Crystal structure of bis(acetylacetonato-κ^2O,O')-(diacetylmethanido-κC)-(3-hydroxypyridine)iridium(III) dihydrate, $Ir(C_5H_7O_2)_3(C_5H_5NO) \cdot 2H_2O$

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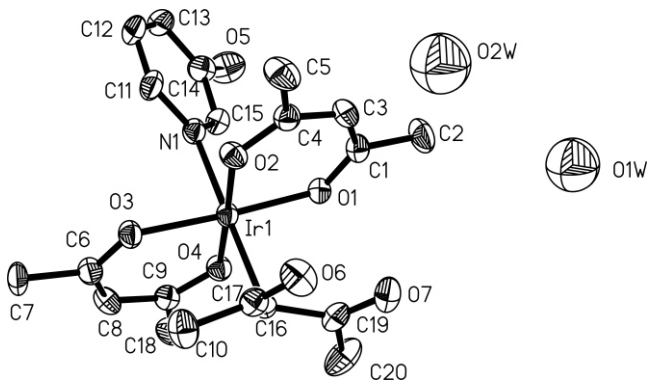

Abstract

$C_{20}H_{30}IrNO_9$, monoclinic, $P2_1/n$ (no. 14), a = 10.574(1) Å, b = 18.662(2) Å, c = 11.888(1) Å, β = 94.178(1)°, V = 2339.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.091, T = 298 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 3-hydroxypyridine. After two weeks, a yelleow crystalline product precipitated. Single crystals were selected from the product for the X-ray diffraction analysis.

Experimental details

The carbon-bound H atoms were positioned geometrically and refined as riding with d(C—H) = 0.93 - 0.98 Å and U_{iso}(H) = 1.2 U_{eq}(C). The oxygen atoms of water are refined isotropically.

Discussion

The asymmetric unit of the title crystal structure consists of a Ir(III) cation, three acetylacetonate anions, one 3-hydroypyridine molecule and two lattice water molecules. The Ir(III) cation is six-coordinated by four oxygen atoms of different acetyacetonate anions, one carbon atom of acetylacetonate anion and one N atom of 3-hydroxypyridine ligand in a slightly distorted octahedral environment. The average O–Ir–O chelating angle is 89.97°. The average Ir—O bond length is 2.016(4) Å, Ir—C bond length is 2.166(6) Å, which agree with the literature data for $Ir(C_5H_7O_2)_3$, $Ir(C_5H_7O_2)_3(H_2O)$ and $Ir(C_5H_7O_2)_3(C_2H_6SO)$ [1-5]. Ir—N bond length is found to be 2.088(5) Å.

In the crystal structure, there are intermolecular hydrogen bonds with d(O5–H5···O1W) = 2.593(8) Å, ∠O5–H5···O1W = 138.1°; d(O1W–H1W···O2W) = 3.101(9) Å, ∠O1W–H1W···O2W = 121.2°; d(O1W–H2W···O6) = 2.850(8) Å, ∠O1W–H2W···O6 = 130.5° and d(O1W–H2W···O7) = 3.019(9) Å, ∠O1W–H2W···O7 = 139.5°.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.12 × 0.14 × 0.19 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	57.55 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II CCD, φ/ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15209, 5290
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3644
$N(param)_{refined}$:	277
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(5)	4*e*	–0.4142	0.0122	0.2191	0.100
H(2A)	4*e*	–0.3341	0.3666	0.0677	0.104
H(2B)	4*e*	–0.3472	0.3659	0.1982	0.104
H(2C)	4*e*	–0.3901	0.2992	0.1250	0.104
H(3)	4*e*	–0.1534	0.3611	0.2915	0.061
H(5A)	4*e*	0.1569	0.3129	0.3911	0.103
H(5B)	4*e*	0.0290	0.3310	0.4437	0.103
H(5C)	4*e*	0.0908	0.3870	0.3661	0.103
H(7A)	4*e*	0.3735	0.0981	0.0584	0.090
H(7B)	4*e*	0.3182	0.0269	0.0047	0.090
H(7C)	4*e*	0.3071	0.0423	0.1333	0.090
H(8)	4*e*	0.1305	0.0396	–0.1012	0.064
H(10A)	4*e*	–0.0720	0.0947	–0.2631	0.094
H(10B)	4*e*	–0.1811	0.0725	–0.1877	0.094
H(10C)	4*e*	–0.0673	0.0202	–0.2020	0.094
H(11)	4*e*	0.0493	0.1243	0.3053	0.064
H(12)	4*e*	–0.0508	0.0488	0.4215	0.073
H(13)	4*e*	–0.2578	0.0142	0.3739	0.074
H(15)	4*e*	–0.2493	0.1289	0.0924	0.057
H(16)	4*e*	0.1121	0.2568	–0.0862	0.047
H(18A)	4*e*	0.3793	0.3191	0.0606	0.099
H(18B)	4*e*	0.3282	0.2728	–0.0429	0.099
H(18C)	4*e*	0.3168	0.2443	0.0801	0.099
H(20A)	4*e*	–0.1517	0.3412	–0.2111	0.129
H(20B)	4*e*	–0.1417	0.2620	–0.1674	0.129
H(20C)	4*e*	–0.0344	0.2941	–0.2366	0.129
O(1W)	4*e*	0.4827(6)	0.9880(4)	0.3404(5)	0.110(2)
H(1W)	4*e*	0.4110	0.9978	0.3069	0.131
H(2W)	4*e*	0.4822	0.9462	0.3691	0.131
O(2W)	4*e*	0.7816(6)	0.9716(4)	0.5816(6)	0.129(3)
H(3W)	4*e*	0.8185	0.9595	0.6446	0.155
H(4W)	4*e*	0.8147	1.0035	0.5421	0.155

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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)	4*e*	–0.00518(2)	0.20807(1)	0.08562(2)	0.0360(1)	0.0362(2)	0.0368(2)	0.0049(1)	–0.00208(9)	–0.0027(1)
N(1)	4*e*	–0.0905(5)	0.1343(3)	0.1883(4)	0.042(3)	0.042(3)	0.040(3)	0.005(2)	–0.002(2)	–0.002(2)
O(1)	4*e*	–0.1715(4)	0.2609(2)	0.0774(3)	0.040(2)	0.037(3)	0.043(3)	0.003(2)	0.002(2)	0.000(2)
O(2)	4*e*	0.0667(4)	0.2588(3)	0.2257(3)	0.041(2)	0.053(3)	0.042(3)	0.002(2)	0.001(2)	–0.010(2)
O(3)	4*e*	0.1570(4)	0.1507(2)	0.0995(3)	0.044(2)	0.049(3)	0.043(3)	0.011(2)	–0.004(2)	–0.003(2)
O(4)	4*e*	–0.0778(4)	0.1538(2)	–0.0510(3)	0.046(3)	0.046(3)	0.043(3)	0.002(2)	0.001(2)	–0.005(2)
O(5)	4*e*	–0.3924(5)	0.0484(3)	0.1864(5)	0.082(4)	0.080(5)	0.089(4)	–0.030(3)	0.016(3)	0.014(3)
O(6)	4*e*	0.1788(5)	0.3773(3)	0.0878(4)	0.067(3)	0.059(4)	0.080(4)	–0.017(3)	0.005(3)	–0.023(3)
O(7)	4*e*	–0.0555(5)	0.3870(3)	–0.0329(4)	0.068(3)	0.056(4)	0.079(4)	0.015(3)	–0.001(3)	0.009(3)
C(1)	4*e*	–0.1975(6)	0.3060(3)	0.1526(6)	0.037(3)	0.036(4)	0.056(4)	0.001(3)	0.003(3)	0.001(3)
C(2)	4*e*	–0.3293(6)	0.3373(4)	0.1342(7)	0.048(4)	0.079(6)	0.082(6)	0.026(4)	0.004(4)	–0.008(5)
C(3)	4*e*	–0.1193(6)	0.3273(4)	0.2447(6)	0.058(4)	0.044(4)	0.052(4)	0.008(3)	0.011(3)	–0.011(3)
C(4)	4*e*	0.0039(6)	0.3051(3)	0.2769(5)	0.049(4)	0.042(4)	0.046(4)	0.000(3)	0.004(3)	0.001(3)
C(5)	4*e*	0.0768(7)	0.3369(5)	0.3787(6)	0.068(5)	0.081(6)	0.055(5)	0.003(4)	–0.001(4)	–0.029(4)
C(6)	4*e*	0.1811(6)	0.1001(4)	0.0323(6)	0.048(4)	0.045(4)	0.052(4)	0.004(3)	0.009(3)	0.001(3)
C(7)	4*e*	0.3063(6)	0.0636(4)	0.0596(6)	0.060(5)	0.052(5)	0.067(5)	0.029(4)	0.000(4)	0.001(4)
C(8)	4*e*	0.1001(6)	0.0771(4)	–0.0591(6)	0.052(4)	0.053(5)	0.056(5)	0.004(3)	0.013(3)	–0.016(3)
C(9)	4*e*	–0.0207(6)	0.1027(4)	–0.0958(5)	0.044(4)	0.038(4)	0.045(4)	–0.004(3)	0.004(3)	–0.002(3)
C(10)	4*e*	–0.0916(7)	0.0696(4)	–0.1962(6)	0.076(5)	0.062(5)	0.051(4)	–0.008(4)	0.004(4)	–0.021(4)
C(11)	4*e*	–0.0335(6)	0.1101(4)	0.2849(6)	0.054(4)	0.050(5)	0.055(4)	0.013(3)	–0.008(3)	0.010(3)
C(12)	4*e*	–0.0931(7)	0.0650(4)	0.3550(6)	0.069(5)	0.060(5)	0.052(5)	0.023(4)	–0.006(4)	0.013(4)
C(13)	4*e*	–0.2154(8)	0.0439(4)	0.3263(6)	0.083(6)	0.048(5)	0.055(5)	0.009(4)	0.015(4)	0.012(4)
C(14)	4*e*	–0.2742(7)	0.0673(4)	0.2263(6)	0.062(5)	0.041(4)	0.058(5)	–0.008(3)	0.013(4)	–0.004(3)
C(15)	4*e*	–0.2091(6)	0.1126(4)	0.1599(5)	0.049(4)	0.046(4)	0.047(4)	0.004(3)	0.001(3)	0.004(3)
C(16)	4*e*	0.0789(6)	0.2842(3)	–0.0244(5)	0.040(3)	0.041(4)	0.036(3)	0.002(3)	0.001(3)	–0.001(3)
C(17)	4*e*	0.1881(6)	0.3214(4)	0.0358(6)	0.045(4)	0.043(4)	0.051(4)	–0.007(3)	0.004(3)	0.003(3)
C(18)	4*e*	0.3143(6)	0.2863(4)	0.0332(7)	0.041(4)	0.060(5)	0.098(6)	–0.003(4)	0.001(4)	–0.005(4)
C(19)	4*e*	–0.0256(7)	0.3301(4)	–0.0746(6)	0.054(4)	0.047(5)	0.054(5)	–0.005(4)	–0.002(3)	0.013(4)
C(20)	4*e*	–0.0945(9)	0.3046(5)	–0.1821(7)	0.106(7)	0.079(7)	0.066(6)	–0.016(5)	–0.035(5)	0.019(5)

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