Crystal structure of amminebis(acetylacetonato-κ^2O,O')-(diacetylmethanido-κC)iridium(III), $Ir(NH_3)(C_5H_7O_2)_3$

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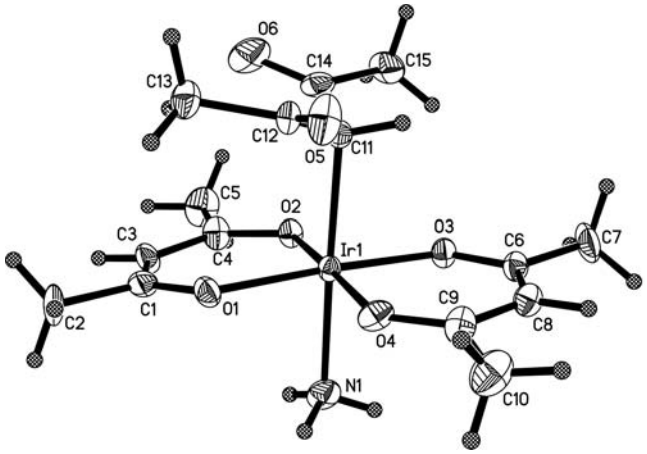

Abstract

$C_{15}H_{24}IrNO_6$, triclinic, $P\bar{1}$ (no. 2), a = 7.949(1) Å, b = 8.096(1) Å, c = 16.143(2) Å, α = 89.888(1)°, β = 78.378(1)°, γ = 61.607(1)°, V = 889.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.045, $wR_{ref}(F^2)$ = 0.185, T = 293 K.

Source of material

The titled complex was prepared by refluxing aquabis-(acetylacetonato-κ^2O,O')-(diacetylmethanido-κC)iridium(III) and a large excess of aqueous ammonia, and then the insoluble substance was filtered off. The filtrate was slowly cooled to room temperature along with evaporation and a yelleow crystalline product precipitated. Single crystals were selected from the product for the X-ray diffraction analysis.

Experimental details

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

Discussion

The Ir atom is six-coordinated and situated in a slightly distorted octahedral environment formed by four oxygen atoms of two acetyacetone ligands, one carbon atom of one acetylacetone ligand and one N atom of ammonia. The average O–Ir–O chelating angle is 89.98°. The average Ir—O bond length of two acetylacetone ligands is 2.02 Å, Ir—C bond length is found to be 2.18 Å, which agree with the literature data of $Ir(C_5H_7O_2)_3$ and $Ir(C_5H_7O_2)_3(H_2O)$ [1-3]. Ir—N bond length is found to be 2.13 Å. The molecular packing of the title crystal structure is stablilized by extensive hydrogen bonds formed between ammonia and carbonyl O atoms of the acetylacetone ligand with d(N···O) of 3.02(2) and 2.91(2) Å.

Table 1. Data collection and handling.

Crystal:	yellow irregular, size 0.08 × 0.15 × 0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	75.30 cm^{-1}
Diffractometer, scan mode:	Bruker APEX II CCD, φ/ω
$2\theta_{max}$:	56.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6267, 3699
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3223
$N(param)_{refined}$:	215
Programs:	SHELXS-97, SHELXL-97, SHELXTL [4], DIAMOND [5]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	1.2557	0.1378	0.3259	0.066
H(1B)	2*i*	1.2818	0.3032	0.3157	0.066
H(1C)	2*i*	1.3572	0.1587	0.2444	0.066
H(2A)	2*i*	0.7291	0.6811	0.5077	0.073
H(2B)	2*i*	0.8454	0.5020	0.5520	0.073
H(2C)	2*i*	0.6285	0.5642	0.5474	0.073
H(3)	2*i*	0.7765	0.2373	0.4863	0.047
H(5A)	2*i*	0.7564	–0.0367	0.3431	0.091
H(5B)	2*i*	0.7788	–0.0245	0.4369	0.091
H(5C)	2*i*	0.9642	–0.1469	0.3639	0.091
H(7A)	2*i*	1.2724	0.0316	0.0035	0.089
H(7B)	2*i*	1.3616	0.1527	–0.0422	0.089
H(7C)	2*i*	1.1423	0.2085	–0.0387	0.089
H(8)	2*i*	1.2592	0.4627	0.0239	0.042
H(10A)	2*i*	1.0694	0.8557	0.1528	0.091
H(10B)	2*i*	1.2386	0.7357	0.0735	0.091
H(10C)	2*i*	1.2827	0.7232	0.1644	0.091
H(11)	2*i*	0.7515	0.5218	0.1492	0.042
H(13A)	2*i*	0.4979	0.8193	0.3778	0.066
H(13B)	2*i*	0.4863	0.6344	0.3619	0.066
H(13C)	2*i*	0.3317	0.8260	0.3373	0.066
H(15A)	2*i*	0.7104	0.1210	0.1671	0.075
H(15B)	2*i*	0.8153	0.2171	0.1099	0.075
H(15C)	2*i*	0.5952	0.2783	0.1123	0.075

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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)	2*i*	0.98857(7)	0.36427(7)	0.25234(3)	0.0283(3)	0.0246(3)	0.0230(3)	–0.0148(2)	–0.0022(2)	–0.0013(2)
N(1)	2*i*	1.260(2)	0.220(2)	0.2901(9)	0.045(7)	0.044(8)	0.054(8)	–0.033(7)	–0.007(6)	0.000(6)
O(1)	2*i*	0.880(2)	0.506(2)	0.3710(6)	0.044(6)	0.042(6)	0.027(5)	–0.025(5)	–0.008(4)	–0.002(4)
O(2)	2*i*	0.942(1)	0.149(1)	0.2854(6)	0.041(5)	0.030(5)	0.028(5)	–0.025(4)	–0.005(4)	0.003(4)
O(3)	2*i*	1.110(2)	0.216(2)	0.1368(6)	0.044(6)	0.039(6)	0.028(5)	–0.031(5)	–0.003(4)	0.001(4)
O(4)	2*i*	1.052(2)	0.574(2)	0.2187(7)	0.039(4)	0.034(4)	0.051(4)	–0.029(3)	–0.008(3)	0.006(3)
O(5)	2*i*	0.608(2)	0.837(2)	0.2200(8)	0.065(8)	0.025(6)	0.046(7)	–0.010(6)	0.005(6)	0.011(5)
O(6)	2*i*	0.501(2)	0.403(2)	0.279(1)	0.053(8)	0.065(9)	0.09(1)	–0.047(7)	–0.008(7)	–0.001(7)
C(1)	2*i*	0.823(2)	0.436(2)	0.435(1)	0.038(8)	0.052(9)	0.036(8)	–0.034(7)	–0.009(6)	0.006(7)
C(2)	2*i*	0.750(3)	0.557(3)	0.5179(9)	0.06(1)	0.06(1)	0.020(7)	–0.028(9)	0.008(7)	–0.007(7)
C(3)	2*i*	0.819(2)	0.269(2)	0.4336(9)	0.053(9)	0.06(1)	0.022(7)	–0.042(9)	–0.010(6)	0.010(6)
C(4)	2*i*	0.870(2)	0.137(2)	0.363(1)	0.048(9)	0.025(7)	0.036(8)	–0.014(7)	–0.008(6)	0.011(6)
C(5)	2*i*	0.839(3)	–0.034(3)	0.378(1)	0.076(9)	0.052(8)	0.066(9)	–0.044(7)	–0.005(7)	0.011(7)
C(6)	2*i*	1.183(2)	0.277(2)	0.0725(9)	0.034(7)	0.032(7)	0.028(7)	–0.015(6)	–0.005(5)	0.007(6)
C(7)	2*i*	1.245(3)	0.157(3)	–0.008(1)	0.06(1)	0.07(1)	0.020(7)	–0.02(1)	–0.004(7)	–0.004(8)
C(8)	2*i*	1.200(2)	0.440(2)	0.075(1)	0.040(8)	0.030(7)	0.037(8)	–0.020(7)	–0.006(6)	0.010(6)
C(9)	2*i*	1.140(2)	0.576(2)	0.145(1)	0.036(8)	0.028(8)	0.040(8)	–0.004(6)	–0.003(6)	0.010(6)
C(10)	2*i*	1.187(3)	0.738(3)	0.133(2)	0.06(1)	0.05(1)	0.07(1)	–0.03(1)	–0.01(1)	0.02(1)
C(11)	2*i*	0.716(2)	0.514(2)	0.2102(9)	0.037(7)	0.055(9)	0.026(7)	–0.032(7)	–0.004(5)	–0.002(6)
C(12)	2*i*	0.599(2)	0.711(2)	0.2522(9)	0.034(8)	0.05(1)	0.024(7)	–0.017(7)	0.001(5)	–0.002(6)
C(13)	2*i*	0.466(2)	0.751(2)	0.340(1)	0.044(9)	0.038(9)	0.037(8)	–0.014(7)	0.001(6)	–0.003(6)
C(14)	2*i*	0.628(2)	0.388(2)	0.219(1)	0.031(7)	0.041(9)	0.051(9)	–0.022(7)	–0.013(6)	–0.001(7)
C(15)	2*i*	0.693(3)	0.237(2)	0.145(1)	0.06(1)	0.05(1)	0.05(1)	–0.025(8)	–0.017(8)	–0.013(8)

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