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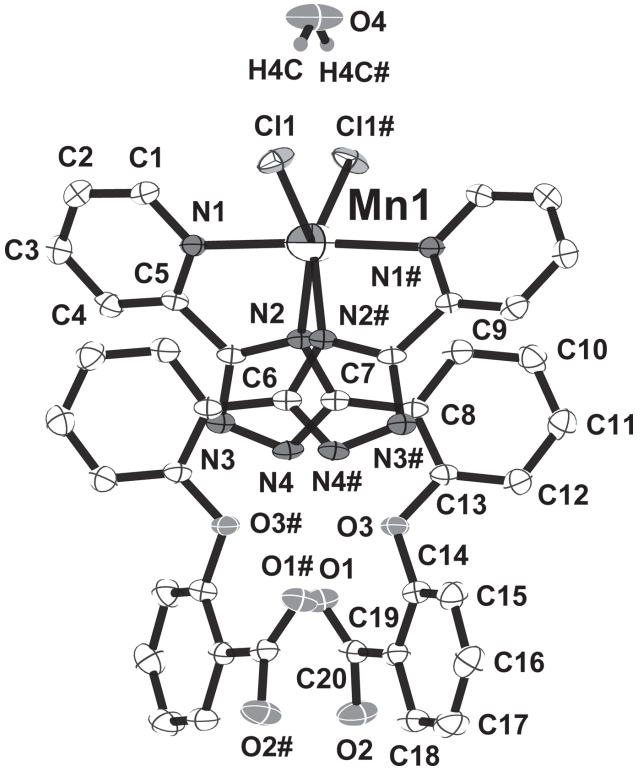
Crystal structure of dichlorido-bis[2-(2-(3-(pyridin-2-yl)-1*H*-1,2,4-triazol-5-yl)phenoxy)benzoic acidmanganese(II) monohydrate, $C_{40}H_{30}N_8O_7MnCl_2$



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Abstract

$C_{40}H_{30}N_8O_7MnCl_2$, monoclinic, $C2/c$ (no. 15), $a = 13.0864(7)$ Å, $b = 25.6216(9)$ Å, $c = 12.2253(7)$ Å, $\beta = 112.027(6)°$, $V = 3799.9(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0413$, $wR_{ref}(F^2) = 0.1001$, $T = 293(2)$ K.

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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.

Table 1: Data collection and handling.

Crystal:	White block
Size:	0.23 × 0.22 × 0.21 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	4.65 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω scan
θ_{max}, completeness:	67.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7181, 3407, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2631
$N(param)_{refined}$:	264
Programs:	SHELX [1], CrysAlis^{PRO} [2]

Source of material

The educt 2-(2-(3-(pyridin-2-yl)-1*H*-1,2,4-triazol-5-yl)phenoxy) benzoic acid (*PTPBA*) was purchased from Jinan Camolai Trading Company (China), and other reagents were commercially available and used directly without further purification. A mixture of $MnCl_2 \cdot 4H_2O$ (19.8 mg, 0.1 mmol) and *PTPBA* (35.8 mg, 0.1 mmol) was dissolved in 20 mL of methanol, and then stirred at ambient temperature for 30 min. The resulting solution was filtrated and shelved in a vibrationless environment for two days. Thus, colourless block crystals were obtained in 52% yield (based on Mn).

Experimental details

The structure was solved by direct methods and refined by the full-matrix least-squares methods on F^2 using the SHELX software [1]. All of the hydrogen atoms were placed in the calculated positions.

Comment

In recent years, transition metal complexes with *N*-heterocyclic carboxylic acid ligands have been generally

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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	0.500000	0.09859(2)	0.750000	0.03430(17)
Cl1	0.40418(6)	0.03924(3)	0.58532(7)	0.0580(2)
N1	0.34923(19)	0.10135(8)	0.8085(2)	0.0377(5)
N2	0.39165(17)	0.16840(7)	0.65912(18)	0.0323(5)
N3	0.27895(18)	0.22931(8)	0.68614(19)	0.0384(5)
N4	0.33926(17)	0.24893(8)	0.62635(19)	0.0384(5)
H4A	0.335513	0.280700	0.602455	0.046*
O1	0.32603(17)	0.35713(7)	0.55292(19)	0.0539(6)
O2	0.33900(19)	0.43873(8)	0.4993(3)	0.0716(7)
H2	0.272488	0.438576	0.484496	0.107*
O3	0.51528(17)	0.30339(7)	0.6139(2)	0.0515(5)
O4	0.000000	0.56446(13)	0.250000	0.1161(18)
H4C[a]	0.034050	0.544900	0.308520	0.139*
H4D[a]	−0.034050	0.544900	0.191480	0.139*
C1	0.3227(3)	0.06364(11)	0.8693(3)	0.0476(7)
H1	0.370793	0.035693	0.897353	0.057*
C2	0.2277(3)	0.06450(12)	0.8918(3)	0.0575(9)
H2A	0.212427	0.037689	0.934723	0.069*
C3	0.1554(3)	0.10517(12)	0.8507(3)	0.0594(9)
H3	0.090546	0.106367	0.865203	0.071*
C4	0.1806(3)	0.14463(11)	0.7869(3)	0.0498(8)
H4	0.132956	0.172606	0.757301	0.060*
C5	0.2781(2)	0.14123(10)	0.7687(2)	0.0344(6)
C6	0.3141(2)	0.18061(9)	0.7035(2)	0.0330(6)
C7	0.4053(2)	0.21261(9)	0.6093(2)	0.0335(6)
C8	0.4759(2)	0.21875(10)	0.5417(2)	0.0353(6)
C9	0.4878(2)	0.17701(11)	0.4746(2)	0.0465(7)
H9	0.451905	0.145676	0.474439	0.056*
C10	0.5526(3)	0.18173(13)	0.4081(3)	0.0569(9)
H10	0.561230	0.153611	0.364204	0.068*
C11	0.6045(3)	0.22875(13)	0.4075(3)	0.0575(8)
H11	0.646600	0.232398	0.361283	0.069*
C12	0.5944(3)	0.27061(12)	0.4752(3)	0.0508(8)
H12	0.630511	0.301884	0.475255	0.061*
C13	0.5307(2)	0.26546(10)	0.5423(2)	0.0383(6)
C14	0.5689(2)	0.35088(10)	0.6292(3)	0.0423(7)
C15	0.6822(3)	0.35301(13)	0.6818(3)	0.0557(8)
H15	0.723212	0.322422	0.702677	0.067*
C16	0.7341(3)	0.40088(13)	0.7033(3)	0.0605(9)
H16	0.810558	0.402621	0.738729	0.073*
C17	0.6731(3)	0.44615(13)	0.6723(3)	0.0619(9)
H17	0.708467	0.478349	0.685316	0.074*
C18	0.5602(3)	0.44375(11)	0.6223(3)	0.0535(8)
H18	0.519768	0.474544	0.603013	0.064*
C19	0.5049(2)	0.39592(10)	0.5997(2)	0.0401(6)
C20	0.3823(3)	0.39409(11)	0.5494(3)	0.0440(7)

[a]Occupancy: 0.5.

investigated in the field of crystal engineering and material science, owning to not only by its intriguing topological structures [3], but also their widely applications in various fields, such as catalysis [4], magnetism [5], luminescence [6], gas selective adsorption [7] and so on. As a typical *N*-heterocyclic carboxylic acid ligand, *PTPBA* contains both heterocyclic nitrogen atoms and the carboxylic group and can provide versatile coordination modes. However, to the best of our knowledge, except for copper *PTPBA* ligands has been reported by our group [8], no other crystal structure containing *PTPBA* have been reported so far.

As is shown in the figure, the asymmetric unit of the title structure contains one half of a Mn(II), one *PTPBA* ligand, one coordinated chloridion and one half of a water molecule. The central Mn(II) is hexa-coordinated by two chlorido ligands (Cl1 and Cl1#) and four nitrogen atoms (N1, N1#, N2 and N2#) derived from *PTPBA* ligands, resulting in a slightly distorted octahedral (MnN_4Cl_2) coordination geometry. The bond angles around Mn(II) vary from 71.67(7)° to 176.54(10)°. The Mn-N1(N1#), Mn-N2(N2#), Mn-Cl1(Cl1#) bond distances are 2.339(2) Å, 2.294(2) Å and 2.4585(8) Å, respectively, which are comparable to those reported for the related [MnN_4Cl_2] geometry [9]. Besides, compared with our previously reported [CuN_3O_3] coordination geometry in [$(PTPBA)_2Cu_2 \cdot CH_3OH$] [8], the carboxyl group in this structure did not involve in coordination, which illustrated that the present work has been further enriched the coordination modes of *PTPBA*.

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