

Tae-Jeong Kim^a and Uk Lee^{b*}^aDepartment of Applied Chemistry, Kyungpook National University, Taegu 702-701, Republic of Korea, and ^bDepartment of Chemistry, Pukyong National University, 599-1 Daeyeon 3-dong Nam-ku, Busan 608-737, Republic of Korea

Correspondence e-mail: uklee@pknu.ac.kr

Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
Disorder in main residue
 R factor = 0.028
 wR factor = 0.067
Data-to-parameter ratio = 16.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.***cis*-(2-Acetylcyclopentanonato- $\kappa^2\text{O},\text{O}'$)-bis(2-pyridylphenyl- κN)iridium(III) dichloromethane solvate**

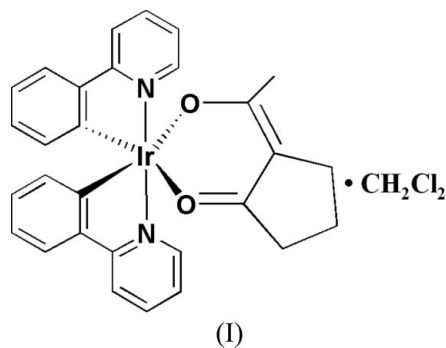
The title compound, $[\text{Ir}(\text{C}_{11}\text{H}_8\text{N})_2(\text{C}_7\text{H}_9\text{O}_2)] \cdot \text{CH}_2\text{Cl}_2$ or $[\text{Ir}(\text{ppy})_2(2\text{-acetylcyclopentanone})]$ (ppy = 2-phenylpyridine), is a neutral mononuclear iridium(III) complex. The complex and solvent molecules both lie on twofold rotation axes. The iridium centre is coordinated by two N atoms and two C atoms from two ppy ligands [$\text{Ir}-\text{N} = 2.044(3)$ Å and $\text{Ir}-\text{C} = 1.992(4)$ Å], and two O atoms from the β -diketonate ligand [$\text{Ir}-\text{O} = 2.11(2)$ and $2.21(2)$ Å], forming a distorted octahedral environment. The structure is stabilized by aromatic $\pi-\pi$ stacking and $\text{C}-\text{H} \cdots \pi$ interactions.

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Comment

This work is related to a previously reported communication on the Ir^{III} complex with the bis(ppy) analogue, *cis*-bis(phenylpyridine)(3-methylpentane-2,4-dionate)iridium(III) (Kim & Lee, 2006). A great number of cationic and neutral Ir^{III} complexes of the types $[\text{Ir}(\text{CN})_2\text{L}_2]\text{X}$ and $[\text{Ir}(\text{CN})_2(\text{LL})]$, where CN represents typically cyclometallating phenylpyridine derivatives, have been reported (Baldo *et al.*, 2000; Pomestchenko *et al.*, 2003; Duan *et al.*, 2003); L and LL are mono- and bidentate chelating anions, respectively, and X is any counter-anion. The title complex, (I), belongs to the latter type, where CN is ppy, and LL is 2-acetylcyclopentanone. The synthesis and electroluminescent properties of (I) have recently been reported by one of the authors and others (Kim, Paik *et al.*, 2004; Kim, Kim *et al.*, 2004).



Compound (I) (Fig. 1) is a solvated neutral mononuclear $[\text{Ir}(\text{ppy})_2(2\text{-acetylcyclopentanone})]$ (ppy is 2-phenylpyridine) complex. The asymmetric unit consists of one half-molecule of the complex and one half-molecule of solvent. Both molecules lie on twofold rotation axes. Bond distances for the coordination of Ir are summarized in Table 1. The Ir atom has a distorted octahedral geometry involving two ppy ligands and a β -diketonate ligand. The dihedral angle made by the N1/C1–C5 ring and the C6–C11 ring of ppy is $2.6(3)^\circ$.

The crystal structure is stabilized by aromatic π - π stacking and $\text{Cl}_2\text{HC}-\text{H}\cdots\pi$ interactions (Fig. 2). The shortest parallel ring centroid separation is $\text{Cg1}\cdots\text{Cg2}^{\text{ii}}$ of 3.748 (7) Å and the shortest distance is $\text{Cg2}\cdots\text{H19(A,B)}-\text{C19}^{\text{v}}$ of 2.60 Å [Cg1 and Cg2 are the centroids of the C1-C5/N and C6-C11 rings; symmetry codes as in Fig. 2].

Experimental

A mixture of $[\text{IrCl}(\text{ppy})_2\text{IrCl}]_2$ (83.6 mg, 0.078 mmol), 2-acetylcyclopentanone (25.2 mg, 0.2 mmol) and potassium carbonate (82.9 mg, 0.6 mmol) in 2-ethoxyethanol (5 ml) was stirred under reflux for 1 h. After cooling the reaction mixture to room temperature, the yellow precipitate was filtered off, and washed with water, followed by two portions of methanol (5 ml). The crude product was separated by chromatography on silica gel with dichloromethane as eluent to give a yellow solid (71.3 mg, 73%). Single crystals suitable for X-ray diffraction were grown by slow diffusion of hexane into a dichloromethane solution.

Crystal data

$[\text{Ir}(\text{C}_{11}\text{H}_8\text{N})_2(\text{C}_7\text{H}_9\text{O}_2)]\cdot\text{CH}_2\text{Cl}_2$
 $M_r = 710.64$
 Monoclinic, $C2/c$
 $a = 12.094$ (1) Å
 $b = 14.889$ (2) Å
 $c = 15.287$ (2) Å
 $\beta = 96.743$ (9)°
 $V = 2733.7$ (6) Å³
 $Z = 4$
 $D_x = 1.727$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 5.11$ mm⁻¹
 $T = 298$ (2) K
 Block, yellow
 $0.50 \times 0.25 \times 0.20$ mm

Data collection

Stoe STADI-4 diffractometer
 ω - 2θ scans
 Absorption correction: numerical
 (*X-SHAPE*; Stoe & Cie, 1996)
 $T_{\text{min}} = 0.431$, $T_{\text{max}} = 0.533$
 3137 measured reflections
 3137 independent reflections
 2873 reflections with $I > 2\sigma(I)$
 $\theta_{\text{max}} = 27.5^\circ$
 3 standard reflections
 frequency: 60 min
 intensity decay: 3.2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.067$
 $S = 1.07$
 3137 reflections
 192 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0313P)^2 + 8.9226P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.70$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.53$ e Å⁻³

Table 1

Selected bond lengths (Å).

Ir1—C11	1.992 (4)	C12—C16	1.468 (8)
Ir1—N1	2.044 (3)	C12—C13	1.500 (9)
Ir1—O1	2.11 (2)	C13—C14	1.478 (8)
Ir1—O2	2.21 (2)	C14—C15	1.479 (9)
O1—C12	1.258 (7)	C15—C16	1.507 (7)
O2—C17	1.269 (8)	C16—C17	1.358 (8)

The 2-acetylcyclopentanone ligand is disordered over a twofold rotation axis and all non-H atoms were restrained to lie in a plane with half-occupancy. Bond distances were restrained to acceptable values; the displacement ellipsoids of the pairs O1/O2, C12/C17 and C13/C18 were set equal to each other. The vibration of C16 was restrained to be nearly isotropic. All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aromatic, 0.97 Å for methylene, and 0.96 Å for methyl H atoms,

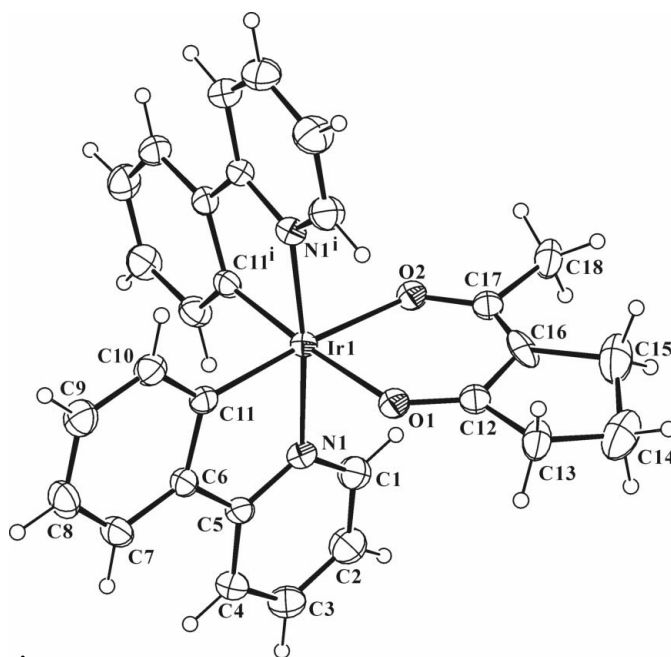


Figure 1

The molecular structure of (I), shown with 30% probability displacement ellipsoids. One disordered component of 2-acetylcyclopentanone and the solvent (CH_2Cl_2) have been omitted. [Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.]

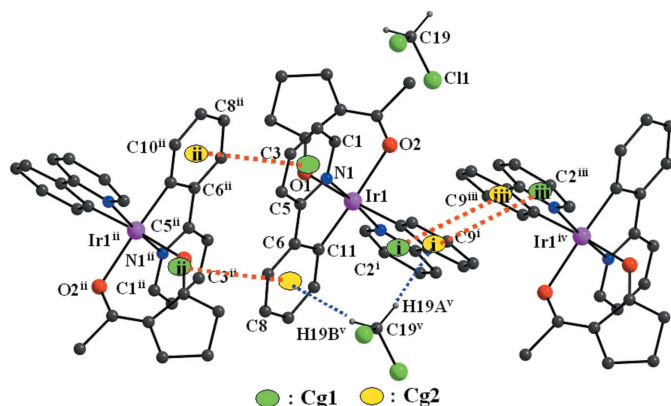


Figure 2

The π - π and $\text{Cl}_2\text{HC}-\text{H}\cdots\pi$ interactions in (I), shown as red and blue dotted lines, respectively. Cg1 and Cg2 are the centroids of the C1-C4/N and C6-C11 rings, respectively. H atoms of ppy have been omitted for clarity. [Symmetry codes: (i) $1 - x, y, \frac{3}{2} - z$; (ii) $1 - x, -y, 1 - z$; (iii) $x, -y, \frac{1}{2} + z$; (iv) $1 - x, -y, 2 - z$; (v) $\frac{1}{2} + x, y - \frac{1}{2}, z$.]

and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic and methylene H atoms, and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: *STADI4* (Stoe & Cie, 1996); cell refinement: *STADI4*; data reduction: *X-RED32* (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS97-2* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97-2* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97-2*.

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