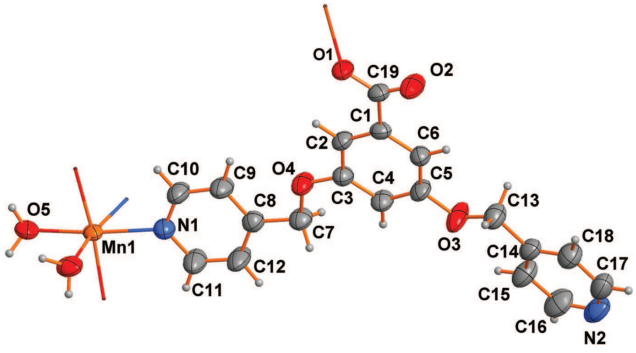
Gui-Hua Zou, Yu-Long Zou and Guang-Zhi Shen*

Crystal structure of *catena*-poly[diaqua-(μ_2-3,5-bis(pyridin-4-ylmethoxy) benzoate-κ^2N:O) manganese(II)] tetrahydrate [(3,5-bis-(pyridin-4-ylmethoxy)-benzoic-κ^1*O*κ^1*N*) manganese(II)] trihydrate, $C_{38}H_{42}MnN_4O_{14}$



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Abstract

$C_{38}H_{42}MnN_4O_{14}$, monoclinic, $C2/c$ (no. 15), $a = 19.165(12)$ Å, $b = 10.116(6)$ Å, $c = 20.734(13)$ Å, $\beta = 99.021(11)°$, $V = 3970(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0486$, $wR_{ref}(F^2) = 0.1442$, $T = 293(2)$ K.

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A part of the title crystal structure is shown in the figure. Uncoordinated water molecules are omitted for clarity. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title complex was synthesized solvothermally in a Teflon-lined stainless steel container by heating a mixture of 3,5-bis(pyridin-4-ylmethoxy)benzoic acid (HL; 0.010 g, 0.03 mmol), $Mn(NO_3)_2$ (0.010 g, 0.06 mmol), and 6 mL of distilled water at 130 °C for 3 days under autogenous pressure. Subsequent cooling to room temperature yielded colorless block crystals.

***Corresponding author: Guang-Zhi Shen,** College of Pharmacy, Mudanjiang Medical University, Mudanjiang, 157011, China, e-mail: pharm361@163.com
Gui-Hua Zou: College of Pharmacy, Mudanjiang Medical University, Mudanjiang, 157011, China
Yu-Long Zou: Mudanjiang Medical University Affiliated Hongqi Hospital, Mudanjiang, 157011, China

Table 1: Data collection and handling.

Crystal:	colorless block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.41 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9384, 3482, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2526
$N(param)_{refined}$:	258
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.000000	1.30976(5)	0.250000	0.0411(2)
N1	0.05752(11)	1.1418(2)	0.20742(12)	0.0486(6)
N2	0.48982(14)	0.1499(3)	0.13329(17)	0.0703(8)
O1	0.07693(10)	0.67927(18)	−0.16487(9)	0.0510(5)
O2	0.15575(11)	0.5292(2)	−0.18336(10)	0.0675(6)
O3	0.29231(12)	0.4464(2)	0.04587(10)	0.0743(7)
O4	0.13381(10)	0.80026(19)	0.06455(9)	0.0545(5)
O5	−0.06622(10)	1.4533(2)	0.29504(10)	0.0600(6)
H5A	−0.099134	1.477855	0.267509	0.090*
H5B	−0.043824	1.524315	0.309879	0.090*
O6	0.22150(19)	0.3070(4)	0.7754(2)	0.1541(16)
H6A	0.245765	0.238495	0.787466	0.231*
H6B	0.180265	0.298446	0.785145	0.231*
O7	0.8295(3)	0.3283(4)	0.3665(2)	0.1718(19)
H7C	0.837161	0.389280	0.394895	0.258*
H7D	0.864181	0.323220	0.345575	0.258*
C1	0.15904(12)	0.6076(2)	−0.07567(13)	0.0403(6)
C2	0.13348(13)	0.6975(3)	−0.03549(13)	0.0436(6)
H2	0.097147	0.754524	−0.052492	0.052*
C3	0.16160(13)	0.7035(3)	0.03029(14)	0.0430(6)
C4	0.21431(14)	0.6176(3)	0.05587(14)	0.0490(7)
H4	0.232674	0.619897	0.100117	0.059*
C5	0.23978(14)	0.5273(3)	0.01478(14)	0.0483(7)
C6	0.21329(13)	0.5212(3)	−0.05036(14)	0.0442(6)
H6	0.231091	0.460540	−0.077311	0.053*
C7	0.15883(15)	0.8114(3)	0.13237(14)	0.0502(7)
H7A	0.209538	0.825284	0.139795	0.060*
H7B	0.148712	0.730945	0.154633	0.060*
C8	0.12243(13)	0.9259(3)	0.15783(14)	0.0454(7)
C9	0.06293(15)	0.9831(3)	0.12394(15)	0.0596(8)
H9	0.042932	0.950155	0.083349	0.072*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C10	0.03300(16)	1.0898(3)	0.15060(16)	0.0609(8)
H10	−0.007246	1.127222	0.126646	0.073*
C11	0.11511(18)	1.0853(3)	0.24000(17)	0.0734(10)
H11	0.133935	1.119814	0.280581	0.088*
C12	0.14830(18)	0.9791(4)	0.21715(17)	0.0736(10)
H12	0.188414	0.943302	0.242107	0.088*
C13	0.33187(16)	0.3709(3)	0.00755(15)	0.0600(8)
H13A	0.354406	0.428378	−0.020467	0.072*
H13B	0.301132	0.310225	−0.019830	0.072*
C14	0.38720(14)	0.2946(3)	0.05280(16)	0.0507(7)
C15	0.39267(16)	0.3007(3)	0.11860(18)	0.0658(9)
H15	0.362194	0.353860	0.137933	0.079*
C16	0.44384(19)	0.2272(4)	0.15650(19)	0.0755(10)
H16	0.446316	0.232107	0.201596	0.091*
C17	0.48430(17)	0.1450(3)	0.0700(2)	0.0724(10)
H17	0.515914	0.091647	0.052179	0.087*
C18	0.43406(17)	0.2146(3)	0.02699(18)	0.0660(9)
H18	0.432258	0.207224	−0.017953	0.079*
C19	0.12844(13)	0.6046(3)	−0.14677(14)	0.0439(7)

Experimental details

Absorption corrections were applied by using multi-scan program. Hydrogen atoms attached to C of the title complex located in difference electron density maps, and treated as riding atoms. The U_{iso} values of the hydrogen atoms of water molecules were set to $1.5U_{eq}$(O) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

Comment

The design and construction of metal-organic coordination polymers have attracted great attention owing to their properties [4–6]. Although many metal-organic coordination polymers based on multicarboxylic acids have been reported [7–10], those on pyridine carboxylate acids are relatively rare. With the aim to construct intruguing coordination architectures, we have engaged in the research of coordination polymer based on HL.

Single crystal structure analysis reveals that the asymmetric unit of the title structure consists of one Mn(II) atom, one L^- ligand, one coordinated water molecule and two free water molecules. The central Mn1 cation coordinated to two nitrogen atoms from two L^- ligand, two oxygen atoms from two different L^- ligands and two oxygen atoms from two coordinated water molecules. L^- ligands link adjacent metal centers to generate infinite one-dimensional chain, which further connected through weak π–π (3.667(2) Å) interactions to form a three-dimensional supramolecular network. The hydrogen bonding further consolidates the structure.

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