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NOTE

Hydrothermal Synthesis and Crystal Structure of *bis*-[4-{(E)-2-(Pyridin-4-yl)vinyl}pyridine] Manganese(II) Thiophene-2,3-dicarboxylate

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One new manganese compound $Mn(OAc)_2{\cdot}4H_2O$ with thiophene-2,3-dicarboxylic acid and bpp [4-{(E)-2-(pyridin-4-yl)vinyl}pyridine] has been successfully synthesized. Compound shows a one-dimensional framework. The 3D supramolecular structure is formed *via* hydrogen bonding formation.

Keywords: Coordination polymer, Crystal structure, Manganese(II).

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[1,2]. Recently, the family of hybrid materials based on thiophene carboxylate 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 formed from thiophene-2,3-dicarboxylic acid[3].

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

General procedure: A mixture of $Mn(OAc)_2.4H_2O$ (0.15 mmol), thiophene-2,3- dicarboxylic acid (0.25 mmol) and bpp [4-{(E)-2-(pyridin-4-yl)vinyl}pyridine] (0.30 mmol) and distilled water (8 mL) was heated in a 25 mL stainless steel reactor with a Teflon liner 403 K for 36 h, followed by slow cooling to room temperature. Yellow crystals of the compound formed.

Diffraction intensity data of the single crystal of the five compounds were collected on a Bruker SMART APEXII 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[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.

X-ray diffraction analysis revealed that the fundamental building unit consists of Mn(II) ion and bpp [4-{(E)-2-(pyridin-4-yl)vinyl}pyridine] as bridging ligands to construct a new coordination polymer (Fig. 1). The asymmetric unit of the title structure contains one Mn(II) ion, one thiophene-2,3-dicarboxylate and three bpp molecules. Manganese atom is six-coordinated in a distorted octahedral manner and two nitrogen atoms (N1 and N3) from two 4-[(E)-2-(pyridin-4-yl)vinyl]-pyridine ligands. The (Mn-N) bond lengths are 2.259(6) Å, 2.329(5) Å, respectively and four oxygen atoms (O3, O4, O5 and O6) from the water molecule. The Mn-O bond lengths are 2.205(5) Å, 2.198(5) Å, 2.241(5) Å and 2.161(4) 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)...N(2)#1, [O...O = 2.701(8) \%A, O--H...O = 128.1\%]; O(2)-H(4W)...O(4)#2, [O...O = 2.753(8)\%A, O--H...O = 178.5\%]; O(3)-H(6W)...O(6), [O...O = 3.096(7)\%A, O--H...O = 179.5\%]; O(4)-H(7W)...N(6)#3, [O...O = 2.723(7)\%A, O--H...O = 172.8\%]; O(4)-H(8W)...O(5), [O...O = 3.256(8)\%A, O--H...O = 179.6\%]; O(6)-H(12W)...O(5), [O...O = 3.024(8) \%A, O--H...O = 120.4\%]; O(9)-H(9)...O(8), [O...O = 2.453(9)\%A, O--H...O = 168.6\%]. Symmetry codes: #1 x, y + 1, z; #2 -x + 1, -y, -z + 1; #3 -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).

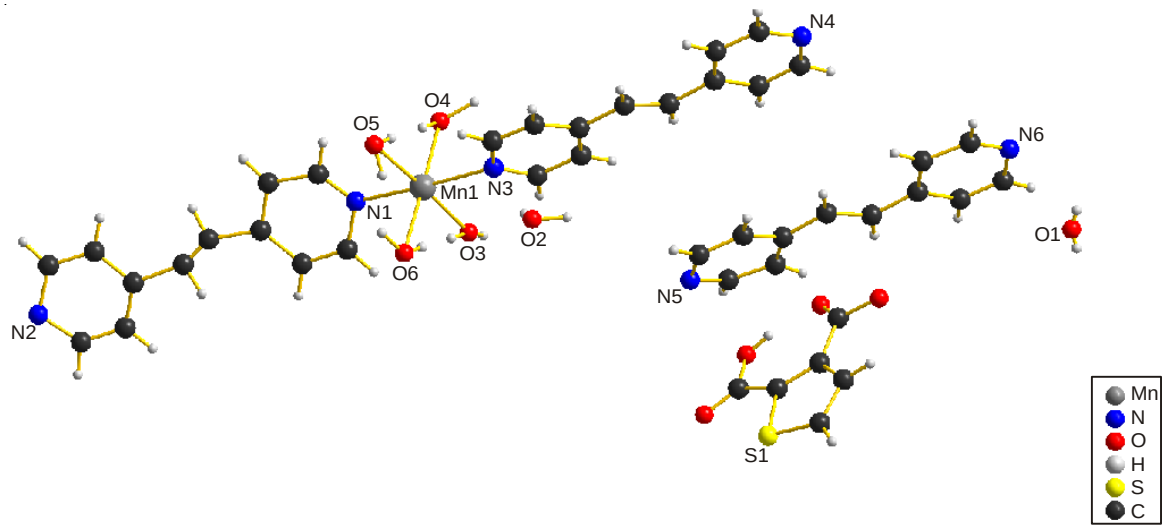

Fig. 1. Molecular structure of the title compound at 30 % probability displacement ellipsoids

TABLE-1
CRYSTALLOGRAPHIC DATA AND STRUCTURE REFINEMENT SUMMARY FOR MANGANESE COMPLEX

Empirical formula	$C_{42}H_{45}N_6O_{10}SMn$	Z, Calculated density (mg/m^3)	2, 0.740
Formula weight	880.84	Absorption coefficient (mm^{-1})	0.228
Crystal system space group	Triclinic, P-1	F(000)	920
Unit cell dimensions	a = 15.3362(19)Å	Limiting indices	$-17 \le h \le 18$
	b = 16.749(2) Å		$-18 \le k \le 20$
	c = 18.706(2)Å		$0 \le l \le 22$
Volume (Å^3)	3955.4(9)	Largest diff. peak and hole (e/Å^3)	0.397 and -0.356
θ Range for data collection	2.29-25.50	Goodness-of-fit on F^2	1.082
Final R indices [I>2σ(I)]	$R_1 = 0.1136$; $wR_2 = 0.3174$	R indices (all data)	$R_1 = 0.2029$; $wR_2 = 0.3406$

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

Mn(1)-O(6)	2.161(4)	Mn(1)-O(5)	2.241(5)
Mn(1)-O(4)	2.198(5)	Mn(1)-N(1)	2.259(6)
Mn(1)-O(3)	73.35(9)	Mn(1)-N(3)	2.329(5)
O(6)-Mn(1)-O(4)	177.22(18)	O(4)-Mn(1)-N(1)	88.43(19)
O(6)-Mn(1)-O(3)	90.3(2)	O(3)-Mn(1)-N(1)	89.06(19)
O(4)-Mn(1)-O(3)	88.5(2)	O(5)-Mn(1)-N(1)	89.79(19)
O(6)-Mn(1)-O(5)	86.8(2)	O(6)-Mn(1)-N(3)	95.15(17)
O(4)-Mn(1)-O(5)	94.3(2)	O(4)-Mn(1)-N(3)	87.38(16)
O(3)-Mn(1)-O(5)	176.9(2)	O(3)-Mn(1)-N(3)	90.28(19)
O(6)-Mn(1)-N(1)	89.03(19)	N(1)-Mn(1)-N(3)	175.8(2)

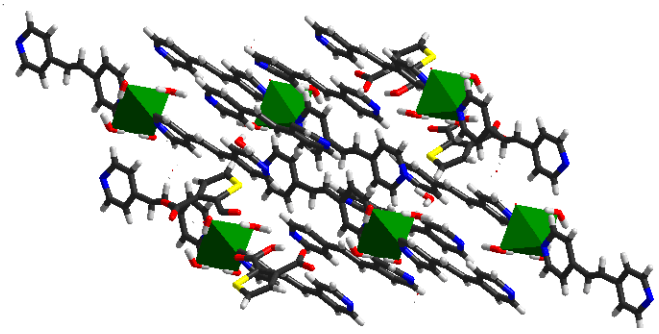

Fig. 2. 3D Structure formed *via* hydrogen bonding interactions

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