

***cis*-(3-Methylpentane-2,4-dionato- κ^2 O,O')-bis(2-phenylpyridine- κ^2 C,N)iridium(III)**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

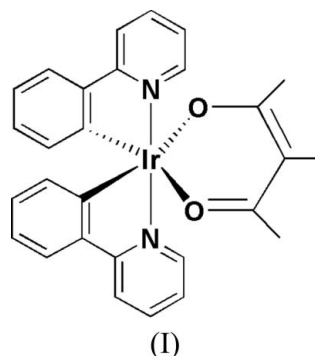
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Key indicatorsSingle-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.010$ Å
 R factor = 0.036
 wR factor = 0.080
Data-to-parameter ratio = 17.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $[\text{Ir}(\text{C}_{11}\text{H}_8\text{N})_2(\text{C}_6\text{H}_9\text{O}_2)]$, is a neutral mononuclear Ir^{III} complex, with the metal centre coordinated by two N atoms and two C atoms from two 2-phenylpyridine ligands [mean Ir–N and Ir–C bond lengths 2.044 (5) and 2.001 (6) Å, respectively], and two O atoms from the β -diketonate ligand [mean Ir–O bond length 2.134 (4) Å], forming a distorted octahedral environment.

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Iridium complexes have recently been the subject of considerable research activity, due to their phosphorescent properties, in connection with organic light-emitting diodes. Thus, a number of cationic and neutral Ir^{III} complexes of the types $[\text{Ir}(\text{CN})_2\text{L}_2]\text{X}$ and $[\text{Ir}(\text{CN})_2(\text{LL})]$ have been described, where CN represents typically cyclometallating phenylpyridine derivatives (Baldo *et al.*, 2000; Pomestchenko *et al.*, 2003; Duan *et al.*, 2003), while L and LL are mono- and bidentate chelating anions, respectively, and X is any counteranion. The title complex, (I), belongs to the latter type, where CN is 2-phenylpyridine (ppy) and LL is the 3-methylpentane-2,4-dionate anion. It should be noted here that the synthesis and electroluminescent properties of (I) have recently been reported by one of the authors (Kim, Paik *et al.*, 2004; Kim, Kim *et al.*, 2004).



Compound (I) (Fig.1) is a neutral mononuclear complex. The Ir^{III} ion is located in a distorted octahedral geometry (Table 1) involving two ppy ligands and a β -diketonate anion. The equatorial plane is formed by atoms O1/O2/C11/C12, the mean deviation from the least-squares plane being 0.086 Å. The Ir^{III} ion is displaced by 0.006 (3) Å from the equatorial plane towards the axial N1 atom. The dihedral angles between rings in the two ppy ligands are 3.71 (4)° (between rings N1/C1–C5 and C6–C11) and 7.9 (4)° (between rings N2/C18–C22 and C12–C17).

Classical inter-ligand interactions, *viz.* hydrogen bonds and π – π and C–H \cdots π interactions, are not detected in the crystal structure of (I).

Experimental

A mixture of [(ppy)₂IrCl]₂ (83.6 mg, 0.078 mmol), 3-methyl-2,4-pentanedione (22.8 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 resulting 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 (I) as a yellow solid (81.3 mg, 85% yield). Single crystals suitable for X-ray diffraction were grown by slow diffusion of hexane into a dichloromethane solution.

Crystal data

[Ir(C ₁₁ H ₈ N) ₂ (C ₆ H ₉ O ₂)]	Z = 4
M _r = 613.70	D _x = 1.748 Mg m ⁻³
Monoclinic, P2 ₁ /n	Mo K α radiation
a = 11.589 (2) Å	μ = 5.75 mm ⁻¹
b = 9.589 (2) Å	T = 298 (2) K
c = 20.993 (2) Å	Tetragonal prism, yellow
β = 91.678 (9)°	0.50 × 0.20 × 0.13 mm
V = 2331.9 (7) Å ³	

Data collection

Stoe STADI-4 four-circle diffractometer	5327 independent reflections
$\omega/2\theta$ scans	4152 reflections with $I > 2\sigma(I)$
Absorption correction: numerical (X-SHAPE; Stoe & Cie, 1996)	$\theta_{\max} = 27.5^\circ$
$T_{\min} = 0.346$, $T_{\max} = 0.581$	3 standard reflections
5327 measured reflections	frequency: 60 min
	intensity decay: 3.5%

Refinement

Refinement on F ²	$w = 1/[\sigma^2(F_o^2) + (0.0265P)^2 + 4.2427P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.080$	$(\Delta/\sigma)_{\max} < 0.001$
S = 1.13	$\Delta\rho_{\max} = 1.37 \text{ e \AA}^{-3}$
5327 reflections	$\Delta\rho_{\min} = -0.71 \text{ e \AA}^{-3}$
301 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

Ir—C12	1.997 (6)	O1—C23	1.266 (7)
Ir—C11	2.005 (6)	O2—C27	1.275 (7)
Ir—N1	2.039 (5)	N1—C1	1.348 (8)
Ir—N2	2.048 (5)	N1—C5	1.358 (8)
Ir—O1	2.123 (4)	N2—C22	1.329 (8)
Ir—O2	2.144 (4)	N2—C18	1.383 (7)
C12—Ir—C11	94.5 (2)	N1—Ir—O1	94.1 (2)
C12—Ir—N1	93.6 (2)	N2—Ir—O1	88.8 (2)
C11—Ir—N1	80.7 (2)	C12—Ir—O2	173.8 (2)
C12—Ir—N2	81.3 (2)	C11—Ir—O2	90.1 (2)
C11—Ir—N2	96.8 (2)	N1—Ir—O2	91.3 (2)
N1—Ir—N2	174.2 (2)	N2—Ir—O2	94.0 (2)
C12—Ir—O1	90.2 (2)	O1—Ir—O2	85.6 (2)
C11—Ir—O1	173.2 (2)		

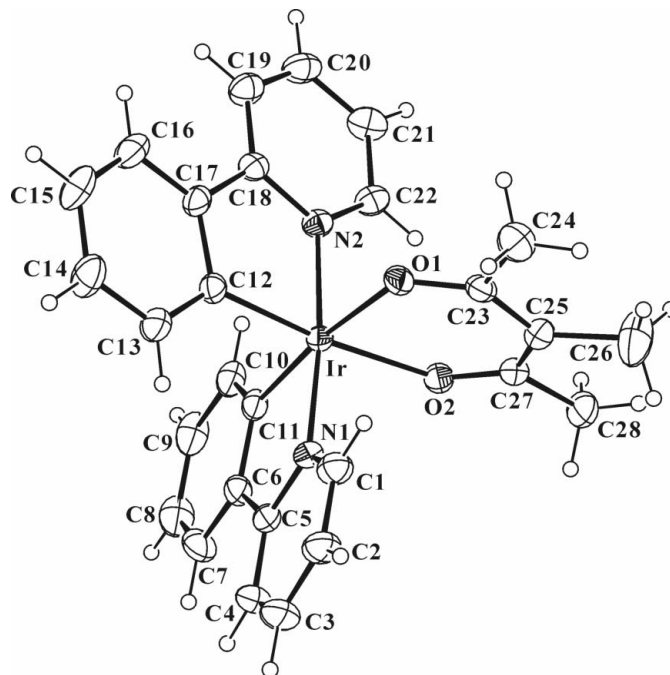


Figure 1

The molecular structure of (I), shown with 30% probability displacement ellipsoids.

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms and 0.96 Å for methyl H atoms, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms, and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms. The highest peak is located 0.83 Å from atom H8.

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

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