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The crystal structure of diaqua-bis(pyrazolo[1,5-*a*]pyrimidine-3-carboxylato-κ^2*N*,*O*)manganese(II), $C_{14}H_{12}N_6O_6Mn$

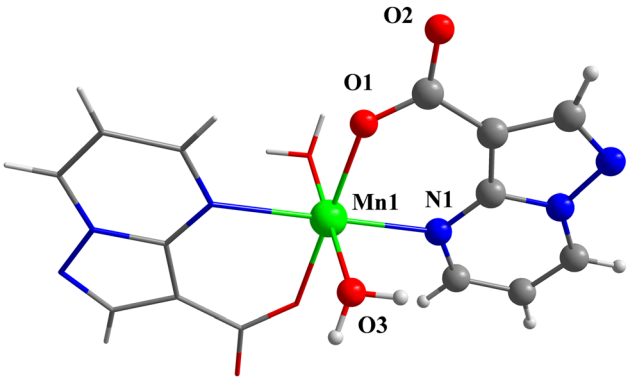

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.35 × 0.33 × 0.31 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.91 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8374, 1352, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1269
$N(param)_{refined}$:	125
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], Olex2 [4]

https://doi.org/10.1515/ncrs-2022-0143
Received March 26, 2022; accepted April 25, 2022;
published online May 5, 2022

Abstract

$C_{14}H_{12}N_6O_6Mn$, monoclinic, $P2_1/n$ (no. 14), a = 7.0839(3) Å, b = 13.1211(4) Å, c = 8.9854(3) Å, β = 112.359(5)°, V = 772.39(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0274, $wR_{ref}(F^2)$ = 0.0725, T = 293 K.

CCDC no.: 2162137

The molecular 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.

Source of material

All chemicals were purchased and used without further purification. The title compound was prepared under the mild hydrothermal condition by the following procedure: a mixture of pyrazolo[1,5-*a*]pyrimidine-3-carboxylic acid (16.3 mg, 0.1 mmol), $Mn(CH_3COO)_2{\cdot}4H_2O$ (24.5 mg, 0.1 mmol) and 5 mL deionized water was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 353 K for three days under autogenous pressure. The reaction was then stopped and cooled to room temperature and filtered. Yellow block crystal were obtained (yield: 67% based on Mn).

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

The pyrazolo[1,5-*a*]pyrimidine and its derivatives contains both a pyrazole and a pyrimidine rings. They are found as building blocks of different pharmaceutically important heterocyclic compounds and they possess a wide range of biological activities [5–9]. In addition, the studying on the structure and properties of related transition metal complexes has been one of the scientific hotspots [10–12]. The rigid pyrazolo[1,5-*a*] pyrimidine-3-carboxylic acid and the corresponding carboxylate can provide both nitrogen atoms and oxygen atoms for the coordination with metal atoms, which makes it a useful ligand.

The title compound, $C_{14}H_{12}N_6O_6Mn$, was synthesized using $Mn(CH_3COO)_2{\cdot}4H_2O$ and pyrazolo[1,5-*a*]pyrimidine-3-carboxylic acid under mild hydrothermal condition.

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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}
Mn1	0.5000	0.5000	0.5000	0.02565 (16)
O1	0.4616 (2)	0.37980 (10)	0.33418 (16)	0.0325 (4)
O2	0.4203 (2)	0.22690 (10)	0.22270 (16)	0.0335 (4)
O3	0.8271 (2)	0.46083 (11)	0.61995 (17)	0.0369 (4)
H3A	0.8394	0.4055	0.6730	0.055*
H3B	0.8869	0.5055	0.6915	0.055*
N1	0.4213 (3)	0.38816 (12)	0.65940 (19)	0.0267 (4)
N2	0.4138 (3)	0.11797 (13)	0.6647 (2)	0.0345 (4)
N3	0.4122 (3)	0.21312 (12)	0.72489 (19)	0.0261 (4)
C1	0.4350 (3)	0.28452 (14)	0.3363 (2)	0.0232 (4)
C2	0.4239 (3)	0.23920 (14)	0.4841 (2)	0.0237 (4)
C3	0.4202 (3)	0.13603 (15)	0.5207 (2)	0.0314 (5)
H3	0.4221	0.0843	0.4506	0.038*
C4	0.4193 (3)	0.28832 (14)	0.6196 (2)	0.0224 (4)
C5	0.4053 (3)	0.23527 (16)	0.8703 (2)	0.0313 (5)
H5	0.4020	0.1839	0.9406	0.038*
C6	0.4034 (3)	0.33438 (17)	0.9093 (2)	0.0337 (5)
H6	0.3966	0.3530	1.0070	0.040*
C7	0.4119 (3)	0.40920 (17)	0.8001 (2)	0.0334 (5)
H7	0.4108	0.4773	0.8286	0.040*

The asymmetric unit of the title crystal structure consists of one half of a Mn^{2+} ion, one fully deprotonated pyrazolo[1,5-*a*]pyrimidine-3-carboxylate anion, one coordinated water molecules. The Mn(II) ion is six-coordinated with two carboxylate O atoms (Mn1–O2 = 2.1150 (13) Å), two pyridine N atoms (Mn1–N1 = 2.2635 (16) Å) from two pyrazolo[1,5-*a*]pyrimidine-3-carboxylate ligands, and two O atoms (Mn1–O3 = 2.2121 (15) Å) from two coordinated water molecules, forming a distorted octahedral geometry. The Mn–O/N distances are within the normal ranges [13, 14]. The complex molecules are connected to each other through O–H···N hydrogen bonds between the coordinated water molecules and the pyrazolo[1,5-*a*]pyrimidine-3-carboxylate ligands to form a layered structure.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the grants from Luoyang Normal University (DT21000009147).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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