Crystal structure of *catena*-aqua(bis(1,3,5-tris(1-imidazolyl)-benzene))zinc(II) perchlorate, $[Zn(C_{15}H_{12}N_6)_2(H_2O)](ClO_4)_2$

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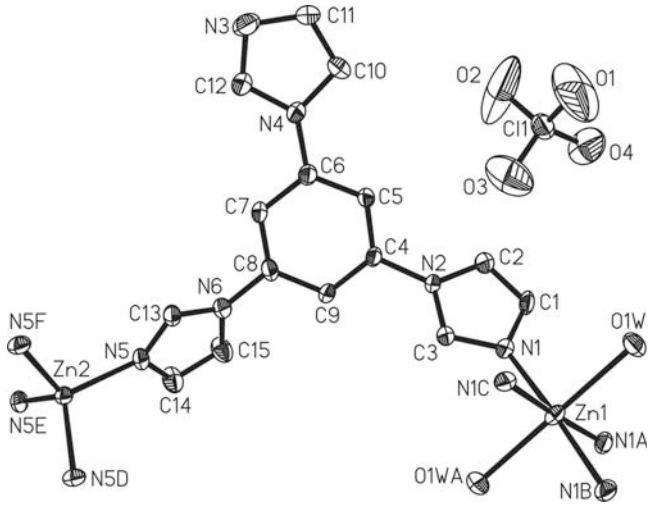

Abstract

$C_{30}H_{26}Cl_2N_{12}O_9Zn$, tetragonal, $I\bar{4}c2$ (no. 120), a = 21.2333(9) Å, c = 15.165(1) Å, V = 6837.1 Å^3, Z = 8, $R_{gt}(F)$ = 0.061, $wR_{ref}(F^2)$ = 0.151, T = 293 K.

Source of material

0.1 mmol isonicotinic acid and 0.1 mmol 1,3,5-tris(1-imidazolyl)benzene were dissolved in 10 ml distilled water. The pH value was adjusted to about 6 with 1 M NaOH solution. Then 0.1 mmol $Zn(ClO_4)_2 \cdot 6H_2O$ dissolved in 5 ml distilled water was added. The mixture was placed in a Teflon-lined stainless vessel (25 ml). The vessel was sealed and heated at 140 °C for 72 h under autogenous pressure and then cooled down to room temperature. The resulting solution was filtered and yellow needle-like single crystals suitable for analysis were obtained.

Experimental details

Hydrogen atoms bonded to carbon atoms were placed geometrically and treated as riding, with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). The water H atom was found from difference Fourier map and refined freely.

Discussion

The rational design and construction of coordination polymers are of great interest because of their intriguing structural topologies in recent years [1-3]. In order to understand the role of N-donor ligands as a second ligand, we tried to synthesize a complex with isonicotinate and 1,3,5-tris(1-imidazolyl)benzene. Unfortunately, only a two-demensional (2D) network structure without the isonicotinate was obtained.

In the title crystal structure, the Zn1 atom is coordinated by two oxygen atoms (O1WA, O1W4A) from two water molecules, and four nitrogen atoms from four 1,3,5-tris(1-imidazolyl)benzene ligands, forming a distorted octahedron. On the othe hand, the Zn2 atom is only coordinated by four nitrogen atoms forming a tetrahedron. More interestingly, the 1,3,5-tris(1-imidazolyl)benzene ligands only use two imidazole N atoms to coordinate Zn(II) atoms, while the third imidazole N atom does not participate the coordination of zinc. Therefore, it links the adjacent Zn atoms into a 2D network. It is worthwhile to note that hydrogen bonding interactions are usually important in the synthesis of supramolecular architecture. There are strong hydrogen bonding interactions between constituting molecules and ClO_4^- ion, which link the complexes into a three-dimensional framework.

Table 1. Data collection and handling.

Crystal:	yellow needle, size 0.16 × 0.20 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.48 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17919, 3377
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2899
$N(param)_{refined}$:	249
Programs:	SHELXS-97 [6], SHELXL-97 [7]

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

Atom	Site	x	y	z	U_{iso}
H(1)	16*i*	–0.0108	0.9122	0.0677	0.046
H(2)	16*i*	0.0728	0.8446	0.0141	0.050
H(3)	16*i*	0.1404	0.9605	0.1936	0.047
H(5)	16*i*	0.1647	0.7756	0.1091	0.040
H(7)	16*i*	0.3521	0.8041	0.1188	0.042
H(9)	16*i*	0.2349	0.9507	0.0880	0.039
H(10)	16*i*	0.1828	0.6714	0.1269	0.082
H(11)	16*i*	0.2382	0.5733	0.1448	0.078
H(12)	16*i*	0.3648	0.7041	0.1371	0.053
H(13)	16*i*	0.4026	0.8927	0.2132	0.044
H(14)	16*i*	0.4267	1.0473	0.0806	0.060
H(15)	16*i*	0.3357	0.9942	0.0173	0.061
H(1WA)	16*i*	–0.094(3)	0.917(3)	0.287(4)	0.05(2)

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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}
Zn(1)	4*a*	0	0	¼	0.0295(4)	U_{11}	0.0484(6)	0.0099(4)	0	0
Zn(2)	4*c*	½	0	¼	0.0250(3)	U_{11}	0.0496(6)	0	0	0
C(1)	16*i*	0.0306(2)	0.9129(2)	0.0880(3)	0.019(2)	0.048(3)	0.049(3)	0.005(2)	–0.006(2)	0.001(2)
C(2)	16*i*	0.0763(2)	0.8757(2)	0.0570(4)	0.033(3)	0.041(3)	0.053(3)	0.001(2)	–0.009(2)	–0.008(3)
C(3)	16*i*	0.1125(2)	0.9395(2)	0.1567(4)	0.026(2)	0.035(3)	0.056(3)	0.001(2)	–0.005(2)	–0.012(3)
C(4)	16*i*	0.1914(2)	0.8662(2)	0.0996(3)	0.022(2)	0.037(3)	0.039(3)	0.005(2)	–0.005(2)	–0.009(2)
C(5)	16*i*	0.1993(2)	0.8024(3)	0.1082(3)	0.022(2)	0.033(3)	0.045(2)	–0.002(2)	0.004(2)	–0.009(2)
C(6)	16*i*	0.2600(2)	0.7786(2)	0.1156(4)	0.029(2)	0.038(3)	0.039(3)	0.003(2)	–0.004(2)	–0.008(2)
C(7)	16*i*	0.3114(2)	0.8197(2)	0.1140(3)	0.021(2)	0.039(3)	0.044(3)	0.002(2)	–0.005(2)	–0.003(2)
C(8)	16*i*	0.3017(2)	0.8827(3)	0.1053(3)	0.024(2)	0.046(3)	0.036(3)	–0.005(2)	0.000(2)	–0.008(2)
C(9)	16*i*	0.2413(2)	0.9078(3)	0.0961(3)	0.023(2)	0.033(3)	0.042(3)	0.004(2)	0.003(2)	–0.001(2)
C(10)	16*i*	0.2262(3)	0.6663(3)	0.1305(6)	0.045(3)	0.036(3)	0.124(7)	–0.012(3)	–0.016(4)	0.002(4)
C(11)	16*i*	0.2572(3)	0.6127(3)	0.1414(6)	0.048(4)	0.029(3)	0.118(7)	0.002(2)	–0.008(4)	–0.006(4)
C(12)	16*i*	0.3261(3)	0.6836(3)	0.1367(3)	0.035(3)	0.044(3)	0.054(3)	0.006(2)	–0.007(2)	–0.003(3)
C(13)	16*i*	0.3975(2)	0.9247(2)	0.1721(4)	0.027(2)	0.036(3)	0.047(3)	–0.001(2)	–0.005(2)	0.004(2)
C(14)	16*i*	0.4100(3)	1.0091(3)	0.0995(4)	0.044(3)	0.048(3)	0.058(3)	–0.013(3)	–0.011(3)	0.024(3)
C(15)	16*i*	0.3603(3)	0.9800(3)	0.0640(4)	0.047(3)	0.049(3)	0.056(3)	–0.015(3)	–0.011(3)	0.017(3)
Cl(1)	16*i*	0.01914(7)	0.72108(7)	0.1573(1)	0.0429(7)	0.0513(8)	0.0571(8)	–0.0090(7)	–0.0056(7)	–0.0014(8)
N(1)	16*i*	0.0516(2)	0.9516(2)	0.1522(3)	0.026(2)	0.034(2)	0.052(2)	0.004(2)	–0.004(2)	–0.007(2)
N(2)	16*i*	0.1289(2)	0.8932(2)	0.1016(3)	0.020(2)	0.029(2)	0.049(2)	0.001(2)	0.002(2)	–0.002(2)
N(3)	16*i*	0.3197(2)	0.6226(2)	0.1470(3)	0.063(3)	0.041(3)	0.065(3)	0.015(2)	–0.011(3)	–0.002(2)
N(4)	16*i*	0.2710(2)	0.7130(2)	0.1255(3)	0.029(2)	0.038(3)	0.050(2)	0.006(2)	–0.001(2)	–0.008(2)
N(5)	16*i*	0.4326(2)	0.9739(2)	0.1678(3)	0.023(2)	0.040(2)	0.050(3)	–0.004(2)	–0.003(2)	0.000(2)
N(6)	16*i*	0.3529(2)	0.9254(2)	0.1100(3)	0.029(2)	0.036(2)	0.049(2)	–0.003(2)	0.001(2)	0.000(2)
O(1)	16*i*	–0.0107(4)	0.6830(5)	0.2186(8)	0.163(8)	0.22(1)	0.30(1)	–0.090(7)	–0.040(8)	0.17(1)
O(2)	16*i*	0.0620(4)	0.6880(7)	0.1128(8)	0.122(6)	0.42(2)	0.23(1)	0.142(9)	–0.080(7)	–0.21(1)
O(3)	16*i*	0.0504(4)	0.7663(3)	0.2067(5)	0.177(8)	0.128(5)	0.092(4)	–0.047(5)	–0.050(5)	–0.018(4)
O(4)	16*i*	–0.0260(4)	0.7448(3)	0.1021(5)	0.106(5)	0.119(6)	0.108(5)	0.029(4)	–0.054(5)	–0.029(4)
O(1W)	8*e*	–0.0744(2)	*x*	¼	0.039(2)	U_{11}	0.063(4)	–0.012(2)	–0.004(2)	$-U_{13}$

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References

1. Fujita, M.; Kwon, Y. J.; Sasaki, O.; Yamaguchi, K.; Ogura, K.: Interpenetrating Molecular Ladders and Bricks. J. Am. Chem. Soc. **117** (1995) 7287-7288.
2. Yaghi, O. M.; Li, H.; Davis, C.; Richardson, D.; Groy, T. L.: Synthetic strategies, structure patterns, and emerging properties in the chemistry of modular porous solids. Acc. Chem. Res. **31** (1998) 474-484.
3. Moulton, B.; Zaworotko, M. J.: From molecules to crystal engineering: supramolecular isomerism and polymorphism in network solids. Chem. Rev. **101** (2001) 1629-1658.
4. Go, Y. B.; Wang, X. Q.; Anokhina, E. V.; Jacobson. A. J.: A chain of changes: influence of noncovalent interactions on the one-dimensional structures of nickel(II) dicarboxylate coordination polymers with chelating aromatic amine ligands. Inorg. Chem. **43** (2004) 5360-5367.
5. Zhang, L. Y.; Liu, G. F.; Zheng, S. L.; Ye, B. H.; Chen, X. M.: Helical ribbons of cadmium(II) and zinc(II) dicarboxylates with bipyridyl-like chelates - syntheses, crystal structures and photoluminescence. Eur. J. Inorg. Chem. (2003) 2965-2971.
6. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
7. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.