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## Structural and photoluminescent studies of non-centrosymmetric manganese(II) *N*-(2-pyridylmethyl)-(L)-alanine) dicyanamide

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### RESEARCH ARTICLE



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### ABSTRACT

The dinuclear compound,  $[\text{Mn}_2(\text{Pyala})_2(\text{Dca})_2(\text{H}_2\text{O})]_n \cdot 2\text{H}_2\text{O}$  (1) (Pyala = *N*-(2-pyridylmethyl)-L-alanine and Dca = dicyanamide anion) has been synthesized and characterized by elemental analysis, IR and single crystal X-ray diffraction techniques. The crystal data for  $\text{C}_{22}\text{H}_{26}\text{Mn}_2\text{N}_{10}\text{O}_6$ : orthorhombic, space group  $P2_12_12_1$  (no. 19),  $a = 10.3728(8)$  Å,  $b = 15.9780(12)$  Å,  $c = 16.3585(13)$  Å,  $V = 2711.2(4)$  Å<sup>3</sup>,  $Z = 4$ ,  $T = 198(2)$  K,  $\mu(\text{MoK}\alpha) = 0.989$  mm<sup>-1</sup>,  $D_{\text{calc}} = 1.559$  g/cm<sup>3</sup>, 129607 reflections measured ( $3.564^\circ \leq 2\theta \leq 60.046^\circ$ ), 7923 unique ( $R_{\text{int}} = 0.0324$ ,  $R_{\text{sigma}} = 0.0155$ ) which were used in all calculations. The final  $R_1$  was 0.0169 ( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.0458 (all data). The obtained non-centrosymmetric dinuclear Mn(II) complex contains two unique Mn(II) cations with similar octahedral coordination environment. Photoluminescent measurements on the complex in the solid state show that it displays strong photoluminescence at 442 nm.

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### 1. Introduction

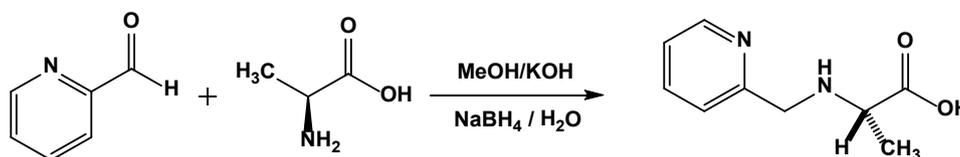
The recent development in designing the novel solid-state structures and preparation of polynuclear complexes with one-, two- or three-dimensional homo- or hetero-thallic infinite frameworks and also mono- and di-nuclear complexes are the subject of great interest due to their widespread applications in various fields like molecular magnetism, photochemical activity, intervalence electron transfer reaction and catalytic reactions [1-3]. The pseudo halide ligand dicyanamide (Dca,  $[\text{N}(\text{CN})_2]^-$ ), which was first utilized by Köhler [4-7], has been widely studied for many years to design polymeric structures [8]. It is a remarkably versatile building block for the construction of metal-organic architectures, since it can act as a mono-, bi- and tri-dentate ligand, yielding a variety of novel structures [9]. A notable feature of metal-Dca coordination polymers is the ability of cations to template

anionic  $[\text{M}(\text{Dca})_3]^-$  networks [10]. The prime cases of previously reported three-dimensional metal-Dca networks are neutral binary systems. The series  $\text{MX}_2$  (X:  $[\text{N}(\text{CN})_2]^-$ ; M:  $\text{Cr}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$  or  $\text{Cu}^{2+}$ ) and related complexes have attracted increased interest because of their rutile-like structures containing chains of doubly-bridged metal atoms with  $\text{M}(\text{NCNCN})\text{M}$  units [11]. The pseudo halide, dca ligand has been seen in many cases to play an instructive role in building coordination polymers.

On the other hand, non-centrosymmetric materials are of particular importance in the field of material chemistry for the large number of symmetry-dependent properties, they can possess, including circular dichroism, pyroelectricity, and non-linear optical behavior [12-14]. While purposefully engineering these materials can be difficult, one method for eliminating centrosymmetry in crystalline materials is co-crystallization with an enantiopure chiral compound [15]. In this

**Table 1.** Crystal data and details of the structure refinement for compound 1.

Parameters	Compound 1
Empirical formula	C <sub>22</sub> H <sub>26</sub> Mn <sub>2</sub> N <sub>10</sub> O <sub>6</sub>
Formula weight	636.41
Temperature (K)	198(2)
Crystal system	Orthorhombic
Space group	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
a (Å)	10.3728(8)
b (Å)	15.9780(12)
c (Å)	16.3585(13)
Volume (Å <sup>3</sup> )	2711.2(4)
Z	4
ρ <sub>calc</sub> (g/cm <sup>3</sup> )	1.559
μ (mm <sup>-1</sup> )	0.989
F(000)	1304.0
Crystal size (mm <sup>3</sup> )	0.280 × 0.220 × 0.100
Radiation	MoKα (λ = 0.71073)
2θ range for data collection (°)	3.564 to 60.046
Index ranges	-12 ≤ h ≤ 14, -22 ≤ k ≤ 22, -22 ≤ l ≤ 22
Reflections collected	129607
Independent reflections	7923 [R <sub>int</sub> = 0.0324, R <sub>sigma</sub> = 0.0155]
Data/restraints/parameters	7923/5/400
Goodness-of-fit on F <sup>2</sup>	1.035
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0169, wR <sub>2</sub> = 0.0455
Final R indexes [all data]	R <sub>1</sub> = 0.0178, wR <sub>2</sub> = 0.0458
Largest diff. peak/hole (e.Å <sup>-3</sup> )	0.22/-0.18
Flack parameter	0.008(2)

**Scheme 1.** Synthetic scheme of *N*-(2-pyridylmethyl)-L-alanine.

way, provided that the chiral compound is not capable of racemization, the potential point groups are limited only to those which are chiral, and therefore non-centrosymmetric. The amino acid L-alanine plays an important role in determining the structure of proteins, due to its structural rigidity. L-alanine has also been shown to be a good candidate for synthesizing non-centrosymmetric co-crystals [16]. Inspired by these considerations, and in continuation with studies on Pyala ligand [17], we elected to study the reaction between manganese (II) and dicyanamide anion in the presence of chiral bridging ligand, *N*-(2-pyridylmethyl)-L-alanine.

## 2. Experimental

### 2.1. Materials and physical measurements

All reagents and solvents used for the synthesis were of reagent grade. Manganese acetate tetrahydrate and sodium dicyanamide were from commercial sources and used without further purification. Ethanol was dried and distilled according to standard methods. Elemental analysis for carbon, nitrogen and hydrogen were carried out on a Fisons instrument 1108 CHNS-O. Luminescence spectra were measured on a HitachiF-4500 spectrophotometer at room temperature. The X-ray diffraction was carried out with a CCD bidimensional diffractometer using monochromatic radiation, λ(MoKα) = 0.71073 Å, operating at 50 kV and 40 mA. The crystal structure of [Mn<sub>2</sub>(Pyala)<sub>2</sub>(Dca)<sub>2</sub>(H<sub>2</sub>O)]<sub>n</sub>·2H<sub>2</sub>O (**1**) was determined by single crystal X-ray diffraction on a Bruker APEX-II using monochromatic MoKα radiation (λ = 0.71073 Å) at a temperature of 198 K and integrated with SAINT-Plus program [18], and absorption corrections were carried out by multi-scan method by SADABS [19]. The structure was solved by direct methods and refined against F<sup>2</sup> by full-matrix least-squares techniques with SHELXTL [20]. All non-hydrogen atoms were refined with anisotropic displacement parameters.

Hydrogen atoms were included from calculated positions and refined riding their respective parent atoms with isotropic displacement parameters.

### 2.2. Synthesis of [Mn<sub>2</sub>(Pyala)<sub>2</sub>(Dca)<sub>2</sub>(H<sub>2</sub>O)]<sub>n</sub>·2H<sub>2</sub>O (**1**)

Synthesis of *N*-(2-pyridylmethyl)-L-alanine (Pyala) was as reported in literature (Scheme 1) [16]. Pyala (0.36 g, 2 mmol) in 10 mL water:ethanol mixture (1:1, v:v) was added drop wise to a 5 mL aqueous solution of Mn(OOCH<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O (0.245 g, 1 mmol), while stirring magnetically at room temperature. After stirring for a further 10 minutes, NaN(CN)<sub>2</sub> (0.18 g, 2 mmol) in 5 mL distilled water was added drop wise and stirring continued for two hours. Suitable parallelepiped colorless crystals for X-ray analysis were obtained from the solution by slow evaporation. Yield: 70%. Anal. calcd. for C<sub>11</sub>H<sub>13</sub>MnN<sub>5</sub>O<sub>3</sub>; C, 41.52; H, 4.12; N, 22.01. Found: C, 41.46; H, 4.07; N, 22.59%.

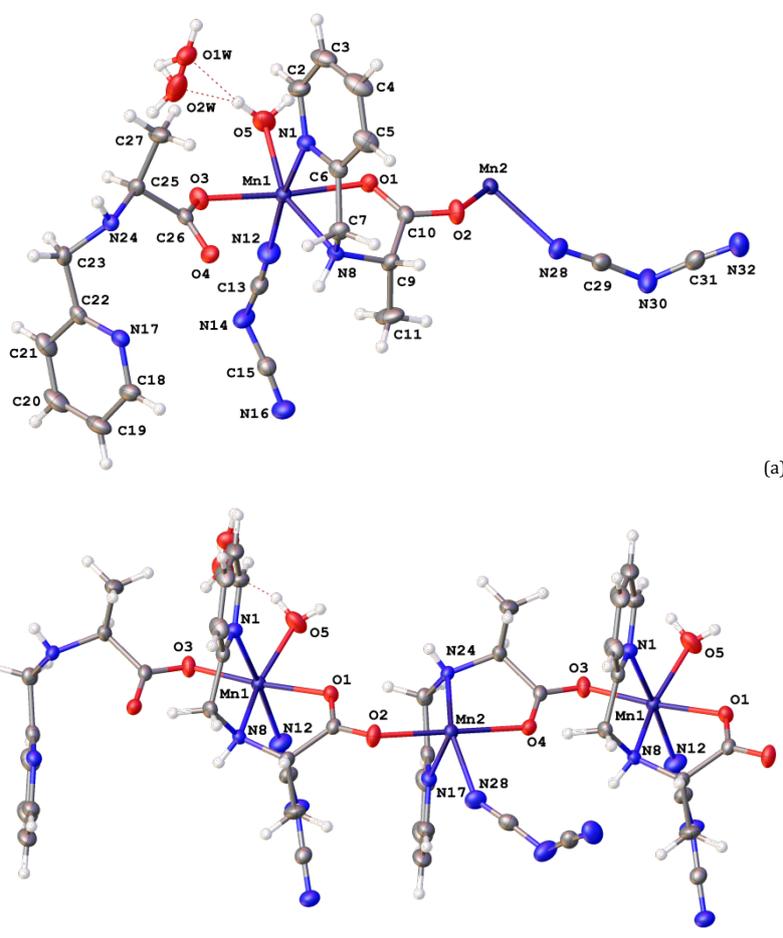
## 3. Results and discussion

The crystal structure refinement data for compound **1** is presented in Table 1. The selected bond lengths and angles are given in Table 2. The structure of [Mn<sub>2</sub>(Pyala)<sub>2</sub>(Dca)<sub>2</sub>(H<sub>2</sub>O)]<sub>n</sub>·2H<sub>2</sub>O and its atom numbering scheme is shown in Figure 1. As shown in Figure 1, there are two unique octahedrally coordinated Mn<sup>2+</sup> cations (Mn1 and Mn2) with similar coordination modes. Each Mn<sup>2+</sup> is coordinated to one Pyala ligand via N1, N8 and N24, N17; and bridged by the two carboxylate oxygen atoms (O3, O4 and O1, O2). The octahedral geometry around Mn1 is completed by one water molecule oxygen (O5W) and one Dca nitrogen (N12), while the octahedral geometry around Mn2 is completed by the coordination of two Dca nitrogen atoms, (N28 and N16) in a monodentate manner. Each Mn<sup>2+</sup> center is connected by end-to-end μ-1,5-Dca bridge via N12 and N16 in successive chains to form a 1D structure (Figure 2).

**Table 2.** Selected bond lengths [Å] and bond angles [°] for compound **1**\*.

<b>Bond lengths</b>			
Mn1-O3	2.1383(10)	Mn2-O2	2.1386(10)
Mn1-O5	2.1633(12)	Mn2-O4 <sup>i</sup>	2.1928(10)
Mn1-O1	2.1844(10)	Mn2-N16 <sup>ii</sup>	2.2087(12)
Mn1-N12	2.2069(13)	Mn2-N28	2.2246(12)
Mn1-N8	2.3262(11)	Mn2-N17 <sup>i</sup>	2.2662(11)
Mn1-N1	2.3280(12)	Mn2-N24 <sup>i</sup>	2.3355(11)
<b>Bond angles</b>			
O3-Mn1-O5	90.02(5)	O2-Mn2-O4 <sup>i</sup>	176.77(4)
O3-Mn1-O1	175.11(4)	O2-Mn2-N16 <sup>ii</sup>	90.56(5)
O5-Mn1-O1	86.36(4)	O4 <sup>i</sup> -Mn2-N16 <sup>ii</sup>	92.08(4)
O3-Mn1-N12	93.33(5)	O2-Mn2-N28	94.02(5)
O5-Mn1-N12	95.50(5)	O4 <sup>i</sup> -Mn2-N28	87.82(4)
O1-Mn1-N12	90.30(5)	N16 <sup>ii</sup> -Mn2-N28	90.36(5)
O3-Mn1-N8	107.86(4)	O2-Mn2-N17 <sup>i</sup>	88.71(4)
O5-Mn1-N8	157.12(5)	O4 <sup>i</sup> -Mn2-N17 <sup>i</sup>	88.56(4)
O1-Mn1-N8	74.87(4)	N16 <sup>ii</sup> -Mn2-N17 <sup>i</sup>	177.15(5)
N12-Mn1-N8	97.52(5)	N28-Mn2-N17 <sup>i</sup>	92.43(4)
O3-Mn1-N1	89.80(4)	O2-Mn2-N24 <sup>i</sup>	103.37(4)
O5-Mn1-N1	91.97(5)	O4 <sup>i</sup> -Mn2-N24 <sup>i</sup>	74.26(4)
O1-Mn1-N1	87.04(4)	N16 <sup>ii</sup> -Mn2-N24 <sup>i</sup>	101.99(4)
N12-Mn1-N1	171.89(5)	N28-Mn2-N24 <sup>i</sup>	158.42(4)
N8-Mn1-N1	74.40(4)	N17 <sup>i</sup> -Mn2-N24 <sup>i</sup>	75.51(4)

\* Symmetry transformations used to generate equivalent atoms: (i)  $x+1, y, z$ ; (ii)  $-x+2, y+1/2, -z+3/2$ .



**Figure 1.** Molecular structure (a) and the coordination environment of the Mn1 and Mn2 atoms (b) in compound **1** showing the atom numbering scheme.

In addition, this coordination polymer consists of  $Mn_2N_4O_2$  and  $Mn_1N_3O_3$  octahedral units as illustrated in Figure 3.

The Mn-O bond distances in compound **1** are in the range of 2.1382(5)-2.1926(4) Å, with the Mn- $O_{water}$  bond length (2.1643(5) Å) longer than the Mn- $O_{carboxylate}$  bond length (2.1382(5) Å). Furthermore, all the Mn- $N_{dca}$  bond distances are in the range of 2.2069(6)-2.2255(6) Å and are in good

agreements with previously published results [21-23]. These Mn- $N_{dca}$  bond distances are all shorter than the Mn- $N_{pyridyl}$  bond distances of 2.3326(5)-2.3362(4) Å, also consistent with earlier reports of a similar compound [16,24]. These are clear indications that the Mn- $N_{dca}$  bonds are stronger than the Mn- $N_{pyridyl}$  bonds. Overall the Mn-O bond distances are shorter than the Mn-N bond distances in compound **1**.

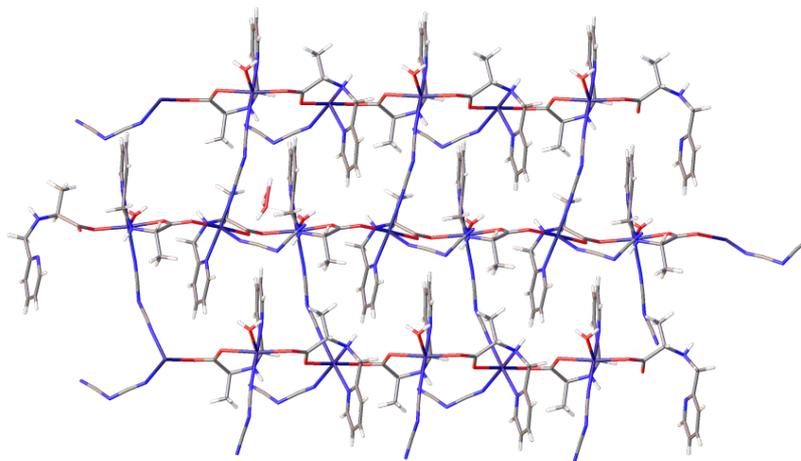


Figure 2. Schematic view of the "S" type double stranded helical chains of compound 1.

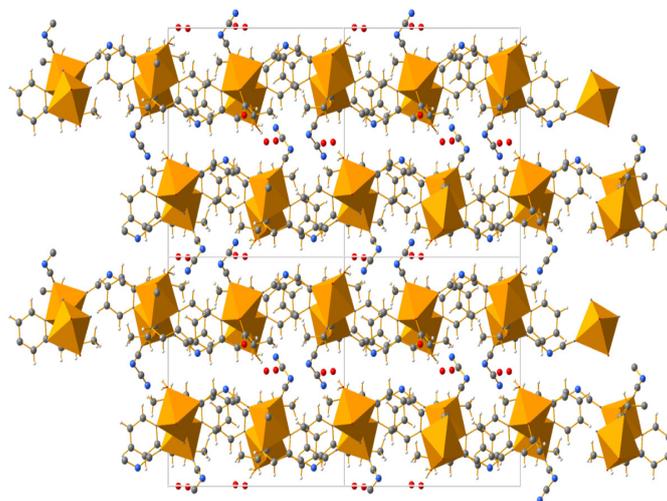


Figure 3. The octahedral chaining of  $\text{MnN}_4\text{O}_2$  and  $\text{MnN}_3\text{O}_3$ .

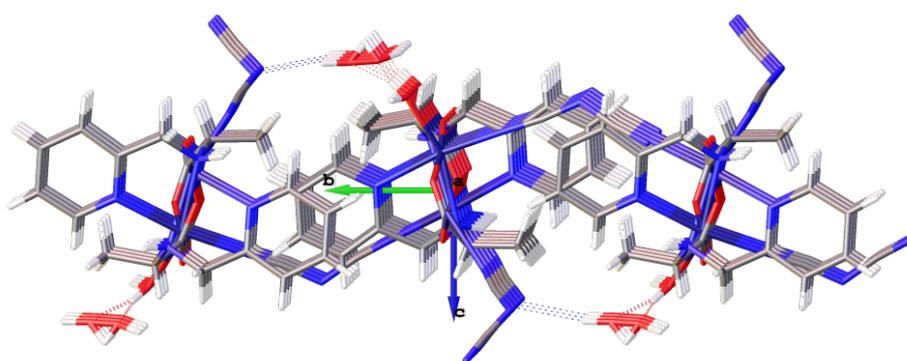


Figure 4. Packing along the *a* axis showing chiral channels.

The basal plane of the octahedron around the Mn1 center (O1, O3 and N12, N1) are almost *trans* to each other, with respective bond angles O1-Mn1-O3 ( $175.102(4)^\circ$ ) and N12-Mn1-N1 ( $171.90(4)^\circ$ ). Similarly, the basal plane of the octahedron around Mn2 center (O2, O4 and N16, N17) are also almost *trans* to each other, with respective bond angles of O2-Mn2-O4 ( $176.78(4)^\circ$ ) and N16-Mn2-N17 ( $177.10(4)^\circ$ ). The shortest Mn-Mn separation through the *pyala*-bridge is  $5.458(4)$  Å, while the distance between successive Mn1-Mn1

or Mn2-Mn2 atoms along the polymeric chain is  $10.373(8)$  Å which is similar to previously reported results of manganese dicyanamide complex [25]. The neighbouring  $\text{Mn}^{2+}$  cation are linked together by double *dca* bridges to form a one-dimensional polymeric chain connected to each other through hydrogen bonding. Compound 1 was found to exhibit some chiral channels as depicted in Figure 4. Intramolecular hydrogen bondings as well, exist in compound 1 and thus help in reinforcing the supramolecular structure.

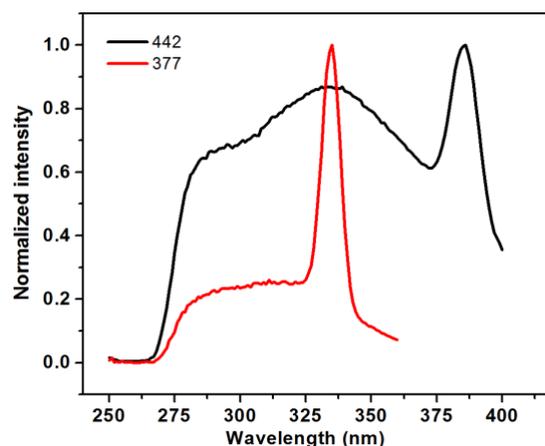


Figure 5. Photoluminescence spectra of compound 1.

### 3.1. Photoluminescent behaviour of compound 1

The photoluminescent (PL) properties of the compound were examined in solid state at room temperature (Figure 5). This compound exhibited strong fluorescent emission at 377 and 442 nm, upon excitation at 341 and 385 nm, respectively. In order to understand the nature of the emission, we examined the photoluminescence property of the free ligand and found that, the strongest emission peak is at 391 and 432 nm ( $\lambda_{ex} = 368$  nm). Therefore, the luminescence of the compound 1 may be attributed to metal to ligand charge transfer (MLCT) or ligand to metal charge (LMCT) [26-28]. These observations suggest that compound 1 may be a good candidate for potential photoactive materials.

## 4. Conclusion

A new non-centrosymmetric manganese (II) coordination polymer of *N*-(2-pyridylmethyl)-(L)-alanine and dicyanamide anion in both terminal unidentate and end-to-end  $\mu$ -1,5-bonding modes has been obtained. The dinuclear compound has two unique  $Mn^{2+}$  centers. Each  $Mn^{2+}$  center is connected by end-to-end  $\mu$ -1,5-dca bridge in successive chains to form a 1-D structure. The octahedron around the manganese (II) centers consists of  $Mn_2N_4O_2$  and  $Mn_1N_3O_3$  moieties. The stability of the complex is enhanced by  $N\cdots H$  and  $O\cdots H$  hydrogen bonding interactions. A photoluminescent measurement on the complex in the solid state shows that it displays strong photoluminescence at 442 nm.

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## Supporting information

CCDC 1508113 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>, or by e-mailing [data\\_request@ccdc.cam.ac.uk](mailto:data_request@ccdc.cam.ac.uk), or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

## Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.

Author contributions: All authors contributed equally to this work.

Ethical approval: All ethical guidelines have been adhered.

Sample availability: Samples of the compounds are available from the author.

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