Xiu-Ju Yin* and Long-Guan Zhu

The crystal structure of bis(acetato- κ^1O)-bis(*N'*-hydroxypyrimidine-2-carboximidamide- κ^2N,N')manganese(II) – methanol (1/2), $C_{14}H_{18}MnN_8O_6$, $2(CH_3OH)'$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.23 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.61 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ω
$\theta_{\max}$, completeness:	26.7°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5225, 2526, 0.023
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2115
$N(\text{param})_{\text{refined}}$:	154
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Diamond [4]

Source of material

Under stirring, 69 mg (0.5 mmol) pmdH₂ *N'*-hydroxypyrimidine-2-carboximidamide was added to the solution of manganese(II) acetate tetrahydrate (49 mg, 0.2 mmol) in methanol (20 mL), which had to be light yellow to clarify the solution by slightly heating. The final solution was filtered and settled at room temperature to yield yellow crystals. Yield: 71 mg, 58% (based on pmdH₂); elemental analysis calcd: C, 37.43; H, 5.10; N, 21.83; Found: C, 37.32; H, 4.840; N, 22.25. IR(KBr): 3472, 3298, 2727, 2375, 2344, 1572, 1388, 1343, 1213, 1190, 849, 818, 655, 616, 502 cm⁻¹.

Experimental details

Single-crystal X-ray diffraction data for the title structure was collected on Oxford Diffraction Xcalibur CCD diffractometer by 'CrysAlisPro' [1].

The structure was solved with the ShelXT [2] structure solution program and refined with XL [3] refinement package. The positions of the hydrogen atoms were generated geometrically. The figure was drawn by Diamond 4.5.2 [4].

Comment

A limited number of coordination compounds based on *N'*-hydroxypyrimidine-2-carboximidamide have been structurally characterized and reported. So far, to the best of our knowledge, only ten examples of coordination compound containing *N'*-hydroxypyrimidine-2-carboximidamide have been reported [5–9]. The title structure is neutral discrete mononuclear manganese complex. The structure design and

<https://doi.org/10.1515/ncrs-2019-0149>

Received February 28, 2019; accepted April 24, 2019; available online May 27, 2019

Abstract

$C_{16}H_{26}MnN_8O_8$, monoclinic, $C2/c$ (no. 15), $a = 15.3188(9)$ Å, $b = 10.9014(7)$ Å, $c = 14.3601(9)$ Å, $\beta = 90.516(5)^\circ$, $Z = 4$, $V = 2398.0(3)$ Å³, $R_{\text{gt}}(F) = 0.0377$, $wR_{\text{ref}}(F^2) = 0.0942$, $T = 293(2)$ K.

CCDC no.: 1874416

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

*Corresponding author: Xiu-Ju Yin, College of Chemistry and Biology Engineering, Hechi University, Yizhou 546300, P.R. China, e-mail: gxyxj505@163.com

Long-Guan Zhu: Department of Chemistry, Zhejiang University, Hangzhou 310027, P.R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.41882(12)	0.43962(17)	0.42520(13)	0.0283(4)
C1M	0.3231(2)	1.1030(3)	0.3637(2)	0.0867(10)
H1MA	0.307854	1.169533	0.404169	0.130*
H1MB	0.294531	1.113673	0.304454	0.130*
H1MC	0.385164	1.101761	0.355215	0.130*
C2	0.49843(12)	0.36342(18)	0.41205(12)	0.0298(4)
C3	0.58402(16)	0.2079(2)	0.45930(18)	0.0550(6)
H3	0.595622	0.142793	0.499390	0.066*
C4	0.64281(15)	0.2342(2)	0.39139(18)	0.0539(6)
H4	0.693700	0.188643	0.384757	0.065*
C5	0.62388(13)	0.3303(2)	0.33350(16)	0.0432(5)
H5	0.662674	0.349827	0.286317	0.052*
C6	0.37944(15)	0.7852(2)	0.21212(15)	0.0432(5)
C7	0.3737(3)	0.8902(3)	0.1456(2)	0.1025(14)
H7A	0.408615	0.957000	0.168557	0.154*
H7B	0.314039	0.916172	0.139737	0.154*
H7C	0.394785	0.864915	0.085881	0.154*
Mn1	0.500000	0.55998(4)	0.250000	0.03079(15)
N1	0.40692(10)	0.52463(15)	0.36426(11)	0.0331(4)
N2	0.36745(11)	0.41508(17)	0.49715(11)	0.0393(4)
H2A	0.321241	0.458116	0.506145	0.047*
H2B	0.380483	0.356188	0.534621	0.047*
N3	0.51053(12)	0.27171(17)	0.47059(12)	0.0425(4)
N4	0.55106(10)	0.39705(15)	0.34274(11)	0.0325(4)
O1	0.33195(9)	0.59297(15)	0.38361(11)	0.0465(4)
H1	0.332859	0.657168	0.353867	0.070*
O1M	0.29675(15)	0.99355(18)	0.40281(14)	0.0707(6)
H1M	0.312162	0.936412	0.369652	0.106*
O2	0.41679(10)	0.68976(14)	0.18477(10)	0.0434(4)
O3	0.34587(12)	0.79693(15)	0.29061(11)	0.0589(5)

synthesis of new manganese complexes are beneficial to the study of magnetic materials.

The asymmetric unit of the title structure contains one *N'*-hydroxypyrimidine-2-carboximidamide, one acetato ligand and one methanol molecule. The bond lengths and angles within structure are unexceptional and lie within the expected ranges. In details, the C—O distances have values between 1.359(1)–1.434(1) Å. The N1—O2 distance is 1.408(1) Å. The N1—C11 distance is 1.276(1) Å, which suggest N=C double bonds. The intermolecular H-bonds, O—H...N ($dO \cdots N = 2.776(2)$ Å; $dH \cdots N = 1.89$ Å; $\angle OHN = 156.1^\circ$, connect neighboring molecular units. Mn adapts a stable six coordinated model. Two pmadH₂ ligand act as bidentate neutral *N,N'* donors, the last two coordination sites were

occupied by O atoms from two acetato ligands. The second H-bond originates from O1M—H1M...O3 $dO-O = 2.789(1)$ Å, angle OHO = 178(2)°.

Acknowledgements: This work was supported by the project of undergraduate teaching reform of higher education in Guangxi (No. 2018JGA254), the subject of '12th Five-Year' Plan of Guangxi Education Science (No. 2015C408), Guangxi Natural Science Foundation (No. 2018GXNSFAA138140) and the project of education department of Guangxi province (No. YB2014331).

References

1. Oxford Diffraction: CrysAlis^{PRO}, Oxford Diffraction Ltd, Abingdon, Oxfordshire, England (2006).
2. Sheldrick, G. M.: SHELXT – integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with *SHELXL*. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Diamond 4.5.2, Crystal Impact GbR, Bonn, Germany (2018).
5. Golr, B.; Chakrabarty, R.; Mukherjee, S.; Song, Y.; Mukherjee, P. S.: Use of 2-pyrimidineamidoxime to generate polynuclear homo-/heterometallic assemblies: synthesis, crystal structures and magnetic study with theoretical investigations on the exchange mechanism. *Dalton Trans.* **39** (2010) 9766–9778.
6. Dolai, M.; Saha, U.; Kumar, G. S.; Ali, M.: Amidoxime-based mononuclear Mn(II) complexes: synthesis, characterization, and studies on DNA binding and nuclease activity. *ChemistrySelect* **3** (2018) 6935–6941.
7. Kalogridis, C.; Palacios, M. A.; Rodriguez-Dieguez, A.; Mota, A. J.; Choquesillo-Lazarte, D.; Brechin, E. K.; Colacio, E.: Heterometallic oximate-bridged linear trinuclear Ni^{II}, M^{III}, Ni^{III} (M^{III} = Mn, Fe, Tb) complexes constructed with the fac-O3 [Ni(HL)3][−] metalloligand (H2L = pyrimidine-2-carboxamide oxime): a theoretical and experimental magneto-structural study. *Eur. J. Inorg. Chem.* **2011** (2011) 5225–m585.
8. Palacios, M. A.; Mota, A. J.; Perea-Buceta, J. E.; White, F. J.; Brechin, E. K.; Colacio, E.: Antiferromagnetic versus ferromagnetic exchange interactions in bis(μ-O_{oximate})dinickel(II) units for a series of closely elated cube shaped carboxamideoximate-bridged Ni₄ complexes. A combined experimental and theoretical magneto-structural study. *Inorg. Chem.* **49** (2010) 10156–10165.
9. Lopez, M. A. P.; White, F.; Brechin, E.: CSD Communication (Private Communication), CCDC 1411912–1411913.
10. Dolai, M.; Saha, U.; Kumar, G. S.; Ali, M.: Amidoxime-based mononuclear Mn(II) complexes: synthesis, characterization, and studies on dna binding and nuclease activity. *ChemistrySelect* **3** (2018) 6935–6941.