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Crystal structure of dibenzoylmethane-κ^2O,O'-bis[2-(2-naphthyl)-3-methoxy-6-methylpyridine-κ^2C^2,N]iridium(III), $Ir(C_{34}H_{28}N_2O_2)_2(C_{15}H_{11}O_2)$

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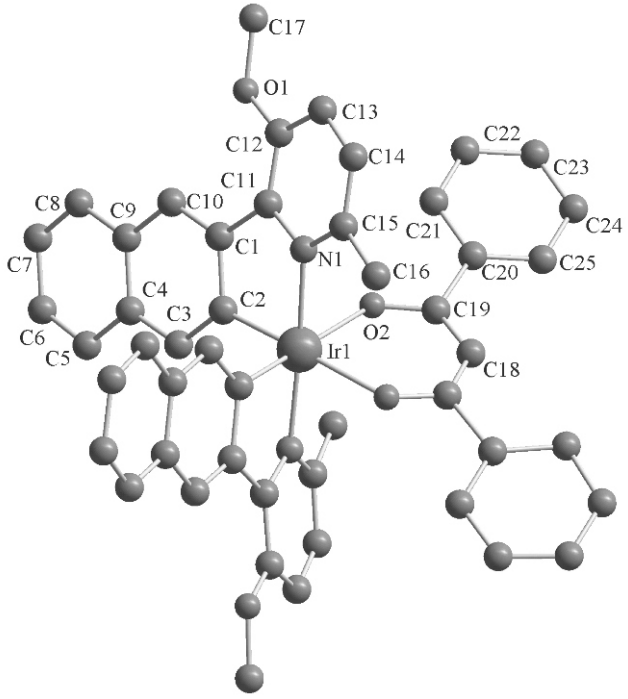

Abstract

$C_{49}H_{39}IrN_2O_4$, monoclinic, *C*12/*c*1 (no. 15), a = 12.2880(9) Å, b = 25.043(2) Å, c = 13.1937(9) Å, β = 102.469(1)°, V = 3964.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.022, $wR_{ref}(F^2)$ = 0.057, T = 296 K.

Source of material

The title compound was obtained from bridge-splitting reaction with dibenzoylmethane and the corresponding Cl-bridged dimers as described in literature [1], using dibenzoylmethane instead of acetylacetonate and recrystallized from dichloromethane-petroleum ether solution at room temperature to give the desired crystals suitable for single-crystal X-ray diffraction analysis.

Discussion

Cyclometalated Ir(III) complexes have emerged as a very interesting class of compounds for various applications [2-3]. They represent the most promising phosphorescent dopants for organic light-emitting diodes (OLED) because of their relatively short excited-state lifetime, high photoluminescence efficiency and excellent color tunability [4-6]. In recent years, various types of cyclometalated iridium(III) complexes have been developed. Compared to homoleptic complexes, the heteroleptic complexes can be synthesized under milder conditions by the reaction of Cl-bridged iridium(III) dimers with ancillary ligands [7-9]. β-Diketonates as ancillary ligands may be an effective way to improve the properties of bis-cyclometalated Ir(III) complexes.

The title compound adopts a distorted octahedral coordination around the iridium(III) center, and the benzene ring (C20-C25) displays rotational disorder with site occupancy factors of 0.64 and 0.36. The central iridium(III) atom is surrounded by two cyclometalating ligands and one ancillary ligand dibenzoylmethane with *cis*-C-C, *trans*-N-N and *cis*-O-O chelate positions. The benzene rings of dibenzoylmethane are disordered. The distance d(Ir—O) = 2.152(3) Å is similar to the reported values of other cyclometalated iridium(III) complexes [10,11]; d(Ir—C) = 1.979(3) Å is slightly longer than those of the corresponding cyclometalated Ir(III) dimers [1.957(11) Å and 1.969(10) Å], while d(Ir—N) = 2.072(3) Å is slightly shorter than those of the corresponding dimers [2.073(8)-2.087(8) Å].

Table 1. Data collection and handling.

Crystal:	brown block, size 0.15 × 0.31 × 0.42 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	34.16 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX-II CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14936, 3684
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3457
$N(param)_{refined}$:	251
Programs:	SHELXS-97, SHELXL-97, SHELXTL [12]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(21)	8*f*	0.64	0.4243	0.7301	1.0091	0.107
H(22)	8*f*	0.64	0.3921	0.7757	1.1529	0.142
H(23)	8*f*	0.64	0.3781	0.8681	1.1495	0.141
H(24)	8*f*	0.64	0.3963	0.9149	1.0024	0.125
H(25)	8*f*	0.64	0.4286	0.8694	0.8586	0.094
H(21')	8*f*	0.36	0.3514	0.7359	0.9639	0.107
H(22')	8*f*	0.36	0.2904	0.7839	1.0915	0.142
H(23')	8*f*	0.36	0.3183	0.8755	1.1046	0.141
H(24')	8*f*	0.36	0.4072	0.9191	0.9902	0.125
H(25')	8*f*	0.36	0.4683	0.8712	0.8626	0.094
H(3)	8*f*		0.3030	0.5703	0.7135	0.052
H(5)	8*f*		0.1466	0.5098	0.7381	0.075
H(6)	8*f*		0.0670	0.4585	0.8458	0.091
H(7)	8*f*		0.1500	0.4503	1.0210	0.085
H(8)	8*f*		0.3126	0.4943	1.0880	0.073

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Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(10)	8*f*		0.4621	0.5568	1.0650	0.056
H(13)	8*f*		0.7745	0.6644	1.1597	0.077
H(14)	8*f*		0.8437	0.6970	1.0234	0.074
H(16A)	8*f*		0.7421	0.6721	0.7623	0.098
H(16B)	8*f*		0.8449	0.6946	0.8425	0.098

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(16C)	8*f*		0.7391	0.7303	0.8051	0.098
H(17A)	8*f*		0.7250	0.5978	1.2575	0.174
H(17B)	8*f*		0.6135	0.5950	1.2968	0.174
H(17C)	8*f*		0.6611	0.6504	1.2722	0.174
H(18)	4*e*		½	0.8232	¾	0.075

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(20)	8*f*	0.64	0.430(1)	0.7952(7)	0.9196(7)	0.050(4)	0.058(3)	0.074(4)	0.001(3)	0.019(4)	–0.016(4)
C(21)	8*f*	0.64	0.4187(9)	0.7672(4)	1.0077(8)	0.101(9)	0.088(5)	0.093(8)	–0.012(6)	0.055(7)	–0.022(5)
C(22)	8*f*	0.64	0.399(1)	0.7945(4)	1.0939(8)	0.14(1)	0.122(7)	0.116(9)	–0.022(9)	0.079(8)	–0.054(7)
C(23)	8*f*	0.64	0.391(1)	0.8498(4)	1.092(1)	0.15(1)	0.099(6)	0.13(1)	0.017(7)	0.08(1)	–0.037(7)
C(24)	8*f*	0.64	0.402(1)	0.8779(4)	1.004(2)	0.100(4)	0.067(4)	0.146(9)	0.016(3)	0.026(5)	–0.042(4)
C(25)	8*f*	0.64	0.421(2)	0.8506(7)	0.918(1)	0.074(5)	0.064(3)	0.101(5)	0.011(3)	0.026(3)	–0.013(3)
Ir(1)	4*e*		½	0.651219(7)	¾	0.0463(1)	0.0367(1)	0.0280(1)	0	0.01221(7)	0
C(20')	8*f*	0.36	0.416(2)	0.799(1)	0.901(1)	0.050(4)	0.058(3)	0.074(4)	0.001(3)	0.019(4)	–0.016(4)
C(21')	8*f*	0.36	0.363(2)	0.7727(8)	0.969(1)	0.101(9)	0.088(5)	0.093(8)	–0.012(6)	0.055(7)	–0.022(5)
C(22')	8*f*	0.36	0.326(2)	0.8014(8)	1.046(2)	0.14(1)	0.122(7)	0.116(9)	–0.022(9)	0.079(8)	–0.054(7)
C(23')	8*f*	0.36	0.343(2)	0.8563(8)	1.053(2)	0.15(1)	0.099(6)	0.13(1)	0.017(7)	0.08(1)	–0.037(7)
C(24')	8*f*	0.36	0.396(3)	0.8824(7)	0.985(3)	0.100(4)	0.067(4)	0.146(9)	0.016(3)	0.026(5)	–0.042(4)
C(25')	8*f*	0.36	0.433(3)	0.854(1)	0.908(2)	0.074(5)	0.064(3)	0.101(5)	0.011(3)	0.026(3)	–0.013(3)
N(1)	8*f*		0.6162(2)	0.6492(1)	0.8895(2)	0.046(2)	0.039(1)	0.036(1)	–0.001(1)	0.011(1)	–0.006(1)
O(1)	8*f*		0.5912(3)	0.6099(2)	1.1472(2)	0.082(2)	0.128(3)	0.027(1)	–0.022(2)	0.006(1)	0.005(2)
O(2)	8*f*		0.4396(2)	0.7141(1)	0.8346(2)	0.071(2)	0.054(2)	0.054(2)	0.003(1)	0.023(1)	–0.005(1)
C(1)	8*f*		0.4757(3)	0.5934(1)	0.9333(2)	0.048(2)	0.040(2)	0.031(2)	0.004(1)	0.012(1)	–0.001(1)
C(2)	8*f*		0.4280(3)	0.5975(1)	0.8238(2)	0.046(2)	0.035(2)	0.029(1)	0.003(1)	0.012(1)	–0.002(1)
C(3)	8*f*		0.3339(3)	0.5682(1)	0.7842(2)	0.056(2)	0.042(2)	0.033(2)	–0.002(2)	0.010(1)	–0.002(1)
C(4)	8*f*		0.2818(3)	0.5349(1)	0.8467(3)	0.057(2)	0.039(2)	0.045(2)	–0.002(2)	0.015(2)	–0.001(1)
C(5)	8*f*		0.1808(4)	0.5071(2)	0.8080(3)	0.070(3)	0.061(2)	0.056(2)	–0.018(2)	0.011(2)	–0.004(2)
C(6)	8*f*		0.1334(4)	0.4764(2)	0.8725(4)	0.078(3)	0.066(3)	0.085(3)	–0.028(2)	0.021(2)	0.001(2)
C(7)	8*f*		0.1830(4)	0.4715(2)	0.9782(4)	0.085(3)	0.056(2)	0.079(3)	–0.015(2)	0.032(2)	0.014(2)
C(8)	8*f*		0.2798(4)	0.4978(2)	1.0179(3)	0.077(3)	0.054(2)	0.055(2)	–0.003(2)	0.024(2)	0.013(2)
C(9)	8*f*		0.3314(3)	0.5306(1)	0.9543(3)	0.059(2)	0.039(2)	0.045(2)	0.002(2)	0.019(2)	0.005(1)
C(10)	8*f*		0.4288(3)	0.5600(1)	0.9950(2)	0.057(2)	0.052(2)	0.033(2)	0.003(2)	0.014(1)	0.006(1)
C(11)	8*f*		0.5777(3)	0.6251(1)	0.9682(2)	0.050(2)	0.046(2)	0.030(2)	0.004(2)	0.011(1)	–0.001(1)
C(12)	8*f*		0.6370(3)	0.6317(2)	1.0717(3)	0.060(2)	0.068(2)	0.036(2)	0.000(2)	0.006(2)	–0.005(2)
C(13)	8*f*		0.7354(4)	0.6593(2)	1.0918(3)	0.066(3)	0.078(3)	0.043(2)	–0.007(2)	–0.004(2)	–0.008(2)
C(14)	8*f*		0.7755(3)	0.6794(2)	1.0102(3)	0.053(2)	0.066(3)	0.061(2)	–0.013(2)	0.002(2)	–0.008(2)
C(15)	8*f*		0.7169(3)	0.6739(2)	0.9092(3)	0.050(2)	0.048(2)	0.048(2)	–0.003(2)	0.010(2)	–0.005(2)
C(16)	8*f*		0.7651(3)	0.6946(2)	0.8220(3)	0.059(2)	0.076(3)	0.064(3)	–0.018(2)	0.020(2)	–0.002(2)
C(17)	8*f*		0.6528(5)	0.6136(3)	1.2520(3)	0.103(4)	0.205(7)	0.033(2)	–0.021(4)	–0.001(2)	0.011(3)
C(18)	4*e*		½	0.7861(3)	¾	0.044(3)	0.083(4)	0.062(3)	0	0.016(2)	0
C(19)	8*f*		0.4570(3)	0.7630(2)	0.8261(3)	0.041(2)	0.045(2)	0.072(2)	–0.001(2)	0.005(2)	–0.000(2)

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