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Carbonyldichlorohydridobis(trimethylphosphine)iridium(III)

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The crystal structure of the title compound, [$IrCl_2H(C_3H_9P)_2(CO)$] or [$IrCl_2(H)(CO)(PMe_3)_2$], has been determined. The compound is an octahedral iridium(III) complex with *trans* trimethylphosphine and *cis* chloride ligands.

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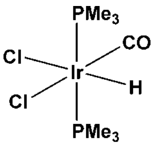
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(O–C) = 0.011 Å
R factor = 0.042
wR factor = 0.100
Data-to-parameter ratio = 24.8

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

Comment

The molecular structure of the title compound, (1), is similar to that of its triphenylphosphine analogue, (2) (Charmant *et al.*, 2004). Although the molecular structures of these two compounds are similar, the crystal structures differ in that the trimethylphosphine complex reported in this paper crystallizes in the lower symmetry space group *Pn* and shows no disorder between the carbonyl ligand and the chloride ligand *trans* to it. All attempts to obtain a better refinement of the structure in higher symmetry space groups with disordered ligands were unsuccessful. This difference between the two structures can readily be attributed to the differing packing requirements of the methyl and phenyl groups of the phosphine ligands.



(1)

Experimental

A solution of *trans*-[$IrCl(CO)(PMe_3)_2$] (0.080 g, 0.196 mmol) in toluene (2 ml) was prepared in a Young's tap tube. This was frozen in liquid nitrogen, the vessel evacuated and B_2F_4 (0.43 mmol) condensed on to the solution. The bright yellow reaction mixture was sealed *in vacuo* and allowed to warm to room temperature, with stirring once the solvent had melted. After *ca* 1 h, a slightly cloudy, pale-yellow solution resulted. The gaseous components were expanded out of the vessel, the solution filtered and an overlayer of *n*-pentane (5 ml) added. Solvent diffusion at 243 K over a period of several days yielded colourless crystals of the title compound as a minor product.

Crystal data

[$IrCl_2H(C_3H_9P)_2(CO)$]
M_r = 444.26
Monoclinic, *Pn*
a = 6.472 (2) Å
b = 13.156 (4) Å
c = 8.598 (3) Å
β = 97.157 (5)°
V = 726.4 (4) Å^3
Z = 2

D_x = 2.031 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5819 reflections
θ = 2–40°
μ = 9.75 mm^{-1}
T = 173 (2) K
Block, orange
0.20 × 0.10 × 0.05 mm

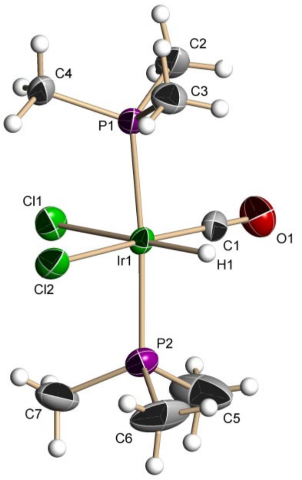


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\min} = 0.310$, $T_{\max} = 0.613$
7494 measured reflections
3176 independent reflections
2964 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.075$
$\theta_{\max} = 27.6°$
$h = -8 \rightarrow 8$
$k = -16 \rightarrow 17$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.100$
$S = 1.05$
3176 reflections
128 parameters
H atoms treated by a mixture of restrained and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0144P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 2.70$ e Å^{-3}
$\Delta\rho_{\min} = -3.57$ e Å^{-3}
Absolute structure: Flack (1983), 1496 Friedel pairs
Flack parameter = 0.149 (13)

Table 1
Selected geometric parameters (Å, °).

C1—Ir1	1.842 (9)	Cl1—Ir1	2.484 (2)
C3—P1	1.830 (10)	Cl2—Ir1	2.401 (3)
C4—P1	1.821 (9)	Ir1—P2	2.345 (3)
C5—P2	1.751 (12)	Ir1—P1	2.352 (3)
C6—P2	1.821 (12)	Ir1—H1	1.60 (2)
C7—P2	1.829 (12)		
C1—Ir1—P2	92.3 (3)	P1—Ir1—Cl1	88.86 (9)
C1—Ir1—P1	93.6 (3)	Cl2—Ir1—Cl1	90.98 (10)
P2—Ir1—P1	174.11 (12)	C1—Ir1—H1	84 (4)
C1—Ir1—Cl2	176.3 (3)	P2—Ir1—H1	93 (4)
P2—Ir1—Cl2	86.63 (9)	P1—Ir1—H1	88 (4)
P1—Ir1—Cl2	87.51 (10)	Cl2—Ir1—H1	92 (4)
C1—Ir1—Cl1	92.6 (3)	Cl1—Ir1—H1	176 (4)
P2—Ir1—Cl1	90.60 (10)		

A restraint of 1.60 (3) Å was applied to the Ir1—H1 distance and the displacement parameter of the hydride ligand atom was constrained to be 1.5 times that of the equivalent Ir1 parameter. The refined Flack (1983) parameter for the structure is significantly greater than zero, indicating that some racemic twinning is present. The highest peak and deepest hole in the electron-density difference map are both found close to Ir1 (0.08 and 0.86 Å, respectively).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SHELXTL* (Bruker, 2001) and *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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