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NOTE

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

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One new cadmium compound using $Cd(OAc)_2{\cdot}2H_2O$, thiophene-2,5-dicarboxylic acid with m.f. $C_{16}H_{26}O_{15}S_2Cd_2$ has been successfully synthesized. The compound has been characterized by X-ray single-crystal diffraction and shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding.

Keywords: Coordination polymer, Crystal structure, Cadmium(II), Thiophene-2,5-dicarboxylic acid.

In recent years, the design and synthesis of novel organic-inorganic 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 hydrothermal 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].

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

A mixture of $Cd(OAc)_2{\cdot}2H_2O$ (0.1 mmol, 0.04 g), thiophene-2,5-dicarboxylic acid (0.50 mmol, 0.08 g) and distilled water (9 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner 413 K for 4 days, followed by slow cooling to room temperature. Colourless crystals of the compound are 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[3]. 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 COMPLEX

Empirical formula	$C_{16}H_{26}O_{15}S_2Cd_2$
Formula weight	747.29
Crystal system space group	Monoclinic, P2(1)/c

Unit cell dimensions	a = 10.4101(19) Å;
	b = 17.176(3) Å;
	c = 6.8466(12) Å

Volume (Å^3)	1158.0(4)
θ range for data collection	2.38 -25.50
Final R indices [I > 2σ(I)]	R_1= 0.0648, wR_2= 0.1853
Z, Calculated density (mg/m^3)	2, 2.143
Absorption coefficient (mm^{-1})	2.092
F(000)	740
Limiting indices	-12 ≤ h ≤ 12; -20 ≤ k ≤ 20; -8 ≤ l ≤ 8
Largest diff. peak and hole (e/Å^3)	2.721 and -2.262
Goodness of fit on F^2	1.085
R indices (all data)	R_1= 0.0807, wR_2= 0.2040

X-ray diffraction analysis revealed that the fundamental building unit consists of Cd(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

TABLE-2
SELECTED BOND LENGTHS
(Å) AND ANGLES (°) FOR COMPLEX

Cd(1)-O(2)	2.307(6)	Cd(1)-O(1)	2.367(6)
Cd(1)-O(5)#1	2.338(5)	Cd(1)-O(6)	2.399(4)
Cd(1)-O(3)	2.337(6)	Cd(1)-O(7)	2.402(5)
Cd(1)-O(4)#1	2.450(5)	O(1)-Cd(1)-O(7)	91.85(18)
O(2)-Cd(1)-O(5)#1	91.4(2)	O(1)-Cd(1)-O(6)	89.27(16)
O(2)-Cd(1)-O(3)	92.8(2)	O(3)-Cd(1)-O(6)	135.75(16)
O(5)#1-Cd(1)-O(3)	83.91(17)	O(5)#1-Cd(1)-O(6)	140.14(16)
O(2)-Cd(1)-O(1)	179.40(17)	O(2)-Cd(1)-O(6)	90.44(18)
O(5)#1-Cd(1)-O(1)	89.15(18)	O(3)-Cd(1)-O(1)	87.1(2)

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

refinement by using geometrical restraints, with d(C–H) = 0.93 Å. The asymmetric unit of the title structure contains one Cd(II) ion, one thiophene-2,5-dicarboxylate molecule. The cadmium atom is six-coordinated in a distorted polyhedronl manner and four oxygen atoms (O1, O6, O4, O7 and O5) from one thiophene-2,5-dicarboxylate molecule (Fig. 1). The Cd-O bond lengths are 2.367(6) Å, 2.399(4) Å, 2.450(5) Å, 2.402(5) Å and 2.338(5) Å, respectively. In addition, the carboxylic and the carboxylate group as well as the water molecule. Some are listed as follows: O(2)-H(3W)···O(6)#3, [O···O = 2.688(7)\%A, O–H···O = 173.2\%]; O(2)-H(4W)···O(1)#4, [O···O = 3.058(7)\%A, O–H···O = 171.1\%]; O(3)-H(5W)···O(1), [O···O = 3.240(8)\%A, O–H···O = 178.1\%]; O(3)-H(6W)···O(7)#5, [O···O = 2.798(8)\%A, O–H···O = 138.4\%]; O(1)-H(1D)···O(4)#6, [O···O = 2.702(7)\%A, O-H···O = 157.4\%]. Symmetry codes: #1 -x,-y,-z; #2 -x+1, -y,-z; #3 -x+1,-y,-z+1; #4 -x,-y,-z+1; #5 x,y,z+1; #6 -x+1, -y+1,-z+1. The chains are further assembled by the intermolecular hydrogen bonding interaction leading to the formation of a 3D framework (Fig. 2).

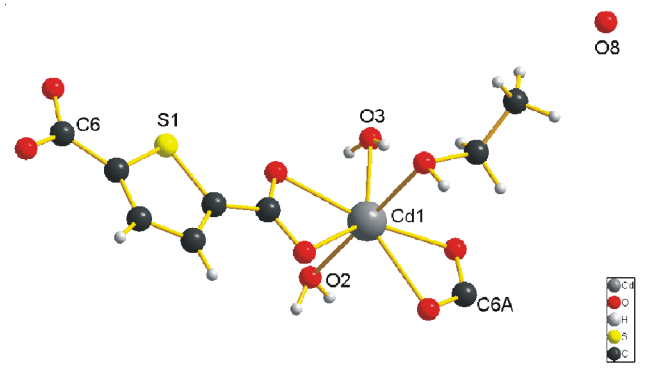

Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

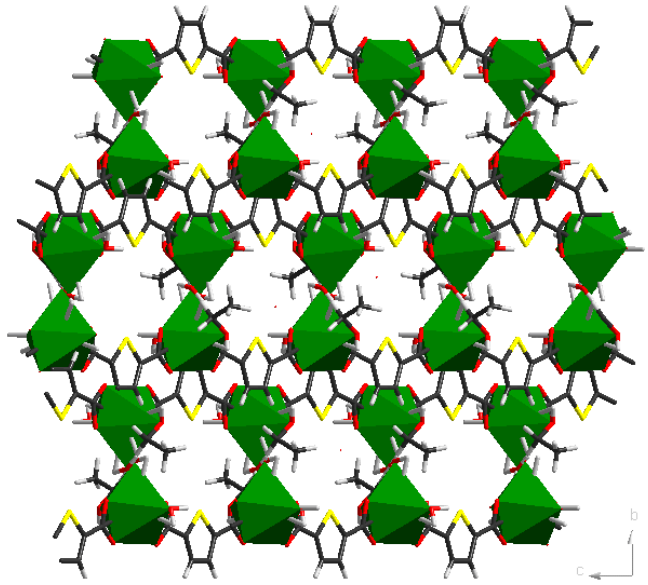

Fig. 2. 3D structure formed *via* hydrogen bonding interactions

REFERENCES

1. Y.C. Liang, R. Cao, W.P. Su, M.C. Hong and W.J. Zhang, *Angew. Chem. Int. Ed.*, **39**, 3304 (2000).
2. B. Zhao, P. Cheng, X.Y. Chen, C. Cheng, W. Shi, D.Z. Liao, S.P. Yan and Z.H. Jiang, *J. Am. Chem. Soc.*, **126**, 3012 (2004).
3. G.M. Sheldrick, SHELXTL97, Program for the Refinement of Crystal Structure, University of Gottingen, Germany (1997).