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Dichlorobis(triphenylphosphine)iron(II)

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In the title compound, $[FeCl_2(C_{18}H_{15}P)_2]$, the Fe atom exhibits a distorted tetrahedral coordination involving two Cl^- ions and two P donor functions of the triphenylphosphine ligands, with angles P—Fe—P = 111.25 (3)° and Cl—Fe—Cl = 124.42 (4)°.

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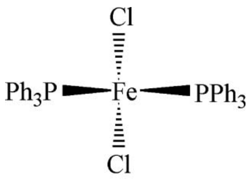
Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.107
Data-to-parameter ratio = 18.6

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

Comment

Only very few structures with a central $FeCl_2P_2$ core unit are known. A search in the Cambridge Structural Database [*MOGUL* Version 1.7; Allen (2002)] yielded a few hits, *e.g.* GAWDUD (Hermes & Girolami, 1988), RADMEP (Higham *et al.*, 2004) and WOFSUF (Renkema *et al.*, 1999), all with tetrahedrally coordinated iron. During our efforts to synthesize new phosphorus-bridged and phosphine-substituted multinuclear iron complexes, the novel title compound, (I), was isolated as a by-product.



(I)

The molecule lies on a crystallographic twofold axis which passes through the Fe centre. Fe—Cl and Fe—P bonds of

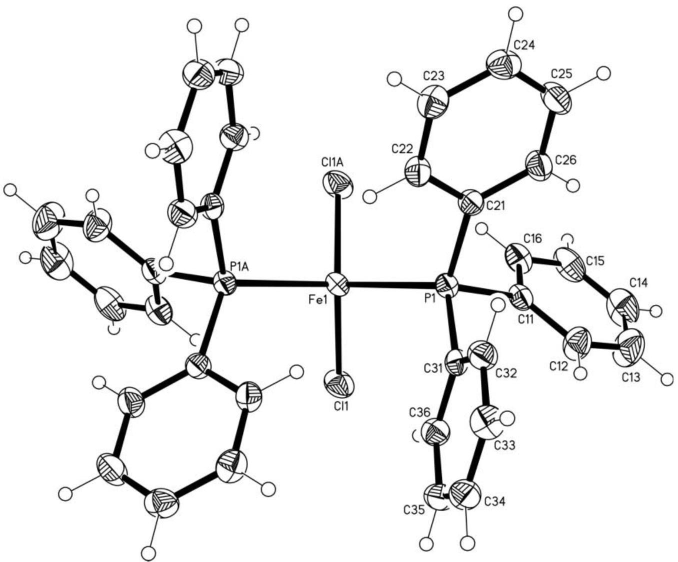


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code (A): $-x, y, \frac{1}{2} - z$.]

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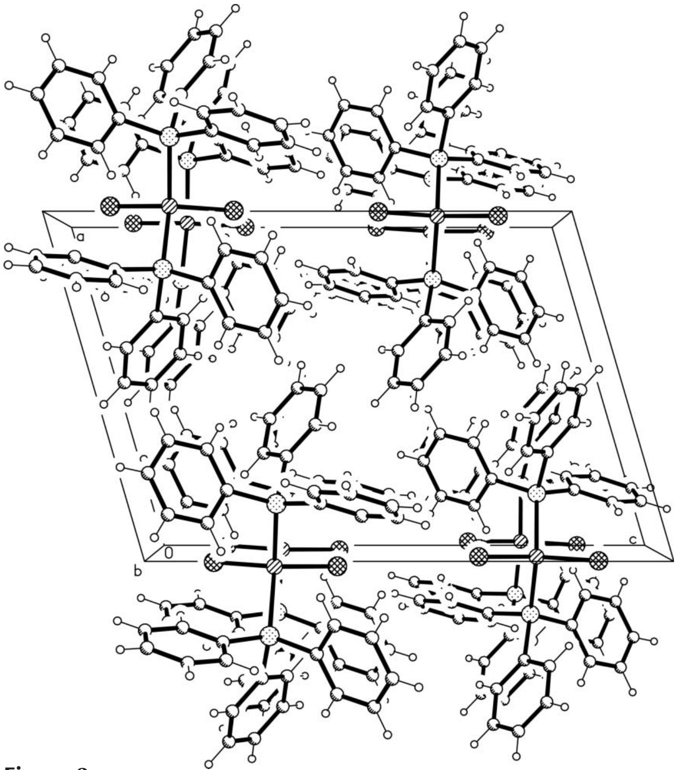

Figure 2
The crystal packing of (I), viewed along [010].

2.2193 (6) and 2.4759 (6) Å, respectively, are both shorter than the corresponding bond lengths in $[FeCl_2(PtBu_2Me)_2]$ (WOFSUF) of 2.2602 (5) and 2.5057 (5) Å, reflecting the better acceptor ability of the PPh_3 ligand compared with PBu_2Me. The Cl—Fe—Cl and P—Fe—P angles of 124.42 (4) and 111.25 (3)°, respectively, are large and differ by 13.2°. In WOFSUF, the respective angles are distinctly smaller at 110.97 (3) and 106.37 (3)°, and differ by only 4.6°. A similar relation for angles for (I) holds for the related nickel chloride (CLTPNI03; Brammer & Stevens, 1989) and bromide complexes (BPHPNI; Jarvis *et al.*, 1968), whereas cobalt (BIHGEE; Carlin *et al.*, 1982) and zinc chloride (GUXVAW; Vontragool *et al.*, 2003) compounds exhibit completely different patterns with respective angles of 117.28 (3) and 115.88 (2)° for Co and both 115.0 (2)° for Zn. The shortest intermolecular contact for (I) is C16—H16*A*···Cl1($-x$, y, $-z + \frac{1}{2}$), with H···Cl = 2.839 Å and an angle of 152.43° (corrected for C—H = 1.08 Å).

Experimental

In a glove-box under an argon atmosphere, a solution of $FeCl_3$ (1.62 g, 10 mmol) in ethanol (25 ml) was added dropwise to a solution of PPh_3 (10.48 g, 40 mmol) in tetrahydrofuran (THF, 50 ml). The resulting black mixture was combined with small pieces of P_4 (0.31 g, 10 mmol). Whilst stirring for 18 h, the colour of the solution brightened to pale yellow. The solvent was removed under reduced pressure. The residue was dissolved in a mixture of THF/ethanol. Colourless crystals were obtained using the vapor pressure equalization method with this THF/ethanol solution in the presence of *n*-hexane. ^{31}P NMR (C_6D_6): δ 31.9 (*s*, 2P, PPh_3).

Crystal data

$[FeCl_2(C_{18}H_{15}P)_2]$
$M_r = 651.29$
Monoclinic, $P2/c$
$a = 11.7937$ (13) Å
$b = 8.1652$ (9) Å
$c = 17.0760$ (19) Å
$\beta = 106.202$ (2)°
$V = 1579.1$ (3) Å^3
$Z = 2$

$D_x = 1.370$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4456 reflections
$\theta = 2.5$–28.2°
$\mu = 0.77$ mm^{-1}
$T = 153$ (2) K
Prism, colorless
0.50 × 0.30 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.699$, $T_{max} = 0.842$
9234 measured reflections

3455 independent reflections
2956 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.1°$
$h = -15 \rightarrow 10$
$k = -10 \rightarrow 9$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.107$
$S = 1.05$
3455 reflections
186 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.7177P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—Cl1	2.2192 (6)	Fe1—P1	2.4761 (6)
Cl1—Fe1—Cl1^i	124.42 (4)	Cl1—Fe1—P1	104.47 (2)
Cl1—Fe1—P1^i	106.06 (2)	P1^i—Fe1—P1	111.25 (3)

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

H atoms were placed at calculated positions, riding on their attached C atoms, with isotropic displacement parameters $U_{iso}(H) = 1.2U_{eq}(C)$ and C—H = 0.95 Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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