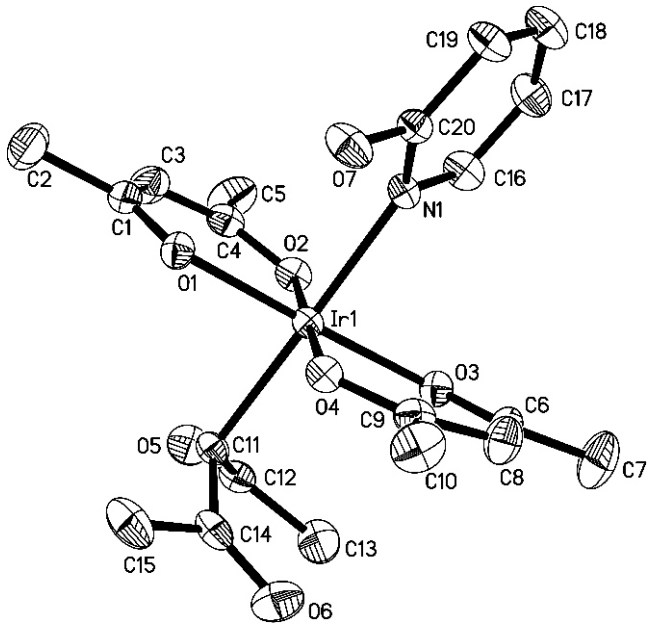
Crystal structure of bis(acetylacetonato-κ^2O,O')-(diacetylmethanido-κC)-(2-hydroxypyridine)iridium(III), $Ir(C_5H_7O_2)_3(C_5H_5NO)$

Qiao-Wen Chang, Chang-Yi Hu, Jia-Lin Chen, Qing-Song Ye, Xi-Zhu Chen, Yao Yu, Li-Qiao Chen and Wei-Ping Liu*

State Key Laboratory of Advanced Technologies for Platinum Metals, Kunming Institute of Precious Metal, Kunming 650106, P. R. China

Received December 24, 2010, accepted and available on-line June 17, 2011; CCDC no. 1267/3344



Abstract

$C_{20}H_{26}IrNO_7$, orthorhombic, *Pbca* (no. 61), a = 9.4798(8) Å, b = 14.646(1) Å, c = 31.842(3) Å, V = 4421.0 Å^3, Z = 8, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.074, T = 293 K.

Source of material

The title complex was prepared by mixing the aqueous solution of aquabis(acetylacetonato-κ^2O,O')(diacetylmethanido-κC)-iridium(III) and equimolar amount of 2-hydroxypyridine. After one week, a yellow crystalline product precipitated. Single crystals were selected from the product for the X-ray diffraction analysis.

Experimental details

All the hydrogen atoms were positioned geometrically with d(C—H) = 0.93 - 0.98 Å, d(O—H) = 0.82 Å and refined as riding with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C) and U_{iso}(H) = 1.5 U_{eq}(O).

Discussion

In the title complex, the Ir atom is six-coordinated and situated in a slightly distorted octahedral environment, formed by four oxygen atoms of two acetylacetone ligands, one carbon atom of one acetylacetone ligand and one N atom of 2-hydroxypyridine. The average O–Ir–O chelating angle is 94.1(1)°. The average Ir—O bond length of two acetylacetone ligands is 2.023(3) Å, Ir—C bond length is found to be 2.162(5) Å, which agree with the literature data of $Ir(C_5H_7O_2)_3$, $Ir(C_5H_7O_2)_3(H_2O)$, $Ir(C_5H_7O_2)_3(C_2H_6SO)$ and $Ir(C_5H_7O_2)_3(CH_3CN) \cdot 1.5H_2O$ [1-5]. Ir—N bond length is found to be 2.140(4) Å. Changes in the coordination mode of one acetylacetonate ligand of tris(acetyacetonato-O,O)iridium(III) lead to the formation of a direct Ir—C bond. The average Ir—O, Ir—C and Ir—N bond lengths are close to each other. Ir—C bond length is found to be between Ir—C σ-bond (2.00 - 2.02 Å) and Ir—C π-bond (2.27 - 2.31 Å). In the crystal structure of the title complex there are two different types of coordinating acac ligands: a conventional bidentate (O-bonded acac ligand) and a γ-C bonded acac ligand, as Periana proposed [3]. The crystal packing of the title complex is stablilized by extensive hydrogen bonds formed between 2-hydroxypyridine and carbonyl O atoms of the acetylacetone ligand with d(O7–H7···O5) = 2.618(5) Å and ∠O7–H7···O5 = 165.9°.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.12 × 0.15 × 0.19 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	60.79 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	28671, 5364
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3751
$N(param)_{refined}$:	269
Programs:	SHELXS-97, SHELXL-97 [6], DIAMOND [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(7)	8*c*	0.1448	0.4073	0.6475	0.072
H(2A)	8*c*	0.5749	0.5605	0.7659	0.093
H(2B)	8*c*	0.4403	0.4994	0.7710	0.093
H(2C)	8*c*	0.4429	0.5772	0.7373	0.093
H(3)	8*c*	0.6736	0.3963	0.7603	0.061
H(5A)	8*c*	0.8133	0.2073	0.7118	0.099
H(5B)	8*c*	0.7781	0.2484	0.7560	0.099
H(5C)	8*c*	0.9040	0.2878	0.7298	0.099
H(7A)	8*c*	0.6912	0.2567	0.5108	0.107
H(7B)	8*c*	0.5368	0.2539	0.4932	0.107
H(7C)	8*c*	0.5805	0.1894	0.5302	0.107
H(8)	8*c*	0.4030	0.3751	0.5125	0.057
H(10A)	8*c*	0.2026	0.5107	0.5569	0.081
H(10B)	8*c*	0.2862	0.5127	0.5144	0.081
H(10C)	8*c*	0.3214	0.5836	0.5498	0.081

* Correspondence author (e-mail: liuweiping0917@126.com)

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11)	8*c*	0.7555	0.5265	0.6508	0.039
H(13A)	8*c*	1.0150	0.3747	0.5808	0.089
H(13B)	8*c*	0.9263	0.4491	0.5572	0.089
H(13C)	8*c*	0.8568	0.3556	0.5694	0.089
H(15A)	8*c*	0.6990	0.6919	0.6098	0.089
H(15B)	8*c*	0.5860	0.6315	0.6326	0.089

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(15C)	8*c*	0.5621	0.6617	0.5860	0.089
H(16)	8*c*	0.5462	0.1989	0.6398	0.053
H(17)	8*c*	0.3963	0.0799	0.6474	0.061
H(18)	8*c*	0.1575	0.1079	0.6580	0.064
H(19)	8*c*	0.0788	0.2567	0.6581	0.055

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ir(1)	8*c*	0.57132(2)	0.39919(1)	0.633268(6)	0.0231(1)	0.0273(1)	0.0325(1)	0.00097(9)	–0.00159(8)	0.00115(9)
N(1)	8*c*	0.4088(4)	0.3007(3)	0.6443(1)	0.029(2)	0.027(2)	0.037(2)	–0.000(2)	–0.004(2)	0.001(2)
O(1)	8*c*	0.5125(3)	0.4787(2)	0.6820(1)	0.029(2)	0.037(2)	0.036(2)	0.003(2)	–0.001(2)	–0.005(2)
O(2)	8*c*	0.6984(3)	0.3185(2)	0.6683(1)	0.029(2)	0.037(2)	0.043(2)	0.004(2)	–0.006(2)	0.007(2)
O(3)	8*c*	0.6220(3)	0.3180(2)	0.5838(1)	0.033(2)	0.035(2)	0.038(2)	0.005(2)	–0.000(2)	–0.003(2)
O(4)	8*c*	0.4408(3)	0.4786(2)	0.5991(1)	0.028(2)	0.031(2)	0.038(2)	0.003(2)	–0.003(2)	0.002(2)
O(5)	8*c*	0.9569(4)	0.4304(3)	0.6506(1)	0.032(2)	0.064(3)	0.066(3)	0.003(2)	–0.002(2)	0.003(2)
O(6)	8*c*	0.7298(5)	0.5475(3)	0.5541(1)	0.076(3)	0.072(3)	0.049(2)	0.004(3)	0.003(2)	0.016(2)
O(7)	8*c*	0.2300(4)	0.4036(3)	0.6515(1)	0.030(2)	0.034(2)	0.079(3)	0.002(2)	0.005(2)	0.001(2)
C(1)	8*c*	0.5563(5)	0.4655(4)	0.7195(2)	0.040(3)	0.043(3)	0.036(3)	–0.010(3)	0.006(3)	–0.004(2)
C(2)	8*c*	0.4984(7)	0.5316(5)	0.7513(2)	0.081(5)	0.062(5)	0.044(3)	0.006(4)	0.007(3)	–0.010(3)
C(3)	8*c*	0.6503(6)	0.3968(4)	0.7319(2)	0.057(4)	0.059(4)	0.036(3)	0.000(4)	–0.005(3)	0.007(3)
C(4)	8*c*	0.7125(5)	0.3302(4)	0.7079(2)	0.032(3)	0.043(4)	0.045(3)	–0.003(3)	–0.005(2)	0.010(3)
C(5)	8*c*	0.8109(7)	0.2623(5)	0.7282(2)	0.066(4)	0.084(5)	0.050(4)	0.029(4)	–0.006(3)	0.016(4)
C(6)	8*c*	0.5502(6)	0.3211(4)	0.5492(2)	0.050(4)	0.047(4)	0.033(3)	–0.006(3)	–0.002(3)	–0.006(3)
C(7)	8*c*	0.5936(7)	0.2487(5)	0.5180(2)	0.096(6)	0.064(5)	0.055(4)	0.027(4)	–0.011(4)	–0.022(4)
C(8)	8*c*	0.4462(6)	0.3836(4)	0.5385(2)	0.049(3)	0.051(4)	0.042(3)	0.009(3)	–0.012(3)	–0.006(3)
C(9)	8*c*	0.3996(5)	0.4572(4)	0.5622(2)	0.027(3)	0.044(3)	0.040(3)	0.000(2)	–0.002(2)	0.006(3)
C(10)	8*c*	0.2928(6)	0.5219(4)	0.5442(2)	0.050(4)	0.061(4)	0.052(3)	0.015(3)	–0.008(3)	0.011(3)
C(11)	8*c*	0.7463(5)	0.4920(4)	0.6246(1)	0.026(3)	0.032(3)	0.041(3)	–0.004(2)	–0.001(2)	0.000(2)
C(12)	8*c*	0.8779(6)	0.4412(4)	0.6194(2)	0.033(3)	0.037(3)	0.048(3)	–0.003(3)	0.003(3)	0.005(3)
C(13)	8*c*	0.9231(6)	0.4016(4)	0.5780(2)	0.041(3)	0.062(4)	0.075(4)	0.003(3)	0.015(3)	–0.013(4)
C(14)	8*c*	0.7052(6)	0.5595(4)	0.5914(2)	0.038(3)	0.035(3)	0.060(4)	–0.012(3)	–0.001(3)	0.000(3)
C(15)	8*c*	0.6315(6)	0.6437(4)	0.6063(2)	0.048(4)	0.034(4)	0.095(5)	–0.001(3)	0.001(4)	0.002(4)
C(16)	8*c*	0.4508(6)	0.2111(4)	0.6437(2)	0.034(3)	0.042(4)	0.056(4)	0.005(3)	–0.004(2)	0.006(3)
C(17)	8*c*	0.3621(7)	0.1394(4)	0.6483(2)	0.054(4)	0.029(3)	0.068(4)	0.000(3)	–0.004(3)	0.003(3)
C(18)	8*c*	0.2205(6)	0.1559(4)	0.6543(2)	0.051(4)	0.038(4)	0.070(4)	–0.013(3)	–0.004(3)	0.007(3)
C(19)	8*c*	0.1745(6)	0.2446(4)	0.6547(2)	0.034(3)	0.045(4)	0.060(4)	–0.008(3)	–0.001(3)	0.005(3)
C(20)	8*c*	0.2689(5)	0.3170(4)	0.6502(2)	0.031(3)	0.031(3)	0.034(3)	0.000(2)	–0.002(2)	0.004(2)

Acknowledgments. This work was financially supported by Science and Technology Development Program of Yunnan Province (grant nos. 2008IA008, 2008CF002, 2009CD133 and 2009CI019).

References

1. Isakova, V. G.; Baidina, I. A.; Morozova, N. B.; Igumenov, I. K.: γ-Halogenated iridium(III)acetylacetonates. Polyhedron **19** (2000) 1097-1103.
2. Chang, Q. W.; Xie, M. J.; Liu, W. P.; Chen, X. Z.; Ye, Q. S.: Bis-(acetylacetonato-2*O*,*O'*)aqua(diacetylmethanido-*C*)iridium(III). Acta Crystallogr. **E65** (2009) m1264.
3. Matsumoto, T.; Periana, R. A.; Taube, D. J.; Yoshida, H.: Regioselective hydrophenylation of olefins catalyzed by an Ir(III) complex. J. Mol. Catal. **A180** (2002) 1-18.
4. Jiang, J.; Xie, M. J.; Chang, Q. W.; Chen, J. L.; Xie, X. T.; Ye, Q. S.; Chen, X. Z.; Yu, Y.; Liu, W. P.: Crystal structure of bis(acetylacetonato-κ^2*O*,*O'*)-(diacetylmethanido-κ*C*)-(dimethylsulfoxide-*S*)iridium(III), $Ir(C_5H_7O_2)_3(C_2H_6SO)$. Z. Kristallogr. NCS **225** (2010) 575-576.
5. Chang, Q. W.; Hu, C, Y.; Pan, Z. F.; Chen, J. L.; Ye, Q. S.; Chen, X. Z.; Yu, Y.; Liu, W. P.: Crystal structure of bis(acetylacetonato-κ^2*O*,*O'*)-(diacetylmethanido-κ*C*)-(acetonitrile-*N*)iridium(III) sesquihydrate, $Ir(C_5H_7O_2)_3(CH_3CN)\cdot 1.5H_2O$. Z. Kristallogr. NCS **225** (2010) 667-668.
6. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
7. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2c. Crystal Impact, Bonn, Germany 1998.