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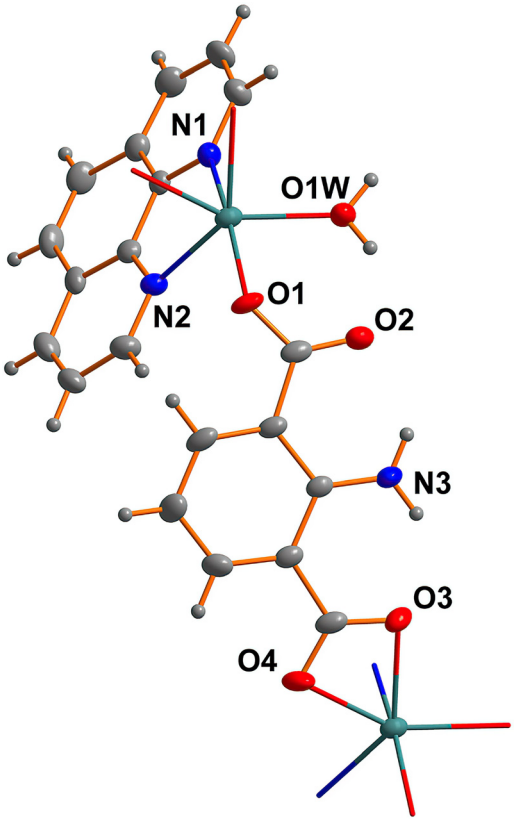
Crystal structure of *catena*-poly[qua-(μ_2-2-aminoisophthalat-κ^3*O,O′:O″*)(1,10-phenanthroline-κ^2*N,N′*)manganese(II)] $C_{20}H_{15}MnN_3O_5$



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Abstract

$C_{20}H_{15}MnN_3O_5$, monoclinic, $P2_1/c$ (no. 14), a = 10.0223(5) Å, b = 9.3406(5) Å, c = 18.6978(10) Å, β = 99.322(6)°, V = 1727.27(16) Å^3, Z = 4, $R_{gt}(F)$ = 0.0376, w$R_{ref}(F^2)$ = 0.0873, T = 250 K.

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A part of the polymeric title structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.81 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	25.7°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7979, 3283, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2674
$N(param)_{refined}$:	265
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3, 4]

Source of material

0.10 mmol $MnCl_2{\cdot}4H_2O$ (0.0198 g), 0.10 mmol 2-aminoisophthalic acid (0.0181 g), 0.10 mmol 1,10-phenanthroline monohydrate (0.0198 g), and 0.20 mmol NaOH (0.008 g) was mixed well under stirring, then transferred to a 25 mL Teflon-lined reactor, heated at 413 K in an oven for 72 h, then cooled naturally. The crystals were washed with deionized water and air-dried, yield 78% based on Mn.

Experimental details

The structure was solved by direct methods with the SHELXS program. All H-atoms from C and N atoms were positioned with idealized geometry and refined isotropically (U_{iso}(H) = 1.2U_{eq}(C) using a riding model with C–H = 0.95 Å and N–H = 0.86 Å. The H-atoms from O atoms positioned

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.5508 (3)	0.1990 (3)	0.50668 (14)	0.0305 (6)
H1	0.609620	0.139784	0.536921	0.037*
C2	0.5576 (3)	0.1986 (3)	0.43244 (14)	0.0331 (6)
H2	0.619747	0.140880	0.414140	0.040*
C3	0.4717 (3)	0.2840 (3)	0.38763 (14)	0.0328 (6)
H3	0.474457	0.285050	0.338159	0.039*
C4	0.3789 (2)	0.3706 (2)	0.41619 (12)	0.0247 (5)
C5	0.2835 (3)	0.4619 (3)	0.37257 (13)	0.0316 (6)
H5	0.280302	0.462893	0.322592	0.038*
C6	0.1984 (3)	0.5460 (3)	0.40232 (13)	0.0315 (6)
H6	0.138875	0.605740	0.372801	0.038*
C7	0.1125 (3)	0.6304 (3)	0.51313 (14)	0.0333 (6)
H7	0.055037	0.696047	0.486341	0.040*
C8	0.1137 (3)	0.6170 (3)	0.58550 (14)	0.0341 (6)
H8	0.057419	0.673652	0.608681	0.041*
C9	0.1998 (3)	0.5179 (3)	0.62471 (13)	0.0298 (6)
H9	0.197231	0.507279	0.673917	0.036*
C10	0.2857 (2)	0.4522 (2)	0.52276 (12)	0.0205 (5)
C11	0.3794 (2)	0.3646 (2)	0.49119 (12)	0.0218 (5)
C12	0.1984 (3)	0.5449 (2)	0.47896 (12)	0.0256 (6)
C13	0.8255 (2)	0.1026 (2)	0.67931 (12)	0.0225 (5)
C14	0.7918 (3)	−0.0064 (3)	0.62948 (13)	0.0287 (6)
H14	0.703141	−0.039440	0.621272	0.034*
C15	0.8842 (3)	−0.0682 (3)	0.59135 (15)	0.0347 (6)
H15	0.858572	−0.140961	0.558025	0.042*
C16	1.0159 (3)	−0.0184 (3)	0.60432 (13)	0.0297 (6)
H16	1.078388	−0.056175	0.577706	0.036*
C17	1.0575 (2)	0.0857 (2)	0.65553 (12)	0.0227 (5)
C18	0.9632 (2)	0.1485 (2)	0.69585 (12)	0.0205 (5)
C19	1.2015 (2)	0.1347 (3)	0.66327 (13)	0.0257 (6)
C20	0.7149 (2)	0.1759 (3)	0.71023 (13)	0.0241 (5)
Mn1	0.42197 (4)	0.26556 (4)	0.65206 (2)	0.02141 (13)
N1	0.2854 (2)	0.4379 (2)	0.59504 (10)	0.0234 (5)
N2	0.4650 (2)	0.2793 (2)	0.53614 (10)	0.0241 (5)
N3	1.0015 (2)	0.2508 (2)	0.74562 (11)	0.0318 (5)
H3A	1.084084	0.279467	0.753634	0.038*
H3B	0.943290	0.287508	0.769420	0.038*
O1	0.59336 (15)	0.13456 (17)	0.68564 (9)	0.0276 (4)
O2	0.74090 (17)	0.27360 (19)	0.75559 (9)	0.0314 (4)
O3	1.24981 (17)	0.22105 (19)	0.71276 (9)	0.0298 (4)
O4	1.27262 (17)	0.09332 (19)	0.61735 (10)	0.0372 (5)
O1W	0.50828 (17)	0.4159 (2)	0.73466 (9)	0.0320 (4)
H1WA	0.588535	0.405971	0.757132	0.048*
H1WB	0.470972	0.472345	0.761097	0.048*

with Q peaks refined isotropically with the distance of O–H = 0.85 Å (U_{iso}(H) = 1.5U_{eq}(O)).

Comment

Mn(II) can coordinate 1,10-phenanthroline with isophthalate or its derivatives to build complexes, including isophthalate [5–9], 4-bromoisophthalate [10], 5-methylisophthalate [11], 5-aminoiodoisophthalate [12], 5-amino-2,4,6-triiodoisophthalate [13], 5-bromoisophthalate [14], 5-iodoisophthalate [15], 5-(pyridin-4-yl)isophthalate [16], 5-phenyl-3,5-dicarboxylate [17], 5-t-butylisophthalate [18], 5-hydroxyisophthalate [19, 20], 5-nitroisophthalate [21, 22], and 5-ferroceneisophthalate [23], etc. To the best of our knowledge, there is no record of 2-substituted isophthalate based Mn(II) complexes with 1,10-phenanthroline. Thus, we reported the title complex.

The asymmetric unit consists of one Mn(II) cation, one 2-amino-isophthalate anion, one 1,10-phenanthroline and one water molecule. The Mn(II) is hexa-coordinated to form an distorted octahedron by two nitrogen atoms from one 1,10-phenanthroline, three oxygen atoms from two 2-aminoisophthalate ligands, and one atom from the coordinating water molecule. The Mn–N distances are 2.265 and 2.281 Å, and the Mn–O distances are in the range of 2.120–2.265 Å, respectively, which agree with similar reported results [5–23]. The Mn(II) cations are linked by the 2-aminoisophthalates to generate 1D structure, further forming a 3D supramolecular structure by hydrogen bonds O1–H1WB···O1 and O1–H1WA···O2 and π···π weak interaction of the 1,10-phenanthroline molecules.

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References

1. CrysAlisPro 1.171.38.43f; Rigaku OD, 2015.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with Shelxl. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. Using phases to determine the space group. *Acta Crystallogr.* 2018, *A74*, a353.
5. Tan X. S., Sun J., Xiang D. F., Tang W. X. Synthesis, crystal structure and magnetic properties of an isophthalate-bridged manganese(II) chain complex. *Inorg. Chim. Acta*. 1997, *255*, 157–161.
6. Devereux M., McCann M., Leon V., Geraghty M., McKee V., Wikaira J. Synthesis and biogical activity of manganese (II) complexes of phthalic and isophtalic acid: X-ray crystal structures of $[Mn(ph)(Phen)_2(H_2O)_2]\cdot 4H_2O$, $[Mn(Phen)_2(H_2O)]_2(Isoph)(Phen)\cdot 12H_2O$ and $\{[Mn(Isoph)(bipy)]_4\cdot 2.75bipy\}$ (phH_2 = phthalic acid; isoph = isophathalic acid; phen

= 1,10-phenanthrolin; bipy = 2,2-bipyridine). *Met. Based Drugs* 2000, *7*, 275–288.
7. Nie J., Liu L., Luo Y., Xu D. Synthesis and structure of a polymeric 1,3-benzenedicarboxylato complex of Mn(II) with phenanthorine. *J. Coord. Chem.* 2001, *53*, 365–371.
8. Bi W.-H., Sun D.-F., Cao R., Li X., Shi Q. Hydrothermal synthesis and crystal structure of $[Mn(phen)-(m-phth)]_n$ (phen=1,10-phenanthroline, m-phth=isophthalate). *Chin. J. Chem.* 2004, *22*, 271–274.
9. Liu D.-W., Zhang L., Niu S.-Y., Li L., Shi Z.-F., Jin J., Chi Y.-X. Synthesis, crystal structure and photo-physical property of manganese(II) coordination polymers. *Chin. J. Inorg. Chem*. 2009, *25*, 2175–2181.
10. Xiao H.-P., Hu M.-L., Yuan J.-X. Bis(μ-4-bromo-isophthalato)-bis-[bis(1,10-phenanthroline)-manganese(II)] tetrahydrate. *Acta Crystallogr*. 2005, *E61*, m443–m445.
11. Yu Z.-L., Hu J.-L. *catena*–Poly[[(1,10-phenanthroline) manganese(II)]-μ_3-5-methyl-isophthalato]. *Acta Crystallogr*. 2008, *E64*, m1522.
12. Wang J.-J., Bao Q.-L., Chen J.-X. Two 2-D layered coordination polymers based on 5-aminoisophthalate and 1,10-phenanthroline. *J. Coord. Chem.* 2013, *66*, 2578–2586.
13. Zhang K.-L., Chang Y., Zhang J.-B., Yuan L.-M., Deng Y., Diao G.-W., Ng S. W. Synthesis, crystal structures and characterization of four coordination polymers based on 5-amino-2,4,6-triiodoisophthalic acid. *J. Solid State Chem.* 2011, *184*, 1263–1272.
14. Wang C.-C., Gao F., Guo X,-X., Jing H.-P., Wang P., Gao S.-J. Hydrothermal syntheses and photocatalytic performance of three Mn-based coordination complexes constructed from 1,10-phenanthroline and polycarboxylic acids. *Trans. Met. Chem.* 2016, *41*, 375–385.
15. Zang S.-Q., Fan Y.-J., Li J.-B., Hou H.-W., Mak T. C. W. Halogen bonding in the assembly of coordination polymers based on 5-iodo-isophthalic acid. *Cryst. Growth Des.* 2011, *11*, 3395–3405.
16. Gao Z.-Q., Li H.-J., Kirillov A. M., Gu J.-Z. Syntheses, crystal structures and magnetic properties of cobalt(II) and manganese(II) coordination polymers constructed from 5-(pyridin-4-yl)isophthalate ligands. *Chin. J. Struct. Chem*. 2014, *33*, 626–634.
17. Jiang J., Huang W., Xu J., Pan F.-F., Wu D.-Y. Influence of steric hindrance of N-heterocyclic ancillary ligands on the structur and magnetic properties of manganese(II) coordination polymers. *Z. Anorg. Allg. Chem.* 2016, *642*, 197–202.
18. Jin R.-F., Yang S.-Y., Li H.-M., Long L.-S., Huang R.-B., Zheng L.-S. Effect of ionic radius on the assemblies of first row transition metal-5-*tert*-butylisophthalates-(2,2′-bipyridine or phenanthroline) coordination compounds. *CrystEngComm* 2012, *14*, 1301–1316.
19. Skakle J. M. S., Foreman M. R. S. J., Plater M. J. *catena*–Poly-[[[(1,10-phenanthroline)-manganese(II)]-μ-(1-hydroxybenzene-3,5-dicarboxylato)]hydrate]. *Acta Crystallogr*. 2001, *E57*, m373–m375.
20. Kariem M., Yawer M., Sheikh H. N. Solvent induced synthesis, structure and properties of coordination polymers based on 5-hydroxyisophthalic acid as linker and 1,10-phenanthroline as auxiliary ligand. *J. Solid State Chem.* 2015, *231*, 239–247.
21. Wang H.-D., Li M.-M., He H.-Y., Jiang F.-B. Poly[bis-(μ-3-5-nitroisophthalato)bis(1,10-phenanthroline) dimanganese(II)]. *Acta Crystallogr*. 2009, *E65*, m416.
22. Li X. M., Wang Q.-W., Liu B., Wang Z.-T. Hydrothermal synthesis and crystal structure of a Mn(II) coordination polymer assembled by 5-nitroisophthalic acid and 1,10-phenanthroline. *Chin. J. Inorg. Chem.* 2011, *27*, 1207–1211.
23. Li X., Wu B.-L., Liu W., Niu C.-Y., Niu Y.-Y., Zhang H.-Y. Syntheses, crystal structures, and magnetic properties of five new coordination compounds bearing ferrocenedicarboxylate ligands. *J. Coord. Chem.* 2009, *62*, 3142–3156.