Asian Journal of Chemistry; Vol. 26, No. 12 (2014), 3665-3666

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2014.16945

NOTE

Hydrothermal Synthesis and Crystal Structure of Hexaaqua-Zinc(II) 5-Nitro-benzene-1,2,3-tricarboxylic Acid

S.H. LI* and W.J. FU

College of Chemistry and Chemical Engineering, Luoyang Normal University, Henan, P.R. China

*Corresponding author: Tel/Fax: +86 379 65515113; E-mail: shihui471022@126.com

Received: 27 December 2013; *Accepted*: 15 April 2014; *Published online*: 5 June 2014; AJC-15317

A new zinc complex with the formula $Zn(C_{18}H_{28}N_2O_{26})$ is formed by reacting $Zn(OAc)_2.2H_2O$ with 5-nitro-benzene-1,2,3-tricarboxylic acid. The compound has been characterized by X-ray single-crystal diffraction, compound shows a one-dimensional framework. The 1D supramolecular structure is formed *via* hydrogen bonding connection.

Keywords: Coordination polymer, Crystal structure, Zinc(II), $Zn(C_{18}H_{28}N_2O_{26})$.

Recently, metal-organic hybrid materials in coordination chemistry and material chemistry research hot spot, on the one hand is because this kind of compounds with unique structures, on the other hand is their unique properties. More carboxylic acid ligands is widely applied in the metal-organic hybrid material preparation[1-4]. Through this kind of carboxylic acid such as 1,3-phthalate and transition metal ions, a lot of the structure, properties, synthesis of different complexes. Zinc is an important element of life, is also a kind of unique fluorescent properties of the elements, study the structure and properties of zinc complexes has important practical significance.

All reagent and solvents employed were commercially available and used as received without further purification.

General procedure: A mixture of $Zn(OAc)_2.2H_2O$ (0.2 mmol), 5-nitro-benzene-1,2,3-tricarboxylic acid (0.20 mmol) and distilled water (20 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner 433 K for three days, followed by slow cooling to room temperature. The sample was cooled to room temperature and filtered. The obtained solid was washed by deionized water and dried. Then the colourless block-shaped crystals were obtained (Fig. 1).

Detection method: Diffraction intensity data of the single crystal of the five compounds were collected on a Bruker SMART APEX-II CCD diffractometer equipped with a graphite monochromated MoK_α radiation (λ = 0.71073 Å) by using a ω-scan mode. All the structures were solved by direct methods and refined by full-matrix least-squares methods on F^2 using the program SHEXL 97[5]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrically calculations and their positions and thermal parameters were fixed during the structure refinement. The crystallographic data and experimental details of structural analyses for coordination polymers are summarized in Table-1. Selected bond and angle parameters are listed in Table-2.

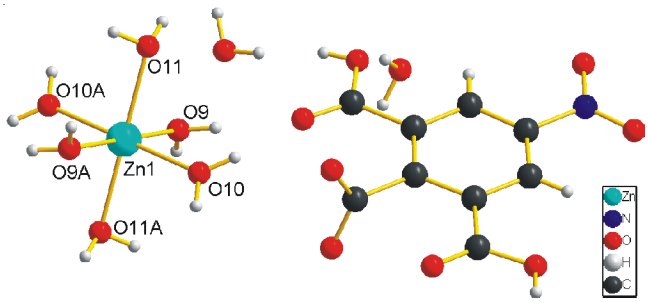

Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

Each symmetric unit containing half zinc ion, six complexing water, one 5-nitro-benzene-1,2,3-tricarboxylic acid. The carboxylic acid groups only 2a carboxylic acid group lose protons, form a negative valence anion. Zinc ion and ligand, six water molecules to form a somewhat distorted octahedral, In addition, In the molecules of the title compound, The bond length of Zn(1)-O(9), Zn(1)-O(10), Zn(1)-O(11) are found to be only 2.125(6), 2.148(6), 2.183(7)% A, respectively. In addition, inspection reveals that there are several obvious hydrogen bond interactions between the adjacent unitsmentioned above involving to the ammonium cation, the carboxylic and the carboxylate group as well as the water molecule. Some are

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY FOR $Zn(C_{18}H_{28}N_2O_{26})$

Empirical formula	$C_{18}H_{28}N_2O_{26}Zn$
Formula weight	753.79
Crystal system space group	Triclinic, P-1
Unit cell dimensions	a = 6.850(19) Å; b = 6.92(2) Å c =19.25(6) Å
Volume (Å^3)	802(4)
θ range for data collection	3.19-25.50
Final R indices [I > 2σ (I)]	R_1 = 0.0787, wR_2 = 0.2106
Z, calculated density (mg/m^3)	1, 1.562
Absorption coefficient (mm^{-1})	0.869
F (000)	388
Limiting indices	-8 ≤ h ≤ 8; -8 ≤ k ≤ 8; -21 ≤ 1 ≤ 23
Largest diff. peak and hole (e/Å^3)	0.750 and-0.755
Goodness-of-fit on F^2	1.089
R indices (all data)	R_1 = 0.0950; wR_2 = 0.2216

TABLE-2
SELECTED BOND LENGTHS (Å) AND ANGLES (°) FOR $Zn(C_{18}H_{28}N_2O_{26})$

Zn(1)-O(9)	2.125(6)	N(1)-O(8)	1.236(6)
Zn(1)-O(10)	2.148(6)	N(1)-O(7)	1.241(6)
Zn(1)-O(11)	2.183(7)	N(1)-C(5)	1.523(8)
O(9)-Zn(1)-O(9)#1	180.00(9)	O(9)-Zn(1)-O(10)#1	87.4(2)
O(9)#1-Zn(1)-O(10)#1	92.6(2)	O(9)#1-Zn(1)-O(10)	87.4(2)
O(10)#1-Zn(1)-O(11)#1	89.51(13)	O(9)-Zn(1)-O(11)#1	89.9(2)
O(10)-Zn(1)-O(11)#1	90.49(13)	O(9)#1-Zn(1)-O(11)#1	90.1(2)
O(10)-Zn(1)-O(11)	89.51(13)	O(8)-N(1)-O(7)	120.9(5)
O(10)#1-Zn(1)-O(11)	90.49(13)	O(8)-N(1)-C(5)	119.1(4)
O(9)#1-Zn(1)-O(11)	89.9(2)	O(7)-N(1)-C(5)	120.0(4)
O(9)-Zn(1)-O(11)	90.1(2)	O(9)-Zn(1)-O(10)	92.6(2)

Symmetry codes: (i) –x + 2, -y + 1, -z

listed as follows: O(1)-H(1)···O(13)#2, [O···O = 2.768(7) Å, O–H···O = 166.4°]; O(6)-H(6)···O(13)#3, [O···O = 2.751(7) Å, O–H···O = 174.6°]; O(9)-H(1W)···O(3)#2, [O···O = 2.860(7) Å, O–H···O = 128.5°]; O(9)-H(2W)···O(2), [O···O = 2.755(8) Å, O–H···O = 162.5°]; O(10)-H(3W)···O(3), [O···O = 2.907(8) Å, O–H···O = 148.2°]; O(10)-H(4W)···O(12)#4, [O···O = 2.843(7) Å, O–H···O = 173.9°]; O(11)-H(5W)···O(12)#5, [O···O = 2.880(8) Å, O–H···O = 177.8°]; O(11)-H(6W)···O(4)#6, [O···O = 2.860(8) Å, O–H···O = 173.4°]; O(12)-H(7W)···O(4)#7, [O···O = 2.991(7) Å, O–H···O = 154.5°]; O(12)-H(8W)···O(9)#7, [O···O = 3.435(8) Å, O–H···O = 163.2°]; O(13)-H(9W)···O(3), [O···O = 2.817(8) Å, O–H···O = 166.0°]; O(13)-H(10W)···O(4)#7, [O···O = 2.798(7) Å, O–H···O = 162.3°]. Symmetry codes: #1 -x + 2, -y + 1, -z; #2 x + 1, y, z; #3 x-1, y-1, z; #4 -x + 1, -y + 1, -z; #5 -x + 2, -y + 2, -z; #6 x + 1,y + 1, z; #7 x, y + 1, z. The chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 1D framework (Fig. 2).

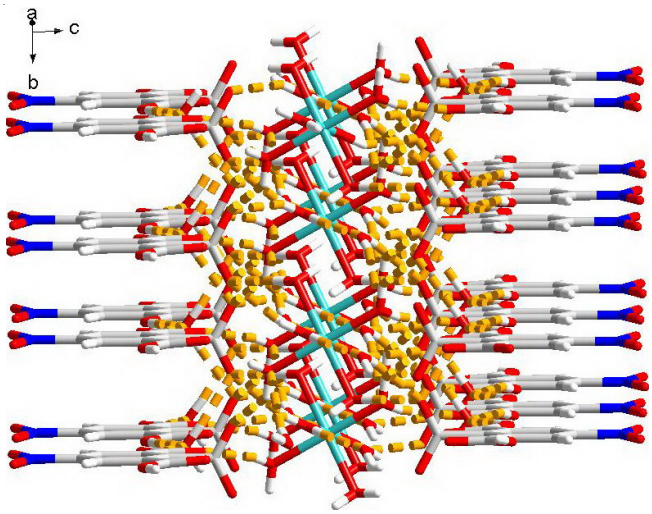

Fig. 2. 1D structure formed *via* hydrogen bonding interactions

REFERENCES

1. J.-F. Ma, J. Yang, Li, S.-Y. Song, H.-J. Zhang, H.-S. Wang and K.-Y. Yang, *Cryst. Growth Des.*, **5**, 807 (2005).
2. M.A. Braverman and R.L. LaDuca, *Cryst. Growth Des.*, **7**, 2343 (2007).
3. N. Zheng, X. Bu, H. Lu, Q. Zhang and P. Feng, *J. Am. Chem. Soc.*, **127**, 11963 (2005).
4. B. Zhao, P. Cheng, Y. Dai, C. Cheng, D.Z. Liao, S.P. Yan, Z.H. Jiang and G.L. Wang, *Angew. Chem. Int. Ed.*, **42**, 934 (2003).
5. G.M. Sheldrick, SHELXTL97, Program for the Refinement of Crystal Structure, University of Gottingen, Germany (1997).