

## Bis(acetylacetonato- $\kappa^2O,O'$ )aqua-(diacetylmethanido- $\kappa C$ )iridium(III)

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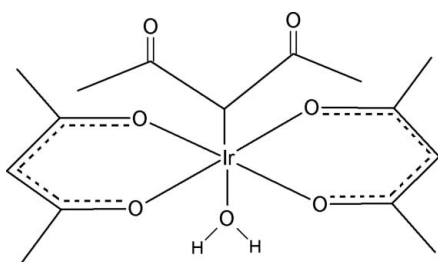
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(C-C) = 0.018$  Å;  $R$  factor = 0.058;  $wR$  factor = 0.127; data-to-parameter ratio = 18.1.

In the crystal structure of the title compound,  $[\text{Ir}(\text{C}_5\text{H}_7\text{O}_2)_3(\text{H}_2\text{O})]$ , the  $\text{Ir}^{\text{III}}$  atom is six-coordinated and situated in a slightly distorted octahedral environment. The complex contains both Ir—O and Ir—C bonds and was isolated from a reaction mixture of  $\text{IrCl}_3(\text{H}_2\text{O})_x$ , pentane-2,5-dione and  $\text{NaHCO}_3$ . O—H...O hydrogen bonding between the water molecules and the carbonyl O atoms of adjacent molecules leads to a layered motif extending parallel to (010).

### Related literature

For background to the title compound, see: Bennett & Mitchell (1976); Bhalla *et al.* (2005); Gibson (1969); Matsumoto *et al.* (2000); Periana *et al.* (2002); Wong-Foy *et al.* (2003). For a related structure, see: Isakova *et al.* (1999); For background on hydrogen bonding, see: Desiraju (1996).



### Experimental

#### Crystal data

$[\text{Ir}(\text{C}_5\text{H}_7\text{O}_2)_3(\text{H}_2\text{O})]$

$M_r = 507.54$

Triclinic,  $P\bar{1}$

$a = 7.7853$  (13) Å

$b = 7.9461$  (13) Å

$c = 16.362$  (3) Å

$\alpha = 77.295$  (2)°

$\beta = 77.927$  (2)°

$\gamma = 60.918$  (1)°

$V = 856.7$  (3) Å<sup>3</sup>

$Z = 2$

Mo  $K\alpha$  radiation

$\mu = 7.82$  mm<sup>-1</sup>

$T = 293$  K

0.15 × 0.13 × 0.09 mm

#### Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\text{min}} = 0.387$ ,  $T_{\text{max}} = 0.539$

7435 measured reflections

3900 independent reflections

2885 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$

$wR(F^2) = 0.127$

$S = 1.03$

3900 reflections

215 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 2.37$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -1.88$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

Ir1—O1	2.015 (8)	Ir1—O2	2.029 (7)
Ir1—O4	2.017 (7)	Ir1—C11	2.131 (11)
Ir1—O3	2.026 (7)	Ir1—O5	2.155 (7)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5D...O7 <sup>i</sup>	0.82	2.24	2.706 (11)	116
O5—H5E...O6 <sup>ii</sup>	0.89	2.11	2.715 (11)	125

Symmetry codes: (i)  $x, y + 1, z$ ; (ii)  $x + 1, y, z$ .

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2069).

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**supplementary materials**

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## Bis(acetylacetonato- $\kappa^2O,O'$ )aqua(diacetylmethanido- $\kappa C$ )iridium(III)

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### Comment

[Ir(acac-O, O)<sub>2</sub>(acac-C<sup>3</sup>)(H<sub>2</sub>O)], as a novel, thermally stable, homogeneous bis-acac-O, O—Ir(III) complex catalyst for the anti-Markovnikov, hydroarylation of olefins, has attracted considerable attention (Periana *et al.*, 2002, Wong-Foy *et al.* 2003, Bhalla *et al.*, 2005). Bennett *et al.* obtained [Ir( $\mu$ -acac-O,*O,C*<sup>3</sup>)(acac-O, O)(acac-C<sup>3</sup>)] and an unknown yellow solid as byproducts in the course of an attempt to improve the synthesis of Ir(acac)<sub>3</sub> (Bennett & Mitchell, 1976). However, they were unable to purify the yellow solid or characterize its structure. Recently Periana *et al.* successfully isolated this yellow product in pure form and proved it to be [Ir(acac-O, O)<sub>2</sub>(acac-C<sup>3</sup>)(H<sub>2</sub>O)] by spectroscopic data (Periana *et al.*, 2002). Despite the thermal and oxidative stability of octahedral iridium (III)-carbon  $\sigma$ -bonded complexes, there are little well characterized  $\gamma$ -C-bonded  $\beta$ -diketone complexes of iridium (III) except [Ir( $\mu$ -acac-O,*O,C*<sup>3</sup>)(acac-O, O)(acac-C<sup>3</sup>)] (Gibson, 1969, Matsumoto *et al.*, 2000).

Recently the single-crystal of the compound was obtained in our laboratory and we report its crystallographic structure. In the complex, the central Ir atom is coordinated by a octahedron of four oxygen atoms of two acetylacetonate ligands, one carbon atom of one acetylacetonate ligand and one oxygen atom of water molecule. The average Ir—O bond length of two acetylacetonate ligands is 2.02 Å, which agree with the literature data of Ir(acac)<sub>3</sub> (Isakova *et al.*, 1999), however, Ir—O bond length of coordinated water is 2.15 Å, which is longer than the former, whereas Ir—C bond length is found to be 2.13 Å. Intermolecular C—H $\cdots$ O hydrogen bond (Desiraju, 1996) and C—H $\cdots$ C hydrogen bond are present in the structure; while the C $\cdots$ O distances are within the range of 3.039 (9)–3.486 (10) Å, C—H $\cdots$ O are found to be within the range of 110–168°. The compound forms a hydrogen-bonded chain in which the O5 water uses two H atom to serve as a donor to the O atoms of adjacent molecules, these weaker interactions giving rise to a three-dimensional framework structure.

### Experimental

The complex was synthesized according literature (see Periana *et al.*, 2002). In a round-bottom flask equipped with a reflux condenser vented to an oil bubble, 5 g of IrCl<sub>3</sub>(H<sub>2</sub>O)<sub>x</sub> (54.34% of Ir, 14.13 mmol), 50 ml of 2, 5-pentadione (48.75 mmol) and 5 g of NaHCO<sub>3</sub> (59.5 mmol) were added. The mixture was heated to gentle reflux with stirring for 48 h. During this time, a yellow solid precipitated. The reaction mixture was cooled to room temperature and the solid was collected as crude product. The yellow solid was dissolved in 200 ml H<sub>2</sub>O at room temperature under vigorous stirring and was filtered. Solution was concentrated under vacuo to give 3.15 g (43.6%) complex as yellow microcrystalline. The compound was crystallized from water to obtain crystals suitable for X-ray structure analysis. <sup>1</sup>H NMR (CD<sub>3</sub>OD, p.p.m.): 5.54(s, 2H, CH), 5.49(s, 1H, CH), 1.96(s, 12H, CH<sub>3</sub>), 1.83(s, 6H, CH<sub>3</sub>). <sup>13</sup>C NMR (dmsO, p.p.m.): 208.4(s, C-acac, C=O), 186.1(s, O-acac, C=O), 102.6(s, O-acac, CH), 48.6(s, C-acac, CH), 31.6(s, C-acac, CH<sub>3</sub>), 26.6(s, O-acac, CH<sub>3</sub>). Anal. Calcd: Ir, 37.95; C, 35.54; H, 4.93. Found: Ir, 37.90; C, 35.48; H, 4.87.

## Refinement

All H atoms were initially located in a difference Fourier map but were positioned with idealized geometry and refined isotropic with  $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$  (1.5 for methyl H atoms) using a riding model with C—H = 0.93 and 0.97 Å).

## Figures

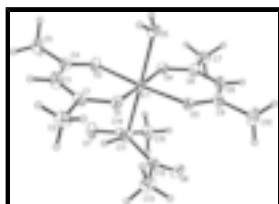


Fig. 1. Molecular view of the complex, with the atomic labeling scheme. Displacement ellipsoids are shown at the 30% probability level.

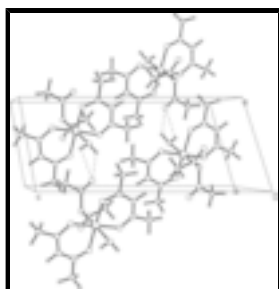


Fig. 2. Packing view, showing the C—H...O hydrogen-bond network between the complexes. Only the H atoms involved in H bonding are drawn. Hydrogen bonds are shown as dashed lines.

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$M_r = 507.54$

Triclinic,  $P\bar{1}$

$a = 7.7853$  (13) Å

$b = 7.9461$  (13) Å

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$\alpha = 77.295$  (2)°

$\beta = 77.927$  (2)°

$\gamma = 60.9180$  (10)°

$V = 856.7$  (3) Å<sup>3</sup>

$Z = 2$

$F_{000} = 492$

$D_x = 1.968$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1164 reflections

$\theta = 1.3$ – $28.4$ °

$\mu = 7.82$  mm<sup>-1</sup>

$T = 293$  K

Block, orange

$0.15 \times 0.13 \times 0.09$  mm

### Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$  K

$\varphi$  and  $\omega$  scans

3900 independent reflections

2885 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$

$\theta_{\text{max}} = 28.4$ °

$\theta_{\text{min}} = 1.3$ °

Absorption correction: Multi-scan  
(SADABS; Sheldrick, 1996)  $h = -10 \rightarrow 10$   
 $T_{\min} = 0.387$ ,  $T_{\max} = 0.539$   $k = -10 \rightarrow 10$   
 7435 measured reflections  $l = -21 \rightarrow 21$

### Refinement

Refinement on  $F^2$  Hydrogen site location: inferred from neighbouring sites  
 Least-squares matrix: full H-atom parameters constrained  
 $R[F^2 > 2\sigma(F^2)] = 0.058$   $w = 1/[\sigma^2(F_o^2) + (0.0469P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $wR(F^2) = 0.127$   $(\Delta/\sigma)_{\max} = 0.001$   
 $S = 1.03$   $\Delta\rho_{\max} = 2.37 \text{ e } \text{\AA}^{-3}$   
 3900 reflections  $\Delta\rho_{\min} = -1.87 \text{ e } \text{\AA}^{-3}$   
 215 parameters Extinction correction: SHELXL97 (Sheldrick, 2008),  
 $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.0013 (5)  
 Secondary atom site location: difference Fourier map

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Ir1	0.36783 (6)	0.62096 (6)	0.25137 (3)	0.02612 (17)
O1	0.3350 (11)	0.7800 (11)	0.1361 (5)	0.0327 (18)
O2	0.6421 (11)	0.4122 (11)	0.2154 (5)	0.0355 (19)
O3	0.1042 (10)	0.8433 (10)	0.2871 (5)	0.0315 (17)
O4	0.4064 (11)	0.4697 (11)	0.3682 (5)	0.0334 (18)
O5	0.5066 (10)	0.7689 (10)	0.2858 (5)	0.0337 (18)
H5D	0.5577	0.8115	0.2430	0.050*

## supplementary materials

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H5E	0.5997	0.6857	0.3187	0.050*
O6	-0.0934 (12)	0.5794 (12)	0.2823 (6)	0.046 (2)
O7	0.4419 (13)	0.1349 (12)	0.2185 (5)	0.048 (2)
C1	0.4627 (18)	0.7249 (17)	0.0700 (7)	0.035 (3)
C2	0.402 (2)	0.8478 (19)	-0.0103 (7)	0.047 (3)
H2A	0.3312	0.8038	-0.0347	0.070*
H2B	0.5167	0.8398	-0.0483	0.070*
H2C	0.3172	0.9801	-0.0006	0.070*
C3	0.6452 (19)	0.5594 (18)	0.0726 (8)	0.046 (3)
H3	0.7256	0.5384	0.0212	0.055*
C4	0.7252 (17)	0.4194 (15)	0.1418 (8)	0.034 (3)
C5	0.9347 (19)	0.256 (2)	0.1293 (9)	0.055 (4)
H5A	0.9385	0.1353	0.1578	0.083*
H5B	1.0224	0.2819	0.1519	0.083*
H5C	0.9756	0.2480	0.0701	0.083*
C6	0.2844 (19)	0.5342 (18)	0.4321 (7)	0.040 (3)
C7	0.334 (2)	0.402 (2)	0.5146 (8)	0.055 (4)
H7A	0.2550	0.3352	0.5289	0.083*
H7B	0.3074	0.4781	0.5582	0.083*
H7C	0.4718	0.3091	0.5092	0.083*
C8	0.1094 (19)	0.7063 (19)	0.4332 (8)	0.044 (3)
H8	0.0380	0.7318	0.4860	0.052*
C9	0.0265 (18)	0.8459 (17)	0.3658 (7)	0.036 (3)
C10	-0.1719 (19)	1.0193 (19)	0.3801 (8)	0.054 (4)
H10A	-0.1759	1.1296	0.3405	0.080*
H10B	-0.1923	1.0475	0.4365	0.080*
H10C	-0.2742	0.9916	0.3725	0.080*
C11	0.2348 (16)	0.4738 (15)	0.2148 (8)	0.034 (3)
H11	0.2784	0.4669	0.1544	0.041*
C12	0.0184 (17)	0.5990 (16)	0.2220 (8)	0.033 (3)
C13	-0.0656 (19)	0.7536 (19)	0.1483 (8)	0.051 (3)
H13A	-0.0591	0.6933	0.1022	0.076*
H13B	0.0101	0.8241	0.1309	0.076*
H13C	-0.2009	0.8417	0.1649	0.076*
C14	0.3079 (17)	0.2716 (16)	0.2559 (7)	0.030 (2)
C15	0.226 (2)	0.2179 (18)	0.3450 (8)	0.046 (3)
H15A	0.3341	0.1330	0.3776	0.070*
H15B	0.1511	0.1529	0.3428	0.070*
H15C	0.1427	0.3337	0.3707	0.070*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ir1	0.0211 (2)	0.0223 (2)	0.0273 (2)	-0.00479 (17)	-0.00288 (16)	-0.00175 (15)
O1	0.028 (4)	0.031 (4)	0.035 (4)	-0.012 (4)	0.001 (4)	-0.007 (4)
O2	0.030 (4)	0.030 (4)	0.039 (5)	-0.008 (4)	0.000 (4)	-0.006 (4)
O3	0.025 (4)	0.027 (4)	0.032 (4)	-0.002 (3)	-0.007 (3)	-0.007 (3)
O4	0.033 (4)	0.041 (5)	0.024 (4)	-0.017 (4)	-0.004 (3)	0.003 (3)

O5	0.021 (4)	0.032 (4)	0.038 (4)	-0.008 (3)	-0.006 (3)	0.003 (4)
O6	0.026 (5)	0.044 (5)	0.058 (6)	-0.010 (4)	-0.005 (4)	0.000 (4)
O7	0.056 (6)	0.028 (4)	0.048 (5)	-0.011 (4)	0.009 (4)	-0.016 (4)
C1	0.045 (7)	0.039 (7)	0.024 (6)	-0.021 (6)	-0.007 (5)	-0.003 (5)
C2	0.059 (9)	0.051 (8)	0.030 (7)	-0.028 (7)	-0.018 (6)	0.011 (6)
C3	0.052 (8)	0.043 (8)	0.029 (7)	-0.012 (7)	-0.003 (6)	-0.004 (6)
C4	0.030 (6)	0.020 (5)	0.049 (7)	-0.009 (5)	0.004 (6)	-0.015 (5)
C5	0.039 (8)	0.054 (9)	0.060 (9)	-0.010 (7)	0.011 (7)	-0.029 (7)
C6	0.046 (8)	0.048 (8)	0.029 (6)	-0.029 (7)	0.002 (6)	0.002 (6)
C7	0.054 (9)	0.078 (11)	0.039 (7)	-0.036 (8)	-0.011 (7)	0.002 (7)
C8	0.044 (8)	0.055 (8)	0.031 (7)	-0.023 (7)	0.009 (6)	-0.016 (6)
C9	0.039 (7)	0.033 (7)	0.036 (7)	-0.017 (6)	0.003 (6)	-0.012 (5)
C10	0.038 (8)	0.049 (8)	0.043 (8)	0.001 (7)	-0.001 (6)	-0.005 (7)
C11	0.021 (6)	0.022 (6)	0.050 (7)	-0.002 (5)	-0.005 (5)	-0.003 (5)
C12	0.030 (6)	0.026 (6)	0.043 (7)	-0.010 (5)	-0.011 (6)	-0.009 (5)
C13	0.043 (8)	0.047 (8)	0.049 (8)	-0.015 (7)	-0.006 (6)	0.003 (6)
C14	0.038 (7)	0.030 (6)	0.032 (6)	-0.021 (5)	-0.009 (5)	-0.006 (5)
C15	0.058 (9)	0.041 (7)	0.042 (7)	-0.024 (7)	-0.003 (6)	-0.005 (6)

*Geometric parameters (Å, °)*

Ir1—O1	2.015 (8)	C5—H5C	0.9600
Ir1—O4	2.017 (7)	C6—C8	1.384 (17)
Ir1—O3	2.026 (7)	C6—C7	1.508 (16)
Ir1—O2	2.029 (7)	C7—H7A	0.9600
Ir1—C11	2.131 (11)	C7—H7B	0.9600
Ir1—O5	2.155 (7)	C7—H7C	0.9600
O1—C1	1.297 (13)	C8—C9	1.388 (17)
O2—C4	1.245 (13)	C8—H8	0.9300
O3—C9	1.302 (13)	C9—C10	1.504 (16)
O4—C6	1.266 (13)	C10—H10A	0.9600
O5—H5D	0.8200	C10—H10B	0.9600
O5—H5E	0.8900	C10—H10C	0.9600
O6—C12	1.208 (13)	C11—C14	1.468 (14)
O7—C14	1.252 (13)	C11—C12	1.477 (15)
C1—C3	1.391 (16)	C11—H11	0.9800
C1—C2	1.468 (15)	C12—C13	1.513 (16)
C2—H2A	0.9600	C13—H13A	0.9600
C2—H2B	0.9600	C13—H13B	0.9600
C2—H2C	0.9600	C13—H13C	0.9600
C3—C4	1.406 (16)	C14—C15	1.520 (15)
C3—H3	0.9300	C15—H15A	0.9600
C4—C5	1.518 (16)	C15—H15B	0.9600
C5—H5A	0.9600	C15—H15C	0.9600
C5—H5B	0.9600		
O1—Ir1—O4	177.1 (3)	O4—C6—C7	115.2 (11)
O1—Ir1—O3	84.6 (3)	C8—C6—C7	117.6 (11)
O4—Ir1—O3	95.1 (3)	C6—C7—H7A	109.5
O1—Ir1—O2	94.2 (3)	C6—C7—H7B	109.5

## supplementary materials

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O4—Ir1—O2	85.9 (3)	H7A—C7—H7B	109.5
O3—Ir1—O2	175.4 (3)	C6—C7—H7C	109.5
O1—Ir1—C11	87.6 (4)	H7A—C7—H7C	109.5
O4—Ir1—C11	95.3 (4)	H7B—C7—H7C	109.5
O3—Ir1—C11	93.5 (4)	C6—C8—C9	128.5 (11)
O2—Ir1—C11	90.9 (4)	C6—C8—H8	115.7
O1—Ir1—O5	91.6 (3)	C9—C8—H8	115.7
O4—Ir1—O5	85.5 (3)	O3—C9—C8	126.0 (11)
O3—Ir1—O5	87.3 (3)	O3—C9—C10	113.8 (10)
O2—Ir1—O5	88.2 (3)	C8—C9—C10	120.2 (11)
C11—Ir1—O5	178.8 (4)	C9—C10—H10A	109.5
C1—O1—Ir1	123.5 (7)	C9—C10—H10B	109.5
C4—O2—Ir1	122.2 (7)	H10A—C10—H10B	109.5
C9—O3—Ir1	121.2 (7)	C9—C10—H10C	109.5
C6—O4—Ir1	121.9 (8)	H10A—C10—H10C	109.5
Ir1—O5—H5D	109.5	H10B—C10—H10C	109.5
Ir1—O5—H5E	109.5	C14—C11—C12	117.6 (10)
H5D—O5—H5E	109.2	C14—C11—Ir1	112.4 (8)
O1—C1—C3	123.7 (10)	C12—C11—Ir1	108.4 (7)
O1—C1—C2	115.6 (11)	C14—C11—H11	105.8
C3—C1—C2	120.7 (11)	C12—C11—H11	105.8
C1—C2—H2A	109.5	Ir1—C11—H11	105.8
C1—C2—H2B	109.5	O6—C12—C11	123.2 (11)
H2A—C2—H2B	109.5	O6—C12—C13	119.2 (11)
C1—C2—H2C	109.5	C11—C12—C13	117.6 (11)
H2A—C2—H2C	109.5	C12—C13—H13A	109.5
H2B—C2—H2C	109.5	C12—C13—H13B	109.5
C1—C3—C4	129.1 (11)	H13A—C13—H13B	109.5
C1—C3—H3	115.5	C12—C13—H13C	109.5
C4—C3—H3	115.5	H13A—C13—H13C	109.5
O2—C4—C3	126.9 (11)	H13B—C13—H13C	109.5
O2—C4—C5	114.0 (11)	O7—C14—C11	120.8 (10)
C3—C4—C5	119.1 (11)	O7—C14—C15	117.0 (10)
C4—C5—H5A	109.5	C11—C14—C15	122.2 (10)
C4—C5—H5B	109.5	C14—C15—H15A	109.5
H5A—C5—H5B	109.5	C14—C15—H15B	109.5
C4—C5—H5C	109.5	H15A—C15—H15B	109.5
H5A—C5—H5C	109.5	C14—C15—H15C	109.5
H5B—C5—H5C	109.5	H15A—C15—H15C	109.5
O4—C6—C8	127.1 (11)	H15B—C15—H15C	109.5
O4—Ir1—O1—C1	96 (6)	C1—C3—C4—C5	-176.5 (12)
O3—Ir1—O1—C1	-179.7 (9)	Ir1—O4—C6—C8	2.3 (17)
O2—Ir1—O1—C1	4.8 (9)	Ir1—O4—C6—C7	179.4 (8)
C11—Ir1—O1—C1	-85.9 (9)	O4—C6—C8—C9	1(2)
O5—Ir1—O1—C1	93.2 (9)	C7—C6—C8—C9	-176.4 (13)
O1—Ir1—O2—C4	0.1 (9)	Ir1—O3—C9—C8	1.1 (16)
O4—Ir1—O2—C4	-177.0 (9)	Ir1—O3—C9—C10	-178.4 (8)
O3—Ir1—O2—C4	-76 (4)	C6—C8—C9—O3	-3(2)
C11—Ir1—O2—C4	87.7 (9)	C6—C8—C9—C10	176.9 (13)



O5—Ir1—O2—C4	-91.4 (9)	O1—Ir1—C11—C14	149.4 (8)
O1—Ir1—O3—C9	-176.0 (8)	O4—Ir1—C11—C14	-30.7 (8)
O4—Ir1—O3—C9	1.1 (8)	O3—Ir1—C11—C14	-126.1 (8)
O2—Ir1—O3—C9	-100 (4)	O2—Ir1—C11—C14	55.2 (8)
C11—Ir1—O3—C9	96.8 (9)	O5—Ir1—C11—C14	101 (17)
O5—Ir1—O3—C9	-84.1 (8)	O1—Ir1—C11—C12	-78.8 (8)
O1—Ir1—O4—C6	81 (6)	O4—Ir1—C11—C12	101.0 (8)
O3—Ir1—O4—C6	-2.7 (9)	O3—Ir1—C11—C12	5.6 (8)
O2—Ir1—O4—C6	172.7 (9)	O2—Ir1—C11—C12	-173.0 (8)
C11—Ir1—O4—C6	-96.8 (9)	O5—Ir1—C11—C12	-127 (16)
O5—Ir1—O4—C6	84.2 (9)	C14—C11—C12—O6	30.9 (17)
Ir1—O1—C1—C3	-6.4 (16)	Ir1—C11—C12—O6	-98.0 (11)
Ir1—O1—C1—C2	172.5 (8)	C14—C11—C12—C13	-146.7 (11)
O1—C1—C3—C4	2(2)	Ir1—C11—C12—C13	84.4 (11)
C2—C1—C3—C4	-176.5 (13)	C12—C11—C14—O7	135.2 (12)
Ir1—O2—C4—C3	-3.7 (17)	Ir1—C11—C14—O7	-97.9 (11)
Ir1—O2—C4—C5	176.3 (7)	C12—C11—C14—C15	-45.0 (16)
C1—C3—C4—O2	3(2)	Ir1—C11—C14—C15	82.0 (12)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O5—H5D $\cdots$ O7 <sup>i</sup>	0.82	2.24	2.706 (11)	116
O5—H5E $\cdots$ O6 <sup>ii</sup>	0.89	2.11	2.715 (11)	125

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*+1, *y*, *z*.

Fig. 1

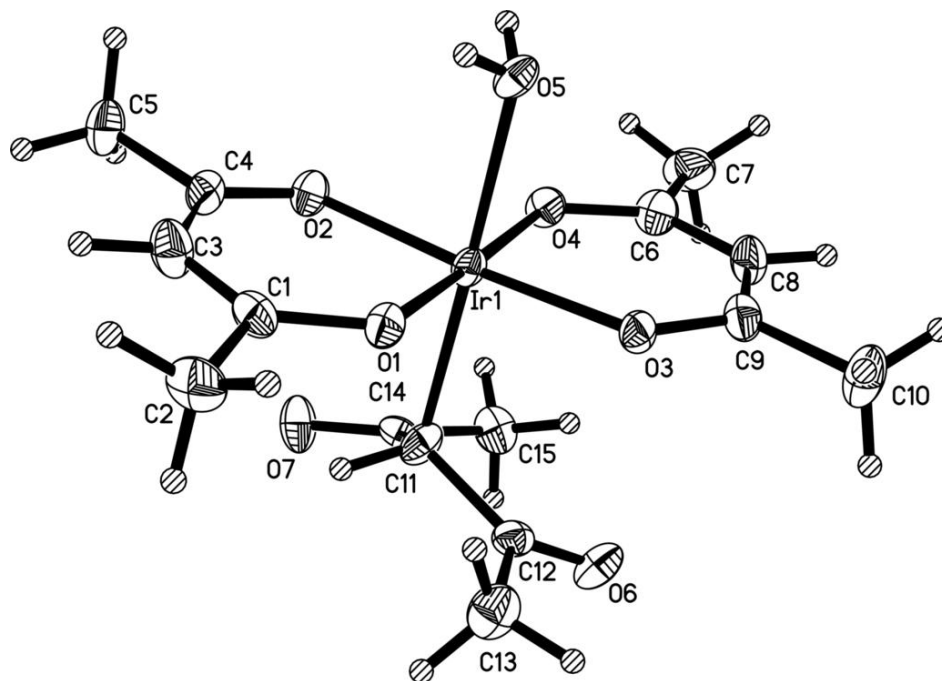


Fig. 2

