Crystal structure of bis(2,2′-biimidazole)zinc(II) benzene-1,2-dicarboxylate monohydrate, $Zn(C_6H_6N_4)_2(C_8H_4O_4)\cdot H_2O$

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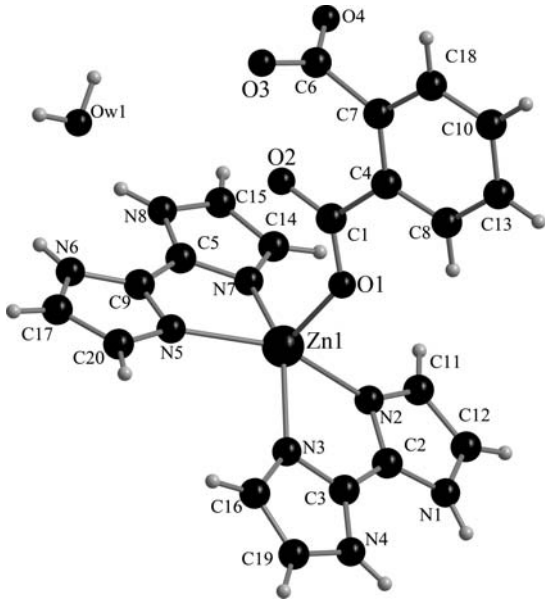

Abstract

$C_{20}H_{18}N_8O_5Zn$, monoclinic, $P12_1/c1$ (no. 14), a = 10.0039(8) Å, b = 11.6195(9) Å, c = 18.197(1) Å, β = 96.404(1)°, V = 2102.0 Å^3, Z = 4, $R_{gf}(F)$ = 0.045, $wR_{ref}(F^2)$ = 0.156, T = 293 K.

Source of material

The title complex was prepared from a mixture of $Zn(OAc)_2\cdot 2H_2O$ (0.184 g), benzene-l,2-dicarboxylic acid (0.166 g), 2,2′-biimidazole (0.270 g) and H_2O (19 ml) in a 30 ml Teflon-lined autoclave under autogenous pressure at 443K for four days. After cooling to room temperature, colorless block crystals were collected by filtration and washed with distilled water (34 % yield based on Zn). Chemical analysis — found:C, 46.9 %; H, 3.6 %; N, 22.4 %; calculated for $C_{20}H_{18}N_8O_5Zn$: C, 46.6 %; H, 3.5 %; N, 21.7 %. IR data are available in the CIF.

Discussion

In the title crystal structure there are one Zn(II) ion, one benzene-l,2-dicarboxylate ligand (DBC), two 2,2′-biimidazole ligands and one free water molecule in the asymmetric unit. The coordination number of zinc(II) atom is five, and it is coordinated by one oxygen atoms from a carboxyl of DBC with d(Zn1—O1) = 1.973(3) Å. This distance is shorter than the normal average Zn—O distance [1], the other oxygen atoms are deprotonated to maintain the charge banlance. The zinc(II) atom is further coordinated by four nitrogen atoms (d(Zn1—N2) = 2.142(3) Å, d(Zn1—N3) = 2.168(3) Å, d(Zn1—N5) = 2.136(4) Å, d(Zn1—N7) = 2.145(3) Å) from two different biimidazole ligands, forming a tetragonal pyramide with the O(N)–Zn–O(N) angles ranging from 77.8(1)° to 152.9(1)°. The two carboxyl groups of one DBC are both deprotonated, as proven by the absence of any strong bands around 1700 cm^{-1} in the IR spectrum [2]. Each dianion is coordinated to a Zn(II) ion in a monodentate coordination mode. In this coordination manner, the title complex forms a zero dimentional structure. In the crystal lattice, the N6···N8^{i} distance of 3.334 Å between the imidazole rings (containing N6, N8) and its symmetry equivalent (symmetry code i: 1–x,1–y,–z), and the N6···N1^{ii} distance of 3.515 Å between the imidazole ring (contains N6)-imidazole ring (contains N1^{ii}) (symmetry code ii: 1–x,–y,–z) in an offset fashion, indicates strong π-π stacking interactions between two neighboring aryl-rings. Otherwise, there are seven types of hydrogen bonds between N atoms from biimidazole and O atoms from both BDC and water molecules. The π-π interactions and hydrogen bonds result in a three-dimensional supramolecular structure. It is clear that the π-π interactions and the N–H···O, Ow–H···O hydrogen bonds stabilize the title structure [3].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.288 × 0.321 × 0.403 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.22 cm^{-1}
Diffractometer, scan mode:	Bruker Apex II CCD, ω
$2\theta_{max}$:	52.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17615, 4166
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3040
$N(param)_{refined}$:	307
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6], DIAMOND [7]

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

Atom	Site	x	y	z	U_{iso}
H(8A)	4*e*	0.7141	0.4548	–0.0044	0.066
H(6A)	4*e*	0.5287	0.4441	–0.1260	0.068
H(1A)	4*e*	0.3571	–0.1997	0.1675	0.064
H(4A)	4*e*	0.1688	–0.2018	0.0468	0.071
H(8B)	4*e*	0.0943	0.2460	0.2047	0.081
H(10A)	4*e*	–0.1058	0.4870	0.3021	0.086
H(11A)	4*e*	0.5890	0.0656	0.2136	0.085
H(12A)	4*e*	0.5373	–0.1279	0.2558	0.085
H(13A)	4*e*	–0.0626	0.2960	0.2819	0.091
H(14A)	4*e*	0.6927	0.2533	0.1640	0.084

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(15A)	4*e*	0.8303	0.4043	0.1151	0.083
H(16A)	4*e*	0.1315	0.0538	–0.0909	0.119
H(17A)	4*e*	0.3241	0.3901	–0.2013	0.098
H(18A)	4*e*	0.0063	0.6243	0.2429	0.104

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(19A)	4*e*	0.0499	–0.1389	–0.0694	0.107
H(20A)	4*e*	0.2108	0.2522	–0.1296	0.102
HW(1A)	4*e*	0.9161	0.5803	–0.0313	0.383
HW(1B)	4*e*	0.9625	0.5989	0.0399	0.383

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}
Zn(1)	4*e*	0.37547(4)	0.18045(3)	0.05438(2)	0.0561(3)	0.0254(2)	0.0580(3)	–0.0039(2)	0.0225(2)	0.0019(2)
N(2)	4*e*	0.4379(3)	0.0424(2)	0.1284(2)	0.061(2)	0.031(2)	0.067(2)	–0.009(1)	0.013(2)	0.001(1)
N(8)	4*e*	0.6876(3)	0.4035(3)	0.0248(2)	0.058(2)	0.045(2)	0.067(2)	–0.011(2)	0.022(2)	0.006(2)
N(7)	4*e*	0.5650(3)	0.2687(3)	0.0685(2)	0.055(2)	0.045(2)	0.070(2)	0.001(2)	0.020(2)	0.015(2)
N(5)	4*e*	0.3706(4)	0.2694(3)	–0.0486(2)	0.069(2)	0.053(2)	0.066(2)	–0.020(2)	0.020(2)	–0.002(2)
N(6)	4*e*	0.4703(3)	0.3957(3)	–0.1135(2)	0.057(2)	0.050(2)	0.066(2)	–0.006(2)	0.023(2)	0.008(2)
N(1)	4*e*	0.3939(3)	–0.1330(3)	0.1658(2)	0.064(2)	0.031(2)	0.065(2)	–0.004(2)	0.010(2)	0.007(2)
N(4)	4*e*	0.1798(4)	–0.1365(3)	0.0260(2)	0.082(2)	0.032(2)	0.062(2)	–0.010(2)	0.005(2)	–0.001(2)
N(3)	4*e*	0.2545(4)	0.0383(3)	0.0072(2)	0.097(3)	0.032(2)	0.062(2)	–0.007(2)	0.005(2)	0.006(2)
O(1)	4*e*	0.2519(3)	0.2603(2)	0.1147(2)	0.088(2)	0.029(1)	0.108(2)	–0.002(1)	0.055(2)	–0.002(1)
O(2)	4*e*	0.3188(3)	0.4395(2)	0.0991(2)	0.061(2)	0.038(1)	0.070(2)	–0.005(1)	0.029(1)	0.001(1)
O(4)	4*e*	0.2784(3)	0.6668(2)	0.1897(2)	0.066(2)	0.037(2)	0.089(2)	–0.009(1)	0.017(2)	0.011(1)
O(3)	4*e*	0.1261(4)	0.6587(2)	0.0925(2)	0.095(2)	0.048(2)	0.067(2)	–0.015(2)	0.018(2)	0.008(1)
C(1)	4*e*	0.2469(4)	0.3670(3)	0.1251(2)	0.050(2)	0.030(2)	0.053(2)	–0.001(2)	0.013(2)	0.005(2)
C(2)	4*e*	0.3625(4)	–0.0513(3)	0.1150(2)	0.056(2)	0.029(2)	0.051(2)	–0.002(2)	0.020(2)	–0.002(2)
C(3)	4*e*	0.2650(4)	–0.0530(3)	0.0514(2)	0.067(2)	0.029(2)	0.055(2)	–0.004(2)	0.021(2)	–0.002(2)
C(4)	4*e*	0.1441(3)	0.4048(3)	0.1756(2)	0.047(2)	0.032(2)	0.049(2)	0.001(2)	0.012(2)	0.006(2)
C(5)	4*e*	0.5768(4)	0.3387(3)	0.0121(2)	0.051(2)	0.034(2)	0.064(2)	–0.002(2)	0.025(2)	0.002(2)
C(6)	4*e*	0.1819(4)	0.6209(3)	0.1526(2)	0.066(3)	0.029(2)	0.067(2)	0.002(2)	0.032(2)	–0.000(2)
C(7)	4*e*	0.1177(4)	0.5192(3)	0.1874(2)	0.055(2)	0.034(2)	0.066(2)	–0.004(2)	0.026(2)	–0.001(2)
C(8)	4*e*	0.0763(5)	0.3235(3)	0.2119(3)	0.087(3)	0.034(2)	0.090(3)	0.000(2)	0.044(3)	0.009(2)
C(9)	4*e*	0.4760(4)	0.3375(3)	–0.0503(2)	0.054(2)	0.032(2)	0.059(2)	–0.001(2)	0.024(2)	0.003(2)
C(10)	4*e*	–0.0433(5)	0.4656(4)	0.2704(3)	0.083(3)	0.053(3)	0.089(3)	0.003(2)	0.059(3)	0.005(2)
C(11)	4*e*	0.5230(5)	0.0170(4)	0.1910(3)	0.078(3)	0.051(3)	0.080(3)	–0.014(2)	–0.002(2)	–0.001(2)
C(12)	4*e*	0.4951(5)	–0.0905(4)	0.2144(3)	0.082(3)	0.056(3)	0.071(3)	–0.009(2)	–0.008(2)	0.008(2)
C(13)	4*e*	–0.0174(5)	0.3529(4)	0.2586(3)	0.092(3)	0.046(2)	0.100(3)	–0.005(2)	0.058(3)	0.016(2)
C(14)	4*e*	0.6738(5)	0.2901(4)	0.1186(3)	0.066(3)	0.071(3)	0.074(3)	0.004(2)	0.012(2)	0.022(2)
C(15)	4*e*	0.7506(5)	0.3738(4)	0.0919(3)	0.064(3)	0.070(3)	0.076(3)	–0.011(2)	0.010(2)	0.007(2)
C(16)	4*e*	0.1597(7)	0.0077(5)	–0.0503(3)	0.153(5)	0.054(3)	0.079(3)	–0.013(3)	–0.041(4)	0.014(2)
C(17)	4*e*	0.3549(5)	0.3636(5)	–0.1542(3)	0.086(4)	0.090(4)	0.068(3)	–0.026(3)	0.005(3)	0.016(3)
C(18)	4*e*	0.0241(6)	0.5470(4)	0.2349(3)	0.123(4)	0.032(2)	0.122(4)	0.001(2)	0.084(4)	–0.003(2)
C(19)	4*e*	0.1143(6)	–0.0987(4)	–0.0387(3)	0.126(5)	0.051(3)	0.083(3)	–0.017(3)	–0.027(3)	0.005(3)
C(20)	4*e*	0.2929(6)	0.2874(5)	–0.1148(3)	0.087(4)	0.095(4)	0.072(3)	–0.041(3)	0.005(3)	0.007(3)
OW(1)	4*e*	0.938(1)	0.5435(9)	0.0066(8)	0.35(2)	0.26(2)	0.33(1)	–0.02(1)	–0.11(1)	0.01(1)

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