

(Willett, 1987); terminal Cu—Cl vs bridging Cu—Cl (Willett, 1988).

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Structure of Dichloro(η^5 -pentamethylcyclopentadienyl)(trimethylphosphine)iridium 0.25 Diethyl Ether Solvate

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Abstract. $[\text{Ir}(\text{C}_{10}\text{H}_{15})(\text{Cl})_2\{\text{P}(\text{CH}_3)_3\}] \cdot \frac{1}{4}(\text{C}_2\text{H}_5)_2\text{O}$, $M_r = 492.94$, tetragonal, $I4$, $a = 17.918$ (3), $c = 11.409$ (1) Å, $V = 3662.9$ Å³, $Z = 8$, $D_x = 1.78$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 76.4$ cm⁻¹, $F(000) = 1887.5$, $T = 296$ K, $R = 0.036$, $wR = 0.047$ for 959 observed reflections. The Ir atom is coordinated to an η^5 -pentamethylcyclopentadienyl group, a PMe_3 ligand and two Cl atoms in a three-legged piano-stool type of structure. Selected bond distances and angles are: Ir—C₅Me₅ centroid 1.85, Ir—P 2.28 (1), Ir—Cl(1) 2.37 (1), Ir—Cl(2) 2.39 (1) Å; Cl(1)—Ir—Cl(2) 93.0 (3), Cl(1)—Ir—P 87.6 (2), Cl(2)—Ir—P 86.9 (2), Cl(1)—Ir—C₅Me₅ centroid 124.0, Cl(2)—Ir—C₅Me₅ centroid 123.0, P—Ir—C₅Me₅ centroid 130.8°.

Experimental. Compound isolated from reaction of $[\text{Cp}^*\text{IrCl}_2]_2$ and PMe_3 in CHCl_3 ($\text{Cp}^* = \eta^5\text{-C}_5\text{Me}_5$); red crystals obtained from chloroform and diethyl ether. A cube-shaped crystal $0.28 \times 0.23 \times 0.13$ mm was mounted on a glass fibre. Intensity data were collected at 296 (1) K with an Enraf–Nonius CAD-4F diffractometer using graphite-monochromatized Mo $K\alpha$ radiation. Lattice parameters were determined from 25 reflections ($15 \leq \theta < 20^\circ$). 1206 independent reflections were measured ($1.6 \leq 2\theta \leq 50^\circ$; $h: 0\text{--}18$, $k: 0\text{--}18$, $l: 0\text{--}12$) by using ω - 2θ scans; 959 of the 1206 reflections were classed as

observed $I_o \geq 2\sigma(I_o)$; scan range $(0.85 + 0.35\tan\theta)^\circ$; scan speed $0.72\text{--}2.75^\circ \text{min}^{-1}$; intensities of two standards (701, $\bar{3}81$) were measured every hour of acquisition time and showed no long term change and had an r.m.s. deviation of 1.8%; data reduction included Lorentz and polarization corrections and an analytical absorption correction, ψ -scan checked, transmission 0.355–0.648 (Alcock, 1969). Structure solved by Patterson synthesis and refined by full-matrix least squares and Fourier methods with the z coordinate of Ir restrained to zero (z is the fourfold axis). The assumption that the space group is $I4$ was confirmed by the successful refinement. The positions of some methyl-group H atoms were located in electron density difference Fourier maps (with $\sin\theta/\lambda \leq 0.3$ Å⁻¹) and were used as input to calculate positions [$d(\text{C—H}) = 0.95$ Å] for all H atoms. Disorder of the solvent diethyl ether appeared to include some superposition of oxygen and carbon sites and gave peaks along the fourfold axis. It was modelled by three C atoms (see Table 1). Final refinement included anisotropic thermal parameters for Ir, Cl and P and isotropic thermal parameters for other non-H atoms. H atoms were included in fixed positions in structure-factor calculations. Refinement was considered complete when the shift/e.s.d. ratio was less than 0.002; 94 parameters varied. Final residual $R = 0.036$, $wR = 0.047$, goodness of fit 0.86; quantity

Table 1. Positional parameters and B_{iso} for $\text{Cp}^*\text{IrCl}_2(\text{PMe}_3)$

	x	y	z	$B_{\text{iso}}/B_{\text{eq}}(\text{\AA}^2)$
Ir	0.63983 (4)	0.19925 (5)	0.0	1.61†
Cl(1)	0.6251 (4)	0.1303 (4)	0.1761 (6)	2.2†
Cl(2)	0.6811 (4)	0.3084 (4)	0.1011 (7)	2.6†
P	0.5214 (4)	0.2426 (4)	0.0272 (5)	1.9†
C(Mel)	0.4501 (13)	0.1693 (13)	0.0121 (36)	2.9 (6)
C(Me2)	0.5019 (16)	0.2755 (16)	0.1790 (27)	3.0 (6)
C(Me3)	0.4955 (20)	0.3232 (19)	-0.0619 (31)	4.7 (8)
C(1)	0.7467 (14)	0.1904 (14)	-0.1071 (23)	1.8 (5)
C(2)	0.7240 (17)	0.1163 (17)	-0.0722 (28)	3.1 (6)
C(3)	0.6470 (18)	0.1069 (18)	-0.1231 (31)	3.3 (7)
C(4)	0.6280 (17)	0.1731 (17)	-0.1898 (26)	2.4 (6)
C(5)	0.6880 (15)	0.2265 (15)	-0.1730 (24)	2.3 (5)
C(11)	0.8197 (16)	0.2258 (16)	-0.0812 (26)	2.9 (6)
C(12)	0.7612 (14)	0.0613 (14)	-0.0134 (41)	3.6 (6)
C(13)	0.6032 (19)	0.0363 (19)	-0.1173 (33)	4.5 (7)
C(14)	0.5645 (19)	-0.1815 (20)	-0.2716 (33)	4.3 (7)
C(15)	0.6943 (19)	0.3024 (19)	-0.2260 (31)	4.1 (7)
C(s1)	0.5000 (0)	0.5000 (0)	0.471 (13)	9.6 (33)‡
C(s2)	0.5000 (0)	0.5000 (0)	0.365 (18)	13.4 (48)‡
C(s3)	0.5000 (0)	0.5000 (0)	0.269 (15)	11.6 (41)‡

† B_{eq} is the mean of the principal axes of the thermal ellipsoid.

‡ Carbon atoms, † multiplicity, modelling the disordered diethyl ether. C(s1) and C(s3) are ascribed to CH_3 groups, C(s2) to O, and the missing two CH_2 groups, which would fall off the fourfold axis, were not observed (since they would be † the occupancy of the axial sites). More complex disorder involving overlapping molecular sites is possible.

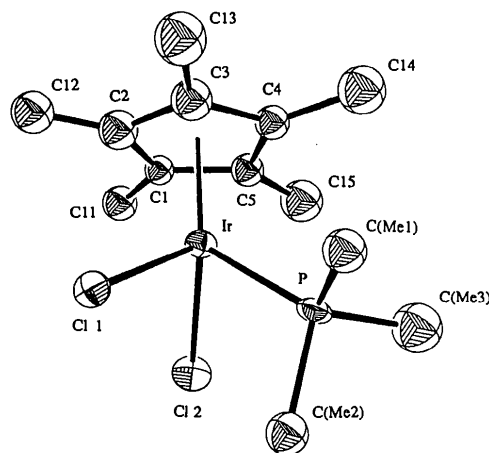
minimized $\sum w(F_o - F_c)^2$, where unit weights were used. The largest peak in the final electron density map is $1.7(2) e \text{\AA}^{-3}$ and is located 1.37\AA from Ir atom, and ρ_{min} is $-1.3(2) e \text{\AA}^{-3}$. Analytical form of scattering factors for the neutral atoms used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2B and 2.3.1); all non-H scattering factors corrected for real and imaginary components of anomalous dispersion. Inspection of $w\Delta^2$ as a function of $\sin\theta/\lambda$, F_o and values of h, k, l showed no unusual features or trends. Positional parameters are listed in Table 1 and bond distances and angles are listed in Table 2.† Programs used were from the *NRCVAX Crystal Structure System* (Gabe, Le Page, Charland, Lee & White, 1989) including their version of *ORTEP* (Johnson, 1965) and were run on a MicroVAX II computer. Fig. 1 shows the title compound with atomic labelling.

Related literature. The structure of the parent compound $[\text{Cp}^*\text{IrCl}_2]$ has been reported (Julis, 1979). The structure of a compound related to the title compound, where alkyl groups replace the chloro ligands, has also been determined (Diversi, Ingrosso, Lucherini, Porzio & Zocchi, 1980).

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54085 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected bond lengths (\AA) and interbond angles ($^\circ$) for $\text{Cp}^*\text{IrCl}_2(\text{PMe}_3)$

Ir—P	2.281 (7)	P—C(Me3)	1.83 (4)
Ir—Cl(1)	2.373 (7)	C(1)—C(2)	1.44 (4)
Ir—Cl(2)	2.387 (7)	C(2)—C(3)	1.51 (4)
Ir—Cp* centroid	1.85	C(3)—C(4)	1.45 (5)
Ir—C(1)	2.28 (3)	C(4)—C(5)	1.45 (4)
Ir—C(2)	2.27 (3)	C(5)—C(1)	1.44 (4)
Ir—C(3)	2.17 (3)	C(1)—C(11)	1.48 (4)
Ir—C(4)	2.23 (3)	C(2)—C(12)	1.37 (4)
Ir—C(5)	2.21 (3)	C(3)—C(13)	1.49 (5)
P—C(Mel)	1.84 (3)	C(4)—C(14)	1.48 (5)
P—C(Me2)	1.86 (3)	C(5)—C(15)	1.49 (4)
Cl(1)—Ir—Cl(2)	93.0 (3)	C(2)—C(1)—C(5)	111 (3)
Cl(1)—Ir—P	87.6 (2)	Ir—C(1)—C(11)	127 (2)
Cl(2)—Ir—P	86.8 (2)	Ir—C(2)—C(12)	128 (3)
Cl(1)—Ir—Cp* centroid	124	Ir—C(3)—C(13)	126 (3)
Cl(2)—Ir—Cp* centroid	123	Ir—C(4)—C(14)	132 (3)
P—Ir—Cp* centroid	131	Ir—C(5)—C(15)	126 (2)
C(Mel)—P—C(Me2)	101 (2)	C(2)—C(1)—C(11)	126 (3)
C(Mel)—P—C(Me3)	110 (2)	C(5)—C(1)—C(11)	124 (3)
C(Me2)—P—C(Me3)	103 (2)	C(1)—C(2)—C(12)	131 (3)
Ir—P—C(Mel)	113 (1)	C(3)—C(2)—C(12)	123 (3)
Ir—P—C(Me2)	114 (1)	C(2)—C(3)—C(13)	124 (3)
Ir—P—C(Me3)	115 (1)	C(4)—C(3)—C(13)	126 (3)
C(1)—C(2)—C(3)	105 (3)	C(3)—C(4)—C(14)	126 (3)
C(2)—C(3)—C(4)	109 (3)	C(5)—C(4)—C(14)	125 (3)
C(3)—C(4)—C(5)	107 (3)	C(4)—C(5)—C(15)	127 (3)
C(4)—C(5)—C(1)	108 (3)	C(1)—C(5)—C(15)	124 (3)

Fig. 1. View of a molecule of $\text{Cp}^*\text{IrCl}_2(\text{PMe}_3)$ with atom labelling.

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