Jingjing Zhou, Wei Xu, Luxiang He and Hongping Xiao*

Crystal structure of tetraaqua-bis(1,4-di(1H-imidazol-1-yl)benzene- κ^1N)manganese(II) 2,3-dihydroxyterephthalate, $C_{32}H_{32}MnN_8O_{10}$

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Abstract

$C_{32}H_{32}MnN_8O_{10}$, monoclinic, $P2/c$ (no. 13), $a = 14.258(3)$ Å, $b = 7.1056(13)$ Å, $c = 17.589(3)$ Å, $\beta = 113.672(3)^\circ$, $V = 1632.0(5)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0314$, $wR_{ref}(F^2) = 0.1019$, $T = 296(2)$ K.

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

Source of material

An aqueous solution (10 ml) containing $Mn(CH_3COO)_2 \cdot 4H_2O$ (0.0262 g) was added slowly to a solution of N,N -dimethylformamide containing 1,4-bis(1-imidazolyl)benzene (0.0220 g)

*Corresponding author: Hongping Xiao, College of Chemistry and Materials Engineering, Wenzhou University, Wenzhou, Zhejiang, 325035, P. R. China, E-mail: hp_xiao@126.com. <https://orcid.org/0000-0001-7242-7976>

Jingjing Zhou, Wei Xu and Luxiang He, College of Chemistry and Materials Engineering, Wenzhou University, Wenzhou, Zhejiang, 325035, P. R. China

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	$0.33 \times 0.28 \times 0.25$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.48 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	26.0° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12767, 3178, 0.026
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2767
$N(\text{param})_{\text{refined}}$:	232
Programs:	Bruker [1], Diamond [2], SHELX [3, 4]

and 2,3-dihydroxy-terephthalic acid (0.0207 g). Light yellow block crystals were obtained after six months (yield ca. 31% based on 2,3-dihydroxy-terephthalic acid ligand). The product is insoluble in water and common organic solvents.

Experimental details

The H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of $C_{\text{sp}^2}-H = 0.93$ Å with $U_{\text{iso}}(H) = 1.2 U_{\text{eq}}(C)$, and $O-H = 0.85$ Å with $U_{\text{iso}}(H) = 1.5 U_{\text{eq}}(O)$, respectively.

Comment

The construction of coordination complexes are attracting interest due to their appealing applications as functional

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Mn1	0.500000	0.80703 (5)	0.250000	0.02913 (13)
O1	0.25705 (10)	0.4818 (2)	0.28085 (9)	0.0556 (4)
O2	0.26093 (10)	0.1697 (2)	0.29412 (11)	0.0569 (4)
O3	0.10219 (10)	−0.01111 (16)	0.27233 (8)	0.0371 (3)
H3	0.163169	0.006497	0.283631	0.056*
O4	0.44705 (9)	0.5885 (2)	0.31100 (10)	0.0540 (4)
H4A	0.490013	0.499245	0.329249	0.081*
H4B	0.386872	0.546856	0.298975	0.081*
O5	0.55447 (9)	1.0349 (2)	0.19283 (9)	0.0524 (4)
H5A	0.511382	1.122076	0.170305	0.079*
H5B	0.614360	1.073014	0.201304	0.079*
N1	0.35172 (10)	0.8013 (2)	0.13610 (9)	0.0335 (3)
N2	0.18624 (10)	0.78609 (18)	0.05468 (8)	0.0273 (3)
N3	−0.23562 (10)	0.70574 (19)	−0.02513 (9)	0.0301 (3)
N4	−0.40392 (12)	0.6923 (2)	−0.09534 (11)	0.0452 (4)
C1	0.25865 (12)	0.7785 (2)	0.13352 (11)	0.0322 (4)
H1	0.244390	0.759501	0.180107	0.039*
C2	0.23779 (13)	0.8150 (3)	0.00393 (11)	0.0347 (4)
H2	0.209242	0.825673	−0.053615	0.042*
C3	0.33840 (14)	0.8247 (3)	0.05500 (12)	0.0378 (4)
H3A	0.391116	0.844469	0.037505	0.045*
C4	0.07810 (11)	0.7709 (2)	0.03145 (9)	0.0254 (3)
C5	0.03873 (12)	0.8197 (2)	0.08855 (10)	0.0297 (4)
H5	0.081974	0.866924	0.140062	0.036*
C6	−0.06495 (13)	0.7990 (2)	0.06981 (10)	0.0306 (4)
H6	−0.091088	0.831185	0.108735	0.037*
C7	−0.12950 (12)	0.7299 (2)	−0.00737 (10)	0.0261 (3)
C8	−0.09007 (12)	0.6835 (2)	−0.06540 (10)	0.0279 (3)
H8	−0.133414	0.638327	−0.117281	0.033*
C9	0.01368 (12)	0.7043 (2)	−0.04637 (10)	0.0273 (3)
H9	0.039938	0.673780	−0.085406	0.033*
C10	−0.31492 (13)	0.7098 (3)	−0.10102 (12)	0.0374 (4)
H10	−0.307295	0.723271	−0.150830	0.045*
C11	−0.38077 (15)	0.6757 (3)	−0.01205 (14)	0.0452 (5)
H11	−0.428839	0.660772	0.010855	0.054*
C12	−0.27869 (15)	0.6839 (3)	0.03202 (13)	0.0408 (4)
H12	−0.244184	0.676342	0.089401	0.049*
C13	0.21673 (13)	0.3290 (3)	0.28298 (10)	0.0357 (4)
C14	0.10492 (11)	0.3271 (2)	0.26810 (9)	0.0243 (3)
C15	0.05327 (11)	0.1567 (2)	0.26047 (9)	0.0237 (3)
C16	0.05149 (12)	0.4967 (2)	0.25813 (10)	0.0282 (3)
H16	0.085687	0.610258	0.262513	0.034*

materials [5–7]. In order to obtain the final structures, it is necessary to select suitable organic ligands [8, 9]. For example, in our previous work, a series of coordination complexes have been obtained by terephthalic acid and its derivative ligands [10, 11]. Herein, we reported a mononuclear, cationic manganese complex.

In the title complex, each Mn(II) is six-coordinated, with two N atoms from two 1,4-bis(1-imidazolyl)benzene

ligands, and four coordinated aqua O atoms, respectively. The cationic complex is located on a twofold axis (see the Figure). The Mn1–N1, Mn1–O4 and Mn1–O5 bond lengths are 2.2548(14), 2.1870(13) and 2.2053(13) Å. The Mn–N and Mn–O bond lengths are found within normal ranges of bond distances in the reported Mn(II) complexes [8, 9]. The O4–Mn1–O5 and N1–Mn1–N1ⁱ (symmetry code: $-x + 1, y, -z + 1/2$) bond angles are 177.86(6)° and 177.92(7)°, other angles O–Mn–O and O–Mn–N are in the range 85.52(8)–92.49(6)°, forming a light distorted octahedral geometry. Each 2,3-dihydroxy-terephthalate dianion acts as a charge balancing ion. Also this ion is located around a twofold axis (see the Figure).

The relatively short interplanar distances of 3.668(2) and 3.701(2) Å between the benzene ring of neighbouring 1,4-bis(1-imidazolyl)benzene ligands indicate possible weak $\pi \cdots \pi$ stacking interactions. The stability of the solid-state structure of the title complex is enhanced significantly by hydrogen bonding interactions. An intramolecular hydrogen bond exists for the hydroxyl atom O3 and the uncoordinated carboxylate atom O2. Intermolecular O–H $\cdots$ N, O–H $\cdots$ O and C–H $\cdots$ O hydrogen bonds link neighbouring anions and mononuclear coordination cations into a three-dimensional network.

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