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NOTE

Hydrothermal Synthesis and Structure of Phenanthroline Manganese(II) Complex

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A new manganese(II) complex with the m.f. $C_{30}H_{20}N_{10}O_2Mn_2$ was synthesized by hydrothermal reaction and characterized by IR spectra and single crystal X-ray diffraction. The crystal is in a Monoclinic system, space group C2/c with unit cell parameters: a = 16.8439(15)Å, b = 13.9842(12)Å, c = 14.4033(14)Å, α = 90º, β = 109.1090(10)º, γ = 90º, V = 3205.7(5)Å^3, Z = 4, Mr = 662.44, Dc = 1.373 Mg/cm^3, μ = 0.831 mm^{-1}, F(000) = 1344, T = 298(2) K, R = 0.0625, wR = 0.1507 for 7979 reflections with I > 2σ(I). The crystal structure analysis shows that the manganese(II) is a six-coordinated in a slightly distorted octahedron environment.

Keywords: Manganese(II) complex, Hydrothermal synthesis, Crystal structure.

There has been increasing interest of 1,10-phenanthroline (phen) transition metal complexes in the design of molecular construction due to their stability and ease of chemical modification[1-4]. In our laboratory, a series of transition metal complexes were synthesized[5-9].

In this paper, we wish to report the synthesis and crystal structure of manganese(II) complex $[Mn(phen)_2(H_2O)_2]\cdot Mn(CN)_3$.

All the reagents were of AR grade and used without further purification. IR spectra were recorded on a Nicolet 380 spectrophotometer. The crystal structure was determined by Siemens SMART CCD area-detector diffractometer.

Synthesis: An ethanolic solution (10 mL) of 1,10-phenanthroline (10 mmol) and aqueous solution (15 mL) of a mixed $Mn(CH_3COO)_2$ (5 mmol) and $K_3[Fe(CN)_6]$ (2 mmol) in presence of $L(ClO_4)_2$ were carried out in a autoclave and heated to 150 ºC for 72 h. After cooling, the yellow clubbed single crystals were obtained. Yield 33 %. IR (KBr, ν_{max}, cm^{-1}): 3560-3200, 2060, 1625, 1433, 890, 732.

Crystal structure determination: A single crystal (0.28 × 0.24 × 0.15) was selected for crystallographic data collection at 298(2) K and structure determinated with graphite monochromatic MoK$_\alpha$ radiation (λ = 0.71073Å). A total of 5278 reflections were collected in the range of 2.18º ≤ θ ≤ 25.02º, of which 2801 reflections were unique with Rint = 0.0477 and R = 0.0625 and wR = 0.1507, where w = 1/[s^2(F$_0^2$) + (0.0769P)^2 + 0.0000P], P = (F$_0^2$ + 2F$_0^2$)/3. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.614 and -0.321 e/Å^3 (CCDC No. 985895), respectively.

The atomic coordinates and thermal parameters are listed in Table-1 and the selected bond lengths and bond angles in Table-2. Respecting, Fig. 1 shows the molecular structure of the title compound. Fig. 2 shows the packing diagram of the title compound. From the Fig. 1, it is easy to see that the manganese(II) ion is six-coordinated with four nitrogen atoms from the two 1,10-phenanthroline ligands and two oxygen atoms form the two water molecules in a slightly distorted octahedron environment.

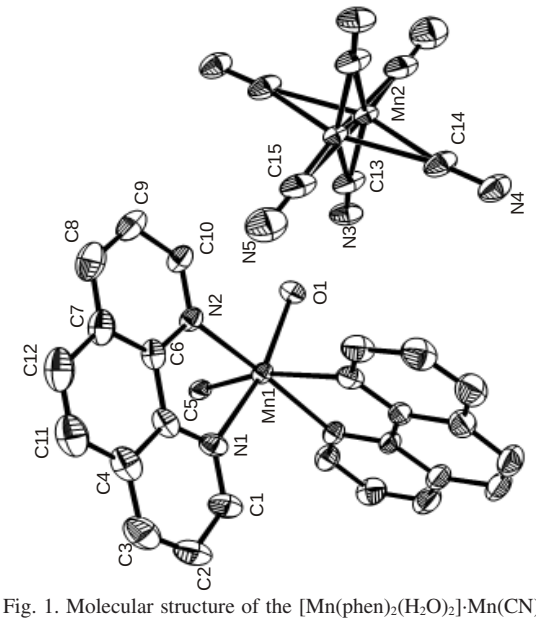

Fig. 1. Molecular structure of the $[Mn(phen)_2(H_2O)_2]\cdot Mn(CN)_3$

TABLE-1
NON-HYDROGEN ATOMIC COORDINATES ($\times 10^4$) AND THERMAL PARAMETERS ($\times 10^3$ Å^2)

Atom	X	Y	Z	U(eq)
MN(1)	5000	6684(1)	7500	49(1)
MN(2)	7727(1)	7367(1)	5122(1)	43(1)
N(1)	4033(2)	7866(3)	7107(4)	66(1)
N(3)	7000(3)	6454(3)	6590(3)	75(1)
N(4)	8505(3)	9005(3)	6460(3)	79(1)
N(5)	6016(4)	8674(5)	4598(5)	115(2)
O(1)	5895(2)	5620(2)	7427(2)	58(1)
C(14)	8137(4)	8435(4)	5923(4)	73(2)

TABLE-2
SELECTED BOND LENGTHS (Å) AND BOND ANGLE (°)

Bond	Length	Angle	(°)	Angle	(°)
MN(1)-N(1)	2.258(4)	N(1)-MN(1)-N(2)	73.93(16)	N(1)#1-MN(1)-N(1)	85.9(2)
MN(1)-N(2)	2.266(4)	C(1)-N(1)-MN(1)	126.5(4)	N(1)#1-MN(1)-N(2)	97.76(15)
MN(1)-O(1)	2.144(3)	O(1)-MN(1)-N(1)	162.94(14)	O(1)#1-MN(1)-O(1)	92.14(15)
MN(2)-C(14)	1.876(6)	O(1)-MN(1)-N(2)	89.31(12)	O(1)#1-Mn(1)-N(1)	93.41(12)
N(1)-C(1)	1.316(6)	Mn(1)-O(1)-H(1C)	111.2	N(2)#1-Mn(1)-N(2)	168.87(19)
O(1)-H(1C)	0.8500	N(3)-C(13)-Mn(2)	169.1(5)	N(5)-C(15)-Mn(2)#2	177.4(8)

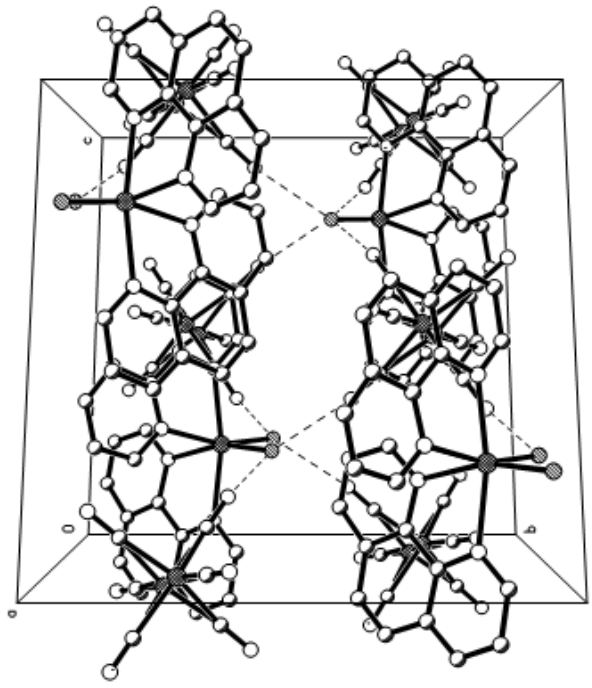

Fig. 2. Molecular packing arrangement in the unit cell

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