

Aquadichloro(η^5 -pentamethylcyclopentadienyl)iridium(III)

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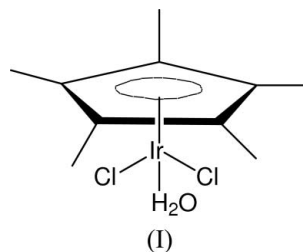
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.015$ Å
 R factor = 0.037
 wR factor = 0.083
Data-to-parameter ratio = 17.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Each Ir^{III} ion in the title compound, $[\text{Ir}(\text{C}_{10}\text{H}_{15})\text{Cl}_2(\text{H}_2\text{O})]$, is six-coordinated by two chloride ions, one water molecule and a pentamethylcyclopentadienyl (Cp^*) ligand in a three-legged piano-stool geometry, assuming that the Cp^* ligand functions as a tridentate ligand. There is hydrogen bonding between the water H atoms and the Cl atoms of adjacent molecules.

Comment

In the discrete electronically neutral title mononuclear iridium(III) complex, (I), the central Ir^{III} atom is in a three-legged piano-stool geometry and is coordinated by two chloride ions, one water molecule and a pentamethylcyclopentadienyl (Cp^*) ligand. The Ir–Cl bond distance (Table 1) is a little longer than in the related complex $[\{\text{Cp}^*\text{IrCl}_2\}_2(\text{pyrazine})]$ (2.38–2.40 Å; Wang *et al.*, 2006). Angles around the Ir^{III} atom in (I) are close to 90°, ranging from 82.1 (2) to 88.77 (10)°. The Cp^* ligand is symmetrically bound to the Ir^{III} atom. The distance between Ir and the least-squares plane of the Cp^* ring is 1.76 Å; this compares well with the values for other iridium complexes containing Cp^* (Wang *et al.*, 2005).



In the crystal structure, the coordinated water molecule forms hydrogen bonds with the coordinated Cl ions of adjacent molecules (Table 2).

Experimental

To a solution of $[\text{Cp}^*\text{IrCl}_2]_2$ (0.1 mmol, 80 mg) in MeCN–H₂O (3:1 *v/v*) was added NaHCO₃ (0.1 mmol, 8 mg) and the mixture was stirred for 1 h at room temperature. Crystals were obtained by evaporation of the resulting red solution over a period of a few days (yield: 27 mg, 33%). Elemental analysis, found: C 28.82, H 4.21%; calculated for C₁₀H₁₇Cl₂IrO: C 28.85, H 4.12%.

Crystal data

$[\text{Ir}(\text{C}_{10}\text{H}_{15})\text{Cl}_2(\text{H}_2\text{O})]$
 $M_r = 416.34$
Monoclinic, $P2_1/c$
 $a = 8.723$ (5) Å
 $b = 7.853$ (5) Å
 $c = 19.591$ (12) Å
 $\beta = 101.662$ (8)°
 $V = 1314.3$ (14) Å³

$Z = 4$
 $D_x = 2.104$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 10.53$ mm⁻¹
 $T = 293$ (2) K
Block, red
0.12 × 0.11 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{\min} = 0.365$, $T_{\max} = 0.419$
 (expected range = 0.304–0.349)

5235 measured reflections
 2303 independent reflections
 2095 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$
 $\theta_{\max} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.083$
 $S = 1.26$
 2303 reflections
 132 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (13.7468P)^2 + 14.5636P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.37 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.42 \text{ e } \text{\AA}^{-3}$

Table 1Selected geometric parameters (\AA , $^\circ$).

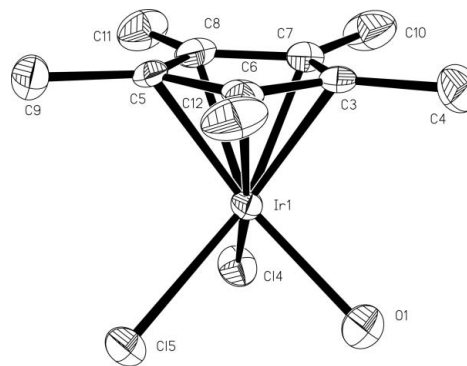
Ir1—O1	2.156 (8)	Ir1—Cl5	2.428 (3)
Ir1—Cl4	2.421 (3)		
O1—Ir1—Cl4	83.3 (3)	Cl4—Ir1—Cl5	88.76 (10)
O1—Ir1—Cl5	82.0 (2)		

Table 2Hydrogen-bond geometry (\AA , $^\circ$).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1A...Cl5	0.85	2.17	3.015 (9)	180
O1—H1B...Cl4 ⁱ	0.85	2.56	3.387 (9)	164

Symmetry code: (i) $-x + 1, -y, -z + 1$.

All water H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with O—H = 0.85 \AA and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. The carbon-bound H atoms were fixed at ideal positions, with C—H distances of 0.96 \AA and with a common

**Figure 1**

The structure of the title compound, (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

isotropic displacement parameter of $U_{\text{iso}}(\text{H}) = 0.12 \text{ \AA}^2$. The maximum electron-density peak is 1 \AA from atom Ir1.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINTE-Plus* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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References

- Bruker (1998). *SMART* (Version 5.6), *SAINTE* (Version 6.0), *SHELXTL* (Version 6.1) and *SADABS* (Version 5.6). Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Wang, J.-Q., Ren, C.-X. & Jin, G.-X. (2005). *Chem. Commun.* pp. 4738–4740.
- Wang, J.-Q., Ren, C.-X. & Jin, G.-X. (2006). *Organometallics*, **25**, 74–81.