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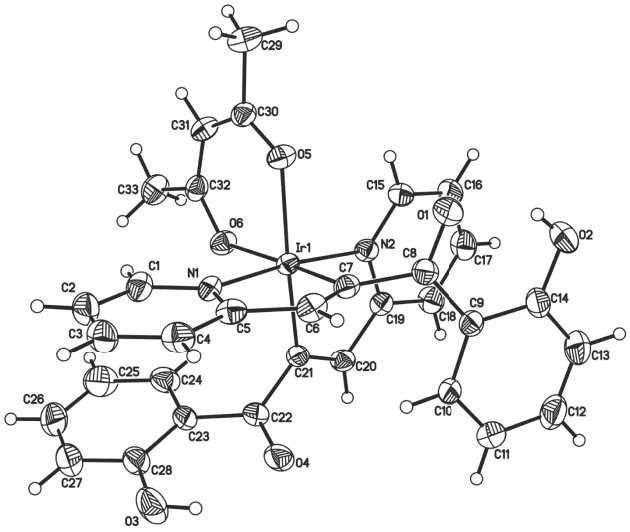
Yun-Lai Ren*, Wen-Hui Wang, Bin-Yu Wang and Zhi-Qiang Wang*

# Crystal structure of (acetylacetonato-κ²*O*:*O*′)bis-((1-(2-hydroxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one)-κ²*C*,*N*)iridium(III), $C_{33}H_{27}IrN_2O_6$

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## Abstract

$C_{33}H_{27}IrN_2O_6$, Triclinic, P-1, $a = 7.4840(11)$ Å, $b = 11.7312(17)$ Å, $c = 17.438(2)$ Å, $V = 1427.0(3)$ Å$^3$, $Z = 2$, $R_{gt}(F) = 0.0280$, $wR_{ref}(F^2) = 0.0677$, $T = 293(2)$ K.

**CCDC no.:** 1267/4362

## Source of material

1-(2-hydroxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one (44.5 mg, 0.2 mmol), acetylacetone (10 mg, 0.1 mmol), and iridium

***Corresponding authors: Yun-Lai Ren,** School of Chemical Engineering and Pharmaceutics, Henan University of Science and Technology, Luoyang, Henan 471003, P. R. China, e-mail: renyunlai2@126.com; and **Zhi-Qiang Wang,** College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, Henan 471022, P. R. China, e-mail: wzq197811@163.com
**Wen-Hui Wang and Bin-Yu Wang:** School of Chemical Engineering and Pharmaceutics, Henan University of Science and Technology, Luoyang, Henan 471003, P. R. China.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Brown, block, size 0.10 × 0.15 × 0.35 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 47.27 cm$^{-1}$ |
| Diffractometer, scan mode: | CCD area detector, $\varphi$ and $\omega$ scans |
| $2\theta_{max}$: | 51° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 10813, 5227 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\sigma(I_{obs})$, 4810 |
| $N(param)_{refined}$: | 383 |
| Programs: | SHELXS-97 (SHELDRICK, 1990) |

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2D) | 2*i* | −0.2367 | 0.4703 | 0.1656 | 0.103 |
| H(3) | 2*i* | 0.4549 | −0.1348 | 0.5031 | 0.115 |
| H(1) | 2*i* | 0.1832 | −0.253 | 0.2994 | 0.061 |
| H(2) | 2*i* | 0.1161 | −0.3793 | 0.4108 | 0.072 |
| H(3A) | 2*i* | −0.0429 | −0.298 | 0.5083 | 0.069 |
| H(4) | 2*i* | −0.1493 | −0.0877 | 0.4878 | 0.063 |
| H(6) | 2*i* | −0.1813 | 0.1321 | 0.39 | 0.055 |
| H(10) | 2*i* | 0.1929 | 0.2327 | 0.3509 | 0.056 |
| H(11) | 2*i* | 0.3925 | 0.3526 | 0.3525 | 0.074 |
| H(12) | 2*i* | 0.333 | 0.5411 | 0.267 | 0.086 |
| H(13) | 2*i* | 0.0756 | 0.6168 | 0.1797 | 0.08 |
| H(15) | 2*i* | −0.0386 | 0.2135 | 0.0721 | 0.055 |
| H(16) | 2*i* | 0.0437 | 0.3694 | −0.0144 | 0.066 |
| H(17) | 2*i* | 0.3229 | 0.4076 | 0.0081 | 0.07 |
| H(18) | 2*i* | 0.511 | 0.2932 | 0.1197 | 0.064 |
| H(20) | 2*i* | 0.5591 | 0.1143 | 0.2515 | 0.049 |
| H(24) | 2*i* | 0.5759 | −0.1713 | 0.261 | 0.069 |
| H(25) | 2*i* | 0.7041 | −0.3811 | 0.2807 | 0.098 |
| H(26) | 2*i* | 0.7137 | −0.5067 | 0.4038 | 0.102 |
| H(27) | 2*i* | 0.6122 | −0.428 | 0.5095 | 0.088 |
| H(29A) | 2*i* | −0.377 | −0.0711 | 0.118 | 0.094 |
| H(29B) | 2*i* | −0.289 | −0.064 | 0.0357 | 0.094 |
| H(29C) | 2*i* | −0.375 | 0.0559 | 0.0639 | 0.094 |
| H(31) | 2*i* | 0.0106 | −0.1687 | 0.0657 | 0.06 |
| H(33A) | 2*i* | 0.4514 | −0.2092 | 0.0687 | 0.093 |
| H(33B) | 2*i* | 0.3068 | −0.2825 | 0.0649 | 0.093 |
| H(33C) | 2*i* | 0.4311 | −0.31 | 0.1427 | 0.093 |

**Table 3:** Atomic displacement parameters (Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Ir(1) | 2*i* | 0.11713(2) | 0.02813(2) | 0.23166(1) | 0.0353(1) | 0.0352(1) | 0.0321(1) | −0.01200(7) | 0.00217(7) | −0.01038(7) |
| N(1) | 2*i* | 0.0575(5) | −0.0953(3) | 0.3256(2) | 0.039(2) | 0.035(2) | 0.043(2) | −0.015(2) | 0.003(2) | −0.012(2) |
| N(2) | 2*i* | 0.1799(5) | 0.1635(3) | 0.1471(2) | 0.042(2) | 0.041(2) | 0.030(2) | −0.013(2) | 0.004(2) | −0.011(2) |
| O(1) | 2*i* | −0.2529(5) | 0.3168(4) | 0.2136(3) | 0.052(2) | 0.058(2) | 0.090(3) | −0.007(2) | −0.023(2) | −0.014(2) |
| O(2) | 2*i* | −0.1712(6) | 0.5171(4) | 0.1593(3) | 0.079(3) | 0.051(2) | 0.062(3) | −0.002(2) | −0.017(2) | −0.001(2) |
| O(3) | 2*i* | 0.4741(7) | −0.2093(4) | 0.5138(2) | 0.105(3) | 0.063(3) | 0.045(2) | −0.010(3) | 0.008(2) | 0.002(2) |
| O(4) | 2*i* | 0.3908(6) | 0.0063(4) | 0.4183(2) | 0.084(3) | 0.054(2) | 0.039(2) | −0.001(2) | −0.009(2) | −0.017(2) |
| O(5) | 2*i* | −0.1211(4) | 0.0275(3) | 0.1653(2) | 0.041(2) | 0.054(2) | 0.050(2) | −0.017(2) | 0.001(2) | −0.021(2) |
| O(6) | 2*i* | 0.2809(4) | −0.1033(3) | 0.1750(2) | 0.045(2) | 0.049(2) | 0.047(2) | −0.012(2) | 0.004(2) | −0.022(2) |
| C(1) | 2*i* | 0.1156(7) | −0.2190(5) | 0.3375(3) | 0.053(3) | 0.045(3) | 0.058(3) | −0.020(2) | 0.005(2) | −0.012(2) |
| C(2) | 2*i* | 0.0767(8) | −0.2950(5) | 0.4044(4) | 0.064(3) | 0.047(3) | 0.066(4) | −0.023(3) | −0.002(3) | 0.001(3) |
| C(3) | 2*i* | −0.0199(8) | −0.2471(5) | 0.4620(3) | 0.062(3) | 0.061(4) | 0.048(3) | −0.029(3) | 0.006(3) | 0.003(3) |
| C(4) | 2*i* | −0.0820(7) | −0.1220(5) | 0.4498(3) | 0.055(3) | 0.064(4) | 0.040(3) | −0.024(3) | 0.009(2) | −0.009(2) |
| C(5) | 2*i* | −0.0445(7) | −0.0465(5) | 0.3805(3) | 0.041(3) | 0.056(3) | 0.037(3) | −0.020(2) | 0.004(2) | −0.014(2) |
| C(6) | 2*i* | −0.1050(7) | 0.0870(5) | 0.3589(3) | 0.043(3) | 0.053(3) | 0.047(3) | −0.015(2) | 0.010(2) | −0.020(2) |
| C(7) | 2*i* | −0.0433(6) | 0.1398(4) | 0.2917(3) | 0.034(2) | 0.041(3) | 0.042(3) | −0.010(2) | 0.001(2) | −0.016(2) |
| C(8) | 2*i* | −0.1062(6) | 0.2736(4) | 0.2557(3) | 0.041(3) | 0.043(3) | 0.043(3) | −0.005(2) | 0.001(2) | −0.015(2) |
| C(9) | 2*i* | 0.0142(6) | 0.3515(4) | 0.2621(3) | 0.046(3) | 0.038(2) | 0.038(2) | −0.007(2) | 0.004(2) | −0.015(2) |
| C(10) | 2*i* | 0.1693(7) | 0.3092(5) | 0.3158(3) | 0.062(3) | 0.037(3) | 0.042(3) | −0.014(2) | −0.003(2) | −0.012(2) |
| C(11) | 2*i* | 0.2885(8) | 0.3812(5) | 0.3171(4) | 0.065(4) | 0.056(3) | 0.063(4) | −0.014(3) | −0.011(3) | −0.019(3) |
| C(12) | 2*i* | 0.252(1) | 0.4938(6) | 0.2662(4) | 0.083(5) | 0.048(3) | 0.091(5) | −0.029(3) | 0.004(4) | −0.019(3) |
| C(13) | 2*i* | 0.0983(9) | 0.5394(5) | 0.2137(4) | 0.084(5) | 0.043(3) | 0.070(4) | −0.021(3) | 0.001(3) | −0.002(3) |
| C(14) | 2*i* | −0.0219(8) | 0.4693(5) | 0.2119(3) | 0.063(3) | 0.038(3) | 0.046(3) | −0.005(2) | 0.001(2) | −0.008(2) |
| C(15) | 2*i* | 0.0722(7) | 0.2301(5) | 0.0821(3) | 0.052(3) | 0.047(3) | 0.037(3) | −0.012(2) | −0.004(2) | −0.009(2) |
| C(16) | 2*i* | 0.1218(8) | 0.3229(5) | 0.0298(3) | 0.073(4) | 0.051(3) | 0.037(3) | −0.015(3) | −0.003(2) | −0.004(2) |
| C(17) | 2*i* | 0.2870(8) | 0.3461(5) | 0.0434(3) | 0.072(4) | 0.052(3) | 0.048(3) | −0.025(3) | 0.013(3) | 0.001(3) |
| C(18) | 2*i* | 0.3990(8) | 0.2779(5) | 0.1097(3) | 0.053(3) | 0.054(3) | 0.051(3) | −0.023(3) | 0.006(2) | −0.002(2) |
| C(19) | 2*i* | 0.3439(6) | 0.1853(4) | 0.1622(3) | 0.042(2) | 0.041(3) | 0.037(2) | −0.015(2) | 0.006(2) | −0.011(2) |
| C(20) | 2*i* | 0.4428(6) | 0.1103(4) | 0.2359(3) | 0.036(2) | 0.046(3) | 0.043(3) | −0.016(2) | −0.000(2) | −0.009(2) |
| C(21) | 2*i* | 0.3549(6) | 0.0343(4) | 0.2805(3) | 0.034(2) | 0.035(2) | 0.040(2) | −0.006(2) | −0.002(2) | −0.012(2) |
| C(22) | 2*i* | 0.4234(6) | −0.0400(4) | 0.3619(3) | 0.037(2) | 0.046(3) | 0.039(3) | −0.010(2) | −0.001(2) | −0.010(2) |
| C(23) | 2*i* | 0.5163(6) | −0.1727(4) | 0.3737(3) | 0.037(2) | 0.039(3) | 0.048(3) | −0.006(2) | −0.007(2) | −0.006(2) |
| C(24) | 2*i* | 0.5823(7) | −0.2223(5) | 0.3113(3) | 0.051(3) | 0.059(3) | 0.055(3) | 0.001(3) | −0.007(2) | −0.020(3) |
| C(25) | 2*i* | 0.659(1) | −0.3482(7) | 0.3229(5) | 0.075(4) | 0.074(5) | 0.090(5) | 0.009(3) | −0.010(4) | −0.042(4) |
| C(26) | 2*i* | 0.666(1) | −0.4226(6) | 0.3964(5) | 0.089(5) | 0.041(3) | 0.112(6) | 0.000(3) | −0.029(4) | −0.014(4) |
| C(27) | 2*i* | 0.6046(9) | −0.3758(6) | 0.4596(4) | 0.079(4) | 0.050(4) | 0.079(5) | −0.016(3) | −0.015(4) | 0.002(3) |
| C(28) | 2*i* | 0.5310(8) | −0.2510(5) | 0.4492(4) | 0.051(3) | 0.050(3) | 0.060(3) | −0.012(2) | −0.008(3) | −0.003(3) |
| C(29) | 2*i* | −0.3074(8) | −0.0290(6) | 0.0807(4) | 0.054(3) | 0.079(4) | 0.066(4) | −0.030(3) | −0.004(3) | −0.026(3) |
| C(30) | 2*i* | −0.1199(7) | −0.0417(5) | 0.1195(3) | 0.049(3) | 0.045(3) | 0.039(3) | −0.022(2) | 0.001(2) | −0.008(2) |
| C(31) | 2*i* | 0.0342(7) | −0.1251(5) | 0.1007(3) | 0.057(3) | 0.052(3) | 0.050(3) | −0.022(3) | 0.000(2) | −0.024(2) |
| C(32) | 2*i* | 0.2189(7) | −0.1523(4) | 0.1272(3) | 0.056(3) | 0.038(3) | 0.040(3) | −0.016(2) | 0.008(2) | −0.014(2) |
| C(33) | 2*i* | 0.3654(8) | −0.2471(5) | 0.0982(4) | 0.067(4) | 0.057(3) | 0.069(4) | −0.015(3) | 0.014(3) | −0.031(3) |

trichloride hydrate (35.3 mg, 0.1 mmol) were dissolved in 20 mL methanol. The mixture was stirred at room temperature for 5 hours. After filtration, the clear solution was left to stand in air for one week to give crystals suitable for X-ray diffraction.

## Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times $U_{eq}$(C, aromatic ring), and $U_{iso}(H) = 1.5$ times $U_{eq}$(C, methyl). The final refinement were made by using geometrical restraints, with C–H = 0.93 Å (aromatic ring), and C–H = 0.96 Å (methyl).

## Discussion

In the last decade, organometallic complexes based on Ir(III) have attracted a great deal of attention for their application in organic light-emitting diodes (OLEDs) [1–3]. It is well known that the ligands can affect the quantum yield of iridium(III) complex. So it is important to select applicable ligands to synthesize excellent Ir(III) organometallic complexes as OLEDs materials. In this paper, we report a new Ir(III)

organometallic complex based on 1-(2-hydroxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one and acetylacetone. The asymmetric unit of the title structure consists of one Ir(III), one acetylacetonate anion, and two 1-(2-hydroxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one anions (Figure). The Ir atom is coordinated by two nitrogen and two carbon donor atoms from two 1-(2-hydroxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one ligands, and two oxygen atoms from one acetylacetonato ligand, to complete a distorted octahedral coordination environment. The Ir—C bond lengths are 2.005(4) Å, the Ir—N bond lengths are 2.048(4) and 2.050(4) Å, and the Ir—O bond lengths are 2.126(3) and 2.136(3) Å, respectively. The bond angles around the Ir(III) are in the ranges of 78.75(17)°–174.91(16)°. For each 1-(2-hydroxyphenyl)-3-(pyridin-2-yl)prop-2-en-1-one ligand, the dihedral angle of the benzene ring and the pyridine ring are 85.36(8)° and 88.82(5)°, respectively.

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