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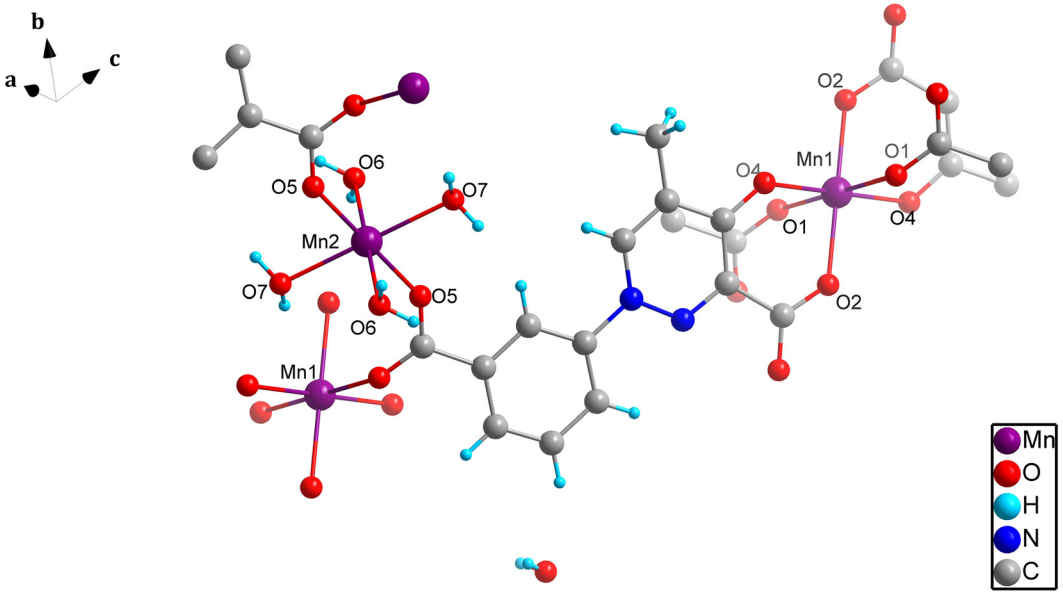
Crystal structure of poly[diaqua-{μ_3-1-(3-carboxylatophenyl)-4-oxo-1,4-dihydropyridazine-3-carboxylato-κ^4O,O′:O″:O‴}manganese(II)] hydrate



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Abstract

$C_{13}H_{14}MnN_2O_8$, triclinic, $P\bar{1}$ (no. 2), a = 6.901(4) Å, b = 8.854(6) Å, c = 12.400(8) Å, α = 72.955(12)°, β = 75.712(11)°, γ = 80.782(11)°, V = 698.7(8) Å^3, Z = 2, $R_{gt}(F)$ = 0.0377, w$R_{ref}(F^2)$ = 0.0763, T = 296 K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data. The list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

Table 1: Data collection and handling.

Crystal:	Clear light colourless block
Size:	0.15 × 0.12 × 0.11 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.00 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3515, 2441, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1829
$N(param)_{refined}$:	226
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

1 Source of material

The mixture of manganese nitrate hexahydrate 28.7 mg (0.1 mmol), 1-(3-carboxyphenyl)-4-oxo-1,4-dihydropyridazine-3-carboxylic acid 27.4 mg (0.1 mmol), NaOH 4 mg (0.1 mmol)

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and ethyl alcohol (10 mL) were placed in the autoclave lined with PTFE and heated at 110 °C for 48 h, then cooled up to room temperature over 24 h. Viridescent block crystals were collected after cooling to room temperature.

2 Experimental details

Using Olex2,[2] the structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL[4] refinement package.

3 Comment

Pyridazine derivatives constitute a particularly significant class of biologically active heterocycles. These compounds have garnered considerable attention as ligands due to their remarkable structural and synthetic versatility, precise tunability, and selectivity towards transition metal atoms.[5,6] Their diverse therapeutic potential, encompassing anti-cancer,[7,8] antidiabetic,[9,10] and antibiotic activities,[11] has been extensively documented. Leveraging this understanding, we have achieved the successful synthesis of a novel pyridazine carboxyl derivative, achieved by incorporating a benzene ring adorned with carboxyl groups onto the pyridazine ring. Simultaneously, numerous syntheses of transition metal complexes utilizing this derivative have been documented.[12–15]

The single-crystal X-ray diffraction analysis reveals that the title complex belongs to the triclinic system, $P\bar{1}$ space group. The asymmetric unit of it comprises two half Mn^{2+}, one (4-PDCA)2-ligand, two coordinated aqua ligands, and one crystalline water molecule, as depicted in the figure. The Mn1 ion was coordinated to six oxygen atoms from four different ligands, including four chelating symmetric oxygen atoms and two monodentate carboxylic oxygen atoms. Mn2 ion was coordinated to two monodentate carboxylic oxygen atoms from two different ligands and four aqua ligands. Two coordination modes of Mn(II) atoms through bridging ligand links form a two-dimensional double layer framework.

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

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

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methyl-4-oxo-1,4-dihydropyridazine-3-carboxylato-κ^2O,O′)-bis(μ2-1-(3-carboxyphenyl-5-methyl-4-oxo-1,4-dihydropyridazine-3-carboxylato-κ^2O:O′)trizinc(II)] Hexahydrate $C_{26}H_{36}N_4O_{20}Zn_2$. *Z. Kristallogr. N. Cryst. Struct.* **2024**, *239*, 159–161.

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