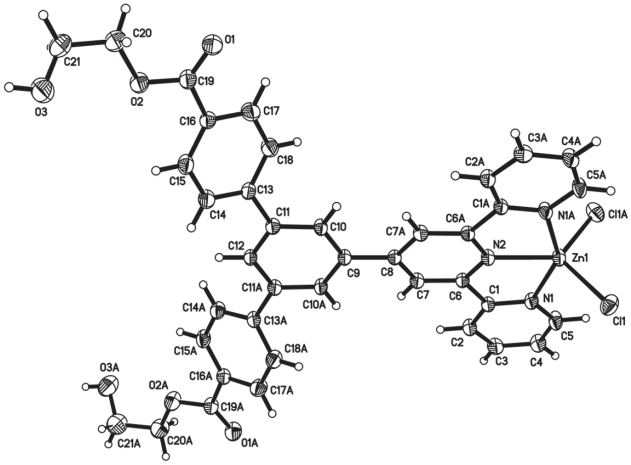
Open Access

Yaping Li, Dajun Sun*, Julia Ming and Guanfang Su

Crystal structure of dichlorido{bis(2-hydroxyethyl)-5′-([2,2′:6′,2″-terpyridin]-4′-yl)-[1,1′:3′,1″-terphenyl]-4,4″-dicarboxylate-κ^3*N*,*N*′,*N*″}zinc(II), $C_{39}H_{31}C_{l2}N_3O_6Zn$



DOI 10.1515/ncrs-2016-0041
Received March 9, 2016; accepted June 15, 2016; available online July 6, 2016

Abstract

$C_{39}H_{31}C_{l2}N_3O_6Zn$, monoclinic, *C*2/*c* (no. 15), $a = 20.7170(16)$ Å, $b = 14.4553(11)$ Å, $c = 11.2717(8)$ Å, $\beta = 90.072(2)°$, $V = 3375.5(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0489$, $wR_{ref}(F^2) = 0.1469$, $T = 293(2)$ K.

CCDC no.: 1485800

Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block Size: 0.23 × 0.21 × 0.17 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.4 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
$2\theta_{max}$, completeness,	50.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9689, 3004, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2259
$N(param)_{refined}$:	236
Programs:	SHELX [11], Bruker programs [12]

Source of material

A mixture of 5′-([2,2′:6′,2″-terpyridin]-4′-yl)-[1,1′:3′,1″-terphenyl]-4,4″-dicarboxylic acid (0.1 mmol, 0.053 g), $ZnCl_2$, (0.2 mmol, 0.014 g), ethylene glycol (3 mL) and H_2O (15 mL) was stirred for twenty minutes. The mixtures were transfered in a 30 mL stainless steel reactor with a Teflon liner and heated from 293 to 443 K in 5 h and a constant temperature was maintained at 443 K for 72 h. After cooling to room temperature, blue crystals were collected in 56.9% yield based on $ZnCl_2$.

Experimental details

The C-bound H atoms were positioned with idealized geometry and were refined with $U_{iso}(H) = 1.2U_{eq}(C)$ using a riding model with C—H = 0.93 Å for aromatic H atoms and C—H = 0.97 Å for methylene H atoms. The O-bound H atom was refined freely with the O—H bond length restrained to 0.85(2) Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

Discussion

Extensive efforts on metal-organic frameworks (MOFs) have not only led to the creation of a huge number of diverse topologies, but also initiated a strategy to construct porous materials with high surface areas, predictable structure, and tunable pore sizes to target some important applications [1–4]. Aromatic carboxylic acids have been widely used in the field

***Corresponding author: Dajun Sun,** Department of Vascular Surgery, The China-Japan Union Hospital of Jilin University, Changchun 130033, P. R. China, e-mail: drsundj@163.com
Yaping Li and Guanfang Su: Department of Ophthalmology, The Second Hospital of Jilin University, Changchun 130041, P. R. China
Julia Ming: St Erik's Eye Hospital, Karolinska Institutet, Polhemsgatan 50, SE-112 82, Stockholm, Sweden

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Zn1	1.0000	0.15335(4)	0.7500	0.0367(2)
Cl1	1.06638(6)	0.24235(7)	0.86198(10)	0.0546(3)
C1	1.06986(17)	0.0198(2)	0.5840(3)	0.0321(8)
C2	1.10626(19)	−0.0134(3)	0.4915(3)	0.0393(9)
H2	1.1100	−0.0767	0.4785	0.047*
C3	1.13727(19)	0.0488(3)	0.4179(4)	0.0438(10)
H3	1.1616	0.0277	0.3542	0.053*
C4	1.1319(2)	0.1413(3)	0.4394(4)	0.0474(11)
H4	1.1527	0.1842	0.3912	0.057*
C5	1.0950(2)	0.1698(3)	0.5343(4)	0.0483(11)
H5	1.0911	0.2328	0.5490	0.058*
C6	1.03358(17)	−0.0403(2)	0.6684(3)	0.0307(8)
C7	1.03407(17)	−0.1357(2)	0.6650(3)	0.0341(9)
H7	1.0571	−0.1665	0.6062	0.041*
C8	1.0000	−0.1856(3)	0.7500	0.0308(11)
C9	1.0000	−0.2886(3)	0.7500	0.0323(11)
C10	0.95741(18)	−0.3377(2)	0.8205(3)	0.0347(9)
H10	0.9290	−0.3055	0.8692	0.042*
C11	0.95610(18)	−0.4344(2)	0.8203(3)	0.0342(8)
C12	1.0000	−0.4827(3)	0.7500	0.0344(12)
H12	1.0000	−0.5470	0.7500	0.041*
C13	0.90794(18)	−0.4810(2)	0.8988(3)	0.0352(9)
C14	0.9204(2)	−0.5609(3)	0.9597(4)	0.0469(10)
H14	0.9592	−0.5917	0.9478	0.056*
C15	0.8755(2)	−0.5966(3)	1.0392(4)	0.0474(10)
H15	0.8851	−0.6504	1.0810	0.057*
C16	0.81705(18)	−0.5530(3)	1.0569(3)	0.0378(9)
C17	0.8028(2)	−0.4734(3)	0.9917(4)	0.0539(11)
H17	0.7630	−0.4443	1.0003	0.065*
C18	0.8484(2)	−0.4383(3)	0.9145(4)	0.0515(11)
H18	0.8390	−0.3848	0.8720	0.062*
C19	0.7677(2)	−0.5856(3)	1.1428(4)	0.0427(10)
C20	0.7259(2)	−0.7098(3)	1.2567(5)	0.0694(15)
H20A	0.7328	−0.6822	1.3341	0.083*
H20B	0.6827	−0.6939	1.2302	0.083*
C21	0.7324(3)	−0.8090(4)	1.2645(6)	0.090(2)
H21A	0.6974	−0.8338	1.3118	0.108*
H21B	0.7293	−0.8355	1.1857	0.108*
N1	1.06463(15)	0.1105(2)	0.6061(3)	0.0377(8)
N2	1.0000	0.0070(3)	0.7500	0.0317(10)
O1	0.72553(15)	−0.5374(2)	1.1826(3)	0.0576(8)
O2	0.77386(15)	−0.6739(2)	1.1720(3)	0.0603(9)
O3	0.7916(2)	−0.8347(3)	1.3158(4)	0.0837(12)
H3A	0.789(3)	−0.890(2)	1.342(6)	0.126*

of coordination chemistry. Some of the reasons are that aromatic carboxylic acids, and the corresponding carboxylates, have good coordination ability for transition metals, and for their capability as bridging and chelating ligands in various coordination modes [5–10].

The asymmetric unit and the symmetry related second half of the title complex is shown in the figure. In the title compound, the Zn(II) ion lies on a twofold rotation axis and displays a slightly trigonal bipyramid geometry defined by three N atoms from pyridine rings and two chlorido ligands. The bond lengths and angles are in a normal range. There are O—H···O hydrogen bonds in the crystal, which furthermore stabilize crystal.

Acknowledgements: The project were supported by the Pharmaceutical industry Promotion Project of Jilin Province (20140311015YY).

References

1. Garudachari, B.; Islor, A. M.; Satyanarayan, M. N.; Gerber, T.; Hosten, E.; Betz, R.: Crystal structure of ethyl 6-methyl-4-oxo-1,4-dihydroquinoline-3-carboxylate, $C_{13}H_{13}NO_3$. Z. Kristallogr. NCS **228** (2013) 301–302.
2. Song, X. Z.; Song, S. Y.; Zhao, S. N.; Hao, Z. M.; Zhu, M.; Meng, X.; Wu, L. L.; Zhang, H. J.: Single-crystal-to-single-crystal transformation of a europium(III) metal-organic framework producing a multi-responsive luminescent sensor. Adv. Funct. Mater. **24** (2014) 4034–4041.
3. Majumder, A.; Gramlich, V.; Rosair, G. M.: Five new cobalt(II) and copper(II)-1,2,4,5-benzenetetracarboxylate supramolecular architectures: syntheses, structures and magnetic properties. Cryst. Growth Des. **2** (2005) 415–420.
4. Jin, Y. B.; Yu, H.; Li, J.; Lei, K. W.: Crystal structure of poly[(μ-2,2′-bipyridine-3,3′,5,5′-tetracarboxylato)hexaaquadicobalt(II)], $C_{14}H_{16}Co_2N_2O_{14}$. Z. Kristallogr. NCS **229** (2014) 79–80.
5. He, J. Y.; Yao, Q. G.; Liu, J. J.; Jia, H. Z.; Yu, H. W.: Crystal structure of dimethyl 4-(4-chlorophenyl)-1,4-dihydropyridine-3,5-dicarboxylate, $C_{15}H_{14}ClNO_4$. Z. Kristallogr. NCS **229** (2014) 123–124.
6. Meng, X.; Song, S. Y.; Song, X. Z.; Zhu, M.; Zhao, S. N.; Wu, L. L.; Zhang, H. J.: A Eu/Tb-codoped coordination polymer luminescent thermometer. Inorg. Chem. Front. **1** (2014) 757–760.
7. Zhu, M.; Song, X. Z.; Song, S. Y.; Meng, X.; Zhao, S. N.; Wu, L. L.; Wang, C.; Zhang, H. J.: A temperature-responsive smart europium metal-organic framework switch for reversiblecapture and release of intrinsic Eu^{3+} ions. Adv. Sci. **2** (2015) 1500012.
8. Barnett, S. A.; Champness, N. R.: Structural diversity of building-blocks in coordination framework synthesis combining $M(NO_3)_2$ junctions and bipyridyl ligands. Coord. Chem. Rev. **246** (2003) 145–168.
9. Zhao, S. N.; Li, L. J.; Song, X. Z.; Zhu, M.; Hao, Z. M.; Meng, X.; Wu, L. L.; Feng, J.; Song, S. Y.; Wang, C.; Zhang, H. J.: Lanthanide ion codoped emitters for tailoring emission trajectory and temperature sensing. Adv. Funct. Mater. **25** (2015) 1463–1469.
10. Meng, X.; Zhong, R. L.; Song, X. Z.; Song, S. Y.; Hao, Z. M.; Zhu, M.; Zhao, S. N.; Zhang, H. J.: A stable, pillar-layer metal-organic framework containing uncoordinated carboxyl groups for separation of transition metal ions. Chem. Commun. **50** (2014) 6406–6408.
11. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
12. Bruker. APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, Wisconsin, USA, 2009.