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NOTE

Hydrothermal Synthesis and Crystal Structure of Thiophene-2,5-dicarboxylate Zinc(II)

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One new zinc complex with formula $Zn(C_6H_6O_6S)$ is formed by reacting $Zn(OAc)_2{\cdot}2H_2O$ and thiophene-2,5-dicarboxylic acid. The complex has been characterized by X-ray single-crystal diffraction, compound shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding connection.

Keywords: Coordination polymer, Crystal structure, Zinc(II), $Zn(C_6H_6O_6S)$.

In recent years, the design and synthesis of novel organic-in organic hybrid materials have provoked significant interest owing to their fascinating properties and great potential applications[1]. Recently, the family of hybrid materials based on thiophene carboxylates have been synthesized under hydro thermal conditions. Herein, we report hydrothermal synthesis and crystal structure of a new hybrid material. To the best of our knowledge, this is the first example of a hybrid material constructed from thiophene-2,5-dicarboxylic acid[2,3].

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

A mixture of $Zn(OAc)_2{\cdot}2H_2O$ (0.1 mmol, 0.0347 g), thiophene-2,5-dicarboxylic acid (0.50 mmol, 0.090 g) and distilled water (8 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner 433 K for four days, followed by slow cooling to room temperature. Colourless crystals of the compound formed.

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 ($\lambda = 0.71073$ 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[4]. 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.

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY FOR $Zn(C_6H_6O_6S)$

Empirical formula	$C_6H_6O_6SZn$
Formula weight	271.54
Crystal system space group	Orthorhombic, C222(1)
Unit cell dimensions	a = 4.974(2) Å b = 12.000(6) Å c = 14.514(7) Å
Volume (Å^3)	866.4(7)
θ range for data collection	2.81-25.48
Final R indices [I > 2σ(I)]	$R_1 = 0.0241$; $wR_2 = 0.0548$
Z, Calculated density (mg/m^3)	4, 2.082
Absorption coefficient (mm^{-1})	3.075
F(000)	544
Limiting indices	-5 ≤ h ≤ 5; -14 ≤ k ≤ 13; -17 ≤ l ≤ 7
Largest diff. peak and hole (e/Å^3)	0.492 and -0.216
Goodness-of-fit on F^2	1.132
R indices (all data)	R_1=0.0301; $wR_2 = 0.0565$

TABLE 2
SELECTED BOND LENGTHS (Å) AND ANGLES (°) FOR $Zn(C_6H_6O_6S)$

Zn(1)-O(1)	1.981(2)	Zn(1)-O(2)	2.022(2)
Zn(1)-O(2)#1	2.022(2)	Zn(1)-O(1)#1	1.981(2)
O(1)#1-Zn(1)-O(2)#1	103.40(9)	O(1)-Zn(1)-O(2)	103.40(9)
O(1)#1-Zn(1)-O(2)	133.41(9)	O(2)#1-Zn(1)-O(2)	99.33(11)
O(1)-Zn(1)-O(1)#1	89.55(14)	O(1)-Zn(1)-O(2)#1	133.41(9)
Zn(1)-O(1)-H(1W)	125.8	C(2)-S(1)-C(2)#2	91.4(2)
Zn(1)-O(1)-H(2W)	117.5	H(1W)-O(1)-H(2W)	113.0

Symmetry codes: #1 x, -y + 1, -z; #2 –x + 1, y, -z + 1/2

The molecular structure of $Zn(C_6H_6O_6S)$ is shown in Fig. 1.

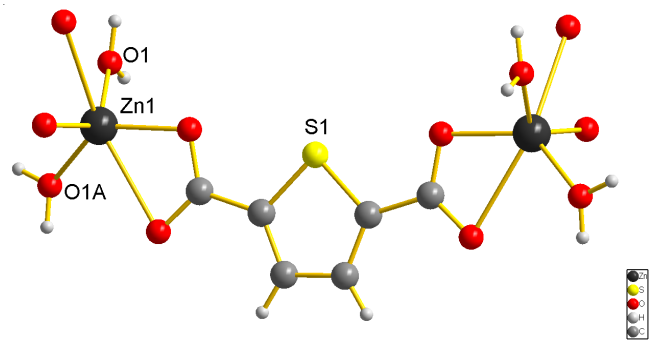

Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

X-ray diffraction analysis revealed that the fundamental building unit consists of Zn(II) ion and thiophene-2,5-dicarboxylate as bridging ligands to construct a new coordination polymer. On the thiophene ring, the hydrogen atoms were assigned with Uiso(H) = 1.2 Ueq(C) and included in the final refinement by using geometrical restraints, with d(C–H) = 0.93 Å. The zinc atom is six-coordinated in a distorted Polyhedronl manner and one oxygen atoms O_2 from thiophene-2,5-dicarboxylate molecules. The Zn-O bond lengths are 2.022(2) Å. The hydrogen bonds as follows: O(1)-H(2W)···S(1)#3, [O···O = 3.486(3) Å, O–H···O = 125.6°]; O(1)-H(2W)···O(2)#3, [O···O = 2.757(3) Å, O--H···O = 157.8°]; O(1)-H(1W)···O(3)#4, [O···O = 2.662(3) Å, O–H···O = 165.1°]. Symmetry codes: #3 x + 1, y, z; #4 x + 1/2, y + 1/2, z. The chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 3D framework (Fig. 2).

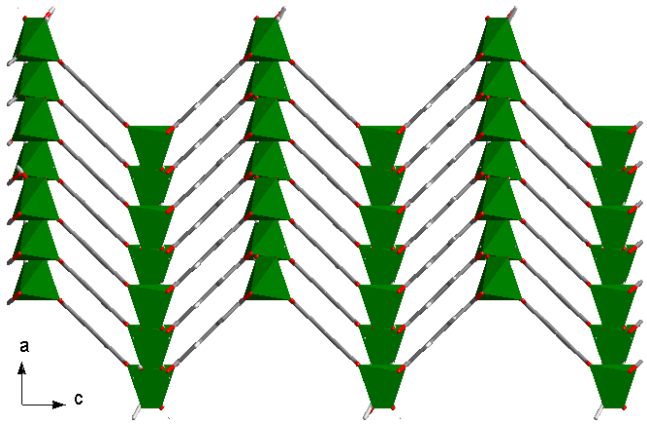

Fig. 2. 3D structure formed *via* hydrogen bonding interactions

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