius, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1990); software used to prepare material for publication: *SHELXL97*.

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