Crystal structure of bis(2-benzoylbenzoato-κ^2O,O')-(2,2′-bipyridine-κ^2N,N')cadmium(II), $Cd(C_{10}H_8N_2)(C_{14}H_9O_3)_2$

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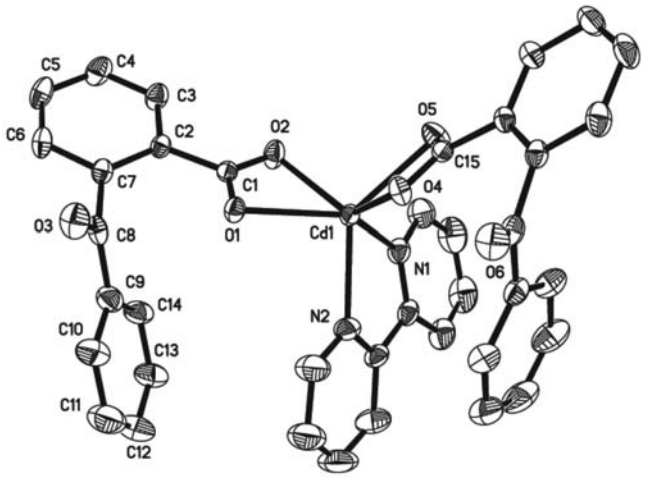

Abstract

$C_{38}H_{26}CdN_2O_6$, monoclinic, $P12_1/c1$ (no. 14), a = 13.385(3) Å, b = 14.130(3) Å, c = 17.139(3) Å, β = 90.66(3)°, V = 3241.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.030, $wR_{ref}(F^2)$ = 0.076, T = 293 K.

Source of material

A mixture of 2-benzoylbenzoic acid (2.00 mmol), 2,2′-bipyridine (0.99 mmol) and cadmium acetate (0.98 mmol) were dissolved in the mixed solvent of ethanol (20 ml) and *N*,*N*-dimethylformamide (1 ml). The pH value of the resultant mixture was adjusted to about 7.0 by adding ten drops of triethylamine solution and the reaction was kept at 348 K for 15 h. Afterwards the system was filtered, and the filtrate was cooled to room temperature slowly. Colorless block-shaped single crystals suitable for X-ray diffraction analysis were obtained after one month.

Experimental details

Hydrogen atoms bound to carbon atoms were positioned geometrically with d(C—H) = 0.93 Å and refined as riding, with U_{iso}(H) = 1.2 U_{eq}(C). In the crystal structure of the title complex, one benzene ring of 2-benzoylbenzoate (C9-C14) is disordered due to high thermal motion at the experiment temperature of 293 K.

Discussion

As an excellent electron donor, 2,2′-bipyridine could coordinate most transitional metals via N atom to produce a series of complexes possessing novel structures and properties [1-4]. 2-Benzoylbenzoic acid is a main raw material of anthraquinone dyes intermediates. It is used to manufacture anthraquinone, and 1-aminoanthraquinone. It can also coordinate metal ions to generate complexes as a good multi-dentate rigid ligand.

The assymetric unit of the crystal structure of the title complex consists of one Cd(II) ion, two 2-benzoylbenzoate and one 2,2′-bipyridine species. The cadmium(II) ion is coordinated by two nitrogen atoms from 2,2′-bipyridine and four oxygen atoms from two 2-benzoylbenzoates in a distorted triangular prismatic arrangement, in which the atoms N2, O4 and O1 construct on plane of the triangular prism and atoms N1, O5 and O2 determine the another one. The dihedral angle between the two planes is 9.4°. The O–Cd1–O angles of the title complex vary from 51.10(6)° to 129.37(8)°, and the N–Cd1–O angles vary from 90.25(8)° to 132.95(7)°. The Cd1—O bond lengths are in the

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.12 × 0.18 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.24 cm^{-1}
Diffractometer, scan mode:	Rigaku Saturn CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	25248, 5708
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4961
$N(param)_{refined}$:	456
Programs:	SHELXS-97, SHELXL-97 [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(3A)	4*e*		0.0833	0.5857	0.2564	0.055
H(4A)	4*e*		–0.0416	0.6572	0.3267	0.067
H(5A)	4*e*		–0.1921	0.5803	0.3432	0.067
H(6A)	4*e*		–0.2194	0.4356	0.2866	0.057
H(10A)	4*e*	0.50	–0.1886	0.1877	0.1320	0.084
H(11A)	4*e*	0.50	–0.2159	0.1538	0.0011	0.103
H(12A)	4*e*	0.50	–0.1770	0.2653	–0.0932	0.091
H(13A)	4*e*	0.50	–0.1108	0.4106	–0.0565	0.078
H(14A)	4*e*	0.50	–0.0835	0.4444	0.0744	0.065
H(10B)	4*e*	0.50	–0.1664	0.1824	0.1065	0.086
H(11B)	4*e*	0.50	–0.1834	0.1758	–0.0282	0.097
H(12B)	4*e*	0.50	–0.1517	0.3093	–0.1029	0.086
H(13B)	4*e*	0.50	–0.1032	0.4492	–0.0427	0.082
H(14B)	4*e*	0.50	–0.0862	0.4558	0.0921	0.068
H(17A)	4*e*		0.5462	0.3305	0.2678	0.050
H(18A)	4*e*		0.6853	0.2562	0.3192	0.063
H(19A)	4*e*		0.7089	0.0967	0.3000	0.069
H(20A)	4*e*		0.5946	0.0110	0.2268	0.058
H(24A)	4*e*		0.3091	–0.0055	0.0452	0.073
H(25A)	4*e*		0.2820	0.0347	–0.0842	0.097
H(26A)	4*e*		0.3798	0.1487	–0.1415	0.097

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Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(27A)	4*e*		0.5024	0.2228	–0.0714	0.092
H(28A)	4*e*		0.5299	0.1844	0.0580	0.068
H(29A)	4*e*		0.3736	0.5375	0.0626	0.079
H(30A)	4*e*		0.4422	0.6132	–0.0437	0.097
H(31A)	4*e*		0.4193	0.5479	–0.1656	0.098

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(32A)	4*e*		0.3195	0.4175	–0.1807	0.079
H(35A)	4*e*		0.1993	0.3172	–0.1891	0.088
H(36A)	4*e*		0.0791	0.2011	–0.1906	0.108
H(37A)	4*e*		0.0301	0.1326	–0.0751	0.102
H(38A)	4*e*		0.099	0.1876	0.0393	0.084

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}
Cd(1)	4*e*		0.24484(1)	0.34955(1)	0.10881(1)	0.0294(1)	0.0427(1)	0.0266(1)	0.00678(7)	0.00136(7)	0.00107(7)
O(1)	4*e*		0.0854(1)	0.3366(1)	0.1671(1)	0.0328(9)	0.031(1)	0.046(1)	0.0022(7)	0.0083(8)	–0.0025(8)
O(2)	4*e*		0.1665(1)	0.4709(1)	0.1698(1)	0.040(1)	0.040(1)	0.071(1)	–0.0083(9)	0.020(1)	–0.013(1)
O(3)	4*e*		–0.1281(2)	0.2621(2)	0.2438(1)	0.063(1)	0.042(1)	0.071(2)	–0.006(1)	0.012(1)	0.011(1)
O(4)	4*e*		0.3190(1)	0.2200(1)	0.1698(1)	0.0280(9)	0.038(1)	0.053(1)	0.0003(7)	–0.0010(8)	0.0049(8)
O(5)	4*e*		0.4031(2)	0.3531(1)	0.1678(1)	0.044(1)	0.034(1)	0.075(2)	–0.0002(8)	–0.019(1)	0.010(1)
O(6)	4*e*		0.3968(2)	–0.0102(1)	0.1763(1)	0.060(1)	0.042(1)	0.065(1)	–0.006(1)	0.007(1)	0.010(1)
N(1)	4*e*		0.3039(2)	0.4374(2)	0.0053(1)	0.042(1)	0.052(2)	0.043(1)	0.002(1)	0.006(1)	0.006(1)
N(2)	4*e*		0.1856(2)	0.2842(2)	–0.0050(1)	0.052(1)	0.057(2)	0.033(1)	–0.005(1)	0.001(1)	–0.004(1)
C(1)	4*e*		0.0910(2)	0.4220(2)	0.1853(1)	0.030(1)	0.033(1)	0.029(1)	0.002(1)	0.000(1)	0.001(1)
C(2)	4*e*		0.0069(2)	0.4676(2)	0.2275(2)	0.033(1)	0.032(1)	0.035(1)	0.007(1)	0.004(1)	0.002(1)
C(3)	4*e*		0.0222(2)	0.5553(2)	0.2620(2)	0.047(2)	0.036(2)	0.056(2)	0.000(1)	0.012(1)	–0.006(1)
C(4)	4*e*		–0.0522(3)	0.5980(2)	0.3045(2)	0.067(2)	0.038(2)	0.064(2)	0.008(2)	0.016(2)	–0.013(2)
C(5)	4*e*		–0.1422(2)	0.5523(2)	0.3138(2)	0.055(2)	0.050(2)	0.063(2)	0.020(2)	0.023(2)	–0.003(2)
C(6)	4*e*		–0.1583(2)	0.4657(2)	0.2799(2)	0.035(1)	0.049(2)	0.060(2)	0.009(1)	0.015(1)	0.005(1)
C(7)	4*e*		–0.0852(2)	0.4221(2)	0.2356(2)	0.034(1)	0.032(1)	0.040(1)	0.007(1)	0.004(1)	0.004(1)
C(8)	4*e*		–0.1122(2)	0.3282(2)	0.2003(2)	0.024(1)	0.041(2)	0.056(2)	0.004(1)	0.003(1)	0.004(1)
C(9)	4*e*	0.50(2)	–0.133(1)	0.3194(9)	0.1162(4)	0.043(4)	0.054(3)	0.055(3)	0.003(3)	–0.009(3)	–0.004(3)
C(10)	4*e*	0.50	–0.173(1)	0.2323(8)	0.0943(6)	0.071(4)	0.072(3)	0.067(4)	–0.014(3)	–0.009(3)	–0.016(3)
C(11)	4*e*	0.50	–0.1894(9)	0.2121(7)	0.0158(7)	0.091(5)	0.091(4)	0.076(4)	–0.028(4)	–0.014(4)	–0.013(4)
C(12)	4*e*	0.50	–0.1661(8)	0.279(1)	–0.0407(4)	0.084(4)	0.076(5)	0.066(4)	–0.014(4)	–0.028(3)	–0.005(3)
C(13)	4*e*	0.50	–0.1264(9)	0.366(1)	–0.0187(5)	0.074(4)	0.064(4)	0.055(3)	0.001(4)	–0.024(3)	–0.004(3)
C(14)	4*e*	0.50	–0.110(1)	0.3862(8)	0.0597(6)	0.055(3)	0.058(3)	0.049(3)	0.008(3)	–0.019(3)	–0.006(3)
C(9')	4*e*	0.50	–0.125(1)	0.3197(9)	0.1127(3)	0.033(3)	0.052(3)	0.053(3)	0.006(3)	–0.010(3)	–0.007(3)
C(10')	4*e*	0.50	–0.154(1)	0.2359(7)	0.0766(6)	0.072(4)	0.075(3)	0.067(4)	–0.015(3)	–0.005(3)	–0.013(3)
C(11')	4*e*	0.50	–0.1639(9)	0.2319(8)	–0.0041(6)	0.089(4)	0.083(4)	0.070(4)	–0.024(4)	–0.014(4)	–0.015(4)
C(12')	4*e*	0.50	–0.1450(8)	0.312(1)	–0.0488(3)	0.076(4)	0.078(5)	0.062(3)	–0.008(4)	–0.024(3)	–0.009(3)
C(13')	4*e*	0.50	–0.1159(9)	0.3958(9)	–0.0128(5)	0.076(4)	0.071(4)	0.057(3)	0.012(4)	–0.023(3)	–0.007(3)
C(14')	4*e*	0.50	–0.106(1)	0.3997(7)	0.0679(6)	0.057(3)	0.062(4)	0.050(3)	0.015(3)	–0.015(3)	–0.005(3)
C(15)	4*e*		0.3974(2)	0.2667(2)	0.1819(1)	0.031(1)	0.033(1)	0.029(1)	0.003(1)	0.002(1)	–0.001(1)
C(16)	4*e*		0.4875(2)	0.2162(2)	0.2137(2)	0.028(1)	0.038(1)	0.034(1)	0.004(1)	0.002(1)	0.007(1)
C(17)	4*e*		0.5562(2)	0.2664(2)	0.2587(2)	0.036(1)	0.044(2)	0.045(2)	0.001(1)	–0.003(1)	0.004(1)
C(18)	4*e*		0.6390(2)	0.2217(2)	0.2900(2)	0.036(2)	0.067(2)	0.055(2)	0.000(1)	–0.011(1)	0.004(2)
C(19)	4*e*		0.6534(2)	0.1267(3)	0.2783(2)	0.037(2)	0.075(2)	0.060(2)	0.015(2)	–0.008(1)	0.016(2)
C(20)	4*e*		0.5851(2)	0.0755(2)	0.2341(2)	0.043(2)	0.045(2)	0.057(2)	0.015(1)	0.004(1)	0.010(1)
C(21)	4*e*		0.5023(2)	0.1200(2)	0.2006(2)	0.033(1)	0.038(1)	0.037(1)	0.005(1)	0.007(1)	0.007(1)
C(22)	4*e*		0.4350(2)	0.0608(2)	0.1495(2)	0.039(1)	0.032(1)	0.045(2)	0.008(1)	0.008(1)	0.003(1)
C(23)	4*e*		0.4211(2)	0.0864(2)	0.0662(2)	0.046(2)	0.040(2)	0.044(2)	0.010(1)	0.007(1)	–0.005(1)
C(24)	4*e*		0.3480(3)	0.0412(3)	0.0224(2)	0.063(2)	0.065(2)	0.055(2)	0.003(2)	0.003(2)	–0.014(2)
C(25)	4*e*		0.3320(3)	0.0648(3)	–0.0553(2)	0.079(3)	0.107(3)	0.056(2)	0.018(2)	–0.010(2)	–0.030(2)
C(26)	4*e*		0.3904(4)	0.1328(3)	–0.0894(2)	0.115(4)	0.089(3)	0.039(2)	0.032(3)	0.006(2)	–0.005(2)
C(27)	4*e*		0.4631(4)	0.1769(3)	–0.0478(2)	0.115(3)	0.070(2)	0.045(2)	0.007(2)	0.023(2)	0.004(2)
C(28)	4*e*		0.4793(3)	0.1541(2)	0.0299(2)	0.074(2)	0.055(2)	0.042(2)	0.001(2)	0.015(2)	–0.002(1)
C(29)	4*e*		0.3615(3)	0.5135(3)	0.0129(2)	0.059(2)	0.068(2)	0.071(2)	–0.014(2)	0.009(2)	0.004(2)
C(30)	4*e*		0.4043(3)	0.5586(3)	–0.0504(3)	0.070(2)	0.072(3)	0.102(3)	–0.017(2)	0.016(2)	0.020(2)
C(31)	4*e*		0.3892(3)	0.5207(3)	–0.1224(3)	0.074(3)	0.097(3)	0.075(3)	–0.002(2)	0.022(2)	0.039(2)
C(32)	4*e*		0.3303(3)	0.4433(3)	–0.1315(2)	0.064(2)	0.090(3)	0.045(2)	0.005(2)	0.014(2)	0.020(2)
C(33)	4*e*		0.2864(2)	0.4029(2)	–0.0664(2)	0.042(2)	0.059(2)	0.034(2)	0.013(1)	0.007(1)	0.009(1)
C(34)	4*e*		0.2152(2)	0.3229(2)	–0.0721(2)	0.049(2)	0.059(2)	0.028(1)	0.012(1)	0.001(1)	–0.002(1)
C(35)	4*e*		0.1771(3)	0.2912(3)	–0.1425(2)	0.098(3)	0.089(3)	0.033(2)	–0.002(2)	–0.005(2)	–0.004(2)
C(36)	4*e*		0.1067(4)	0.2213(3)	–0.1434(3)	0.110(3)	0.102(3)	0.058(2)	–0.010(3)	–0.021(2)	–0.030(2)
C(37)	4*e*		0.0770(3)	0.1811(3)	–0.0755(3)	0.092(3)	0.092(3)	0.071(3)	–0.028(2)	–0.007(2)	–0.021(2)
C(38)	4*e*		0.1186(3)	0.2147(3)	–0.0075(2)	0.079(3)	0.080(3)	0.052(2)	–0.027(2)	0.002(2)	–0.008(2)

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