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Carbonyldichlorohydridobis(triphenylphosphine)iridium(III)

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The crystal structure of the title compound, $[\mathrm{IrCl_2H(C_{18}H_{15}P)_2(CO)}]$ or $[\mathrm{IrCl_2(H)(CO)(PPh_3)_2}]$, has been determined. The compound is an octahedral iridium(III) complex with *trans* phosphine and *cis* chloride ligands. The molecule sits on a crystallographic twofold axis.

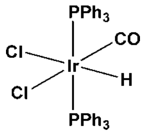
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.013 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.153
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.

Comment

Although the chemistry of Vaska's complex, *trans*-$[\mathrm{IrCl(CO)(PPh_3)_2}]$, (1), has been much studied, a structural characterization of $[\mathrm{IrCl_2(H)(CO)(PPh_3)_2}]$, (2), the product of oxidative addition of HCl to (1), has not yet been reported. Closely related *cis*- and *trans*-dichlorobis(triisopropylphosphine) complexes have been characterized (Capitani & Mura, 1997).



(2)

The title compound, (2), is formed as a minor product from the reaction between B_2F_4 and Vaska's complex, *trans*-$[\mathrm{IrCl(CO)(PPh_3)_2}]$, (1), presumably by oxidative addition of HCl afforded by some decomposition of (1) in its reaction with B_2F_4. The crystal structure shows a complex with the expected octahedral geometry, with the *trans* phosphine ligands bent slightly towards the sterically undemanding hydride ligand [P1—Ir—P1$^{\mathrm{i}}$ = 173.50 (10)°; symmetry code: (i) $-x, y, \frac{1}{2} - z$].

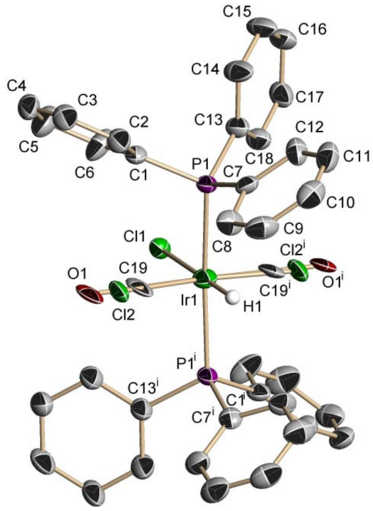


Figure 1
The molecular structure of (2), showing displacement ellipsoids drawn at the 50% probability level. Phenyl H atoms have been omitted for clarity.

It is worth noting that compound (2) is isostructural with a closely related compound, *viz.* *cis*-[$IrCl_2(H)(CO)(PPh_3)$-{$PPh_2(o$-$C_6H_4OH)$}] (Dahlenburg *et al.*, 1997).

Experimental

A solution of *trans*-[$IrCl(CO)(PPh_3)_2$] (0.035 g, 0.045 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 (0.05 mmol) condensed onto 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* 30 min, a colourless precipitate had formed beneath bright yellow mother liquors. All volatiles were removed *in vacuo* and CH_2Cl_2 (5 ml) added, forming a cloudy yellow solution that was subsequently filtered. Storage of the filtrate at 243 K for a period of several days yielded colourless crystals of the title compound as a minor product. The major product of this reaction is [$Ir(BF_2)_3(CO)(PPh_3)_2$] (Lu *et al.*, 2000).

Crystal data

[$IrCl_2H(C_{18}H_{15}P)_2(CO)$]
M_r = 816.66
Monoclinic, $C2/c$
a = 23.548 (6) Å
b = 9.596 (2) Å
c = 15.623 (3) Å
β = 115.505 (6)°
V = 3186.2 (12) Å^3
Z = 4
D_x = 1.702 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6054 reflections
θ = 2–27°
μ = 4.49 mm^{-1}
T = 173 (2) K
Block, orange
0.2 × 0.2 × 0.1 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.440, $T_{\max}$ = 0.640
13634 measured reflections
3675 independent reflections
2347 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.126
$\theta_{\max}$ = 27.6°
$h = -30 \rightarrow 30$
$k = -12 \rightarrow 12$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.153
S = 0.94
3675 reflections
204 parameters
H atoms treated by a mixture of restrained and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0863P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 1.92 e Å^{-3}
$\Delta\rho_{\min}$ = −2.47 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—P1	1.825 (7)	Ir1—Cl2^{i}	2.323 (5)
C7—P1	1.832 (8)	Ir1—P1	2.3549 (18)
C13—P1	1.824 (8)	Ir1—H1	1.60 (2)
Cl1—Ir1	2.465 (3)	C19—O1	1.173 (14)
Ir1—C19	1.840 (14)		
C19—Ir1—Cl2^{i}	171.8 (11)	Cl2^{i}—Ir1—Cl1	95.17 (16)
C19—Ir1—P1	88.3 (8)	P1—Ir1—Cl1	93.25 (5)
Cl2^{i}—Ir1—P1	91.43 (13)	C19—Ir1—H1	87.0 (12)
C19—Ir1—P1^{i}	91.3 (8)	Cl2^{i}—Ir1—H1	84.83 (16)
Cl2^{i}—Ir1—P1^{i}	87.98 (13)	P1—Ir1—H1	86.75 (5)
P1—Ir1—P1^{i}	173.50 (10)	Cl1—Ir1—H1	180.000 (5)
C19—Ir1—Cl1	93.0 (12)		

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

The molecule sits on a crystallographic twofold axis. This accords with the mutually *trans* carbonyl and chloride ligands having exact 50:50 disorder. Sensible bond lengths for the disordered ligands were obtained by applying restraints of 1.85 (3), 2.40 (3) and 1.15 (3) Å to the Ir1—C19, Ir1—Cl2 and C19—O1 distances, respectively. It was also necessary to constrain the displacement parameters of the carbonyl ligand atoms C19 and O1 to be identical in order to obtain satisfactory displacement parameters for these atoms. 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 highest peak and deepest hole in the electron-density difference map are both found close to Ir1 (1.23 and 0.88 Å, respectively).

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

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