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Crystal structure of aquachloro[*N,N'*-bis(3-ethoxysalicylidene)ethylenediiminato]manganese(III) dihydrate, $[Mn(C_{20}H_{22}N_2O_4)Cl(H_2O)] \cdot 2H_2O$

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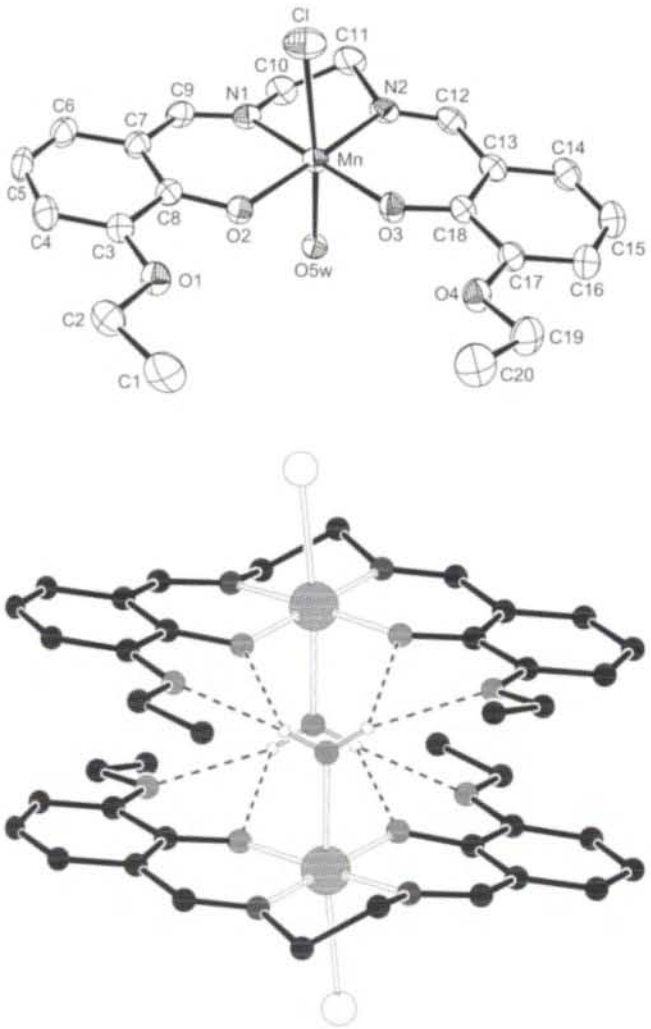

Abstract

$C_{20}H_{28}ClMnN_2O_7$, monoclinic, $P12_1/n1$ (no. 14), a = 11.863(1) Å, b = 13.960(2) Å, c = 14.005(2) Å, β = 103.990(2)°, V = 2250.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.061, $wR_{ref}(F^2)$ = 0.170, T = 293 K.

Source of material

N,N'-Bis(3-ethoxysalicylidene)ethylenediimine, $C_{20}H_{24}N_2O_4$, was synthesized according to the literature procedure [1]. $Mn(CH_3COO)_3 \cdot 2H_2O$ (0.50 g, 1.86 mmol), NaCl (0.11 g, 1.88 mmol) and *N,N'*-bis(3-ethoxysalicylidene)ethylenediimine (0.67 g, 1.88 mmol) in EtOH (70 ml) and H_2O (5 ml) were stirred for 3 h at room temperature and then filtered. The solvent was removed in vacuo, the residue washed with diethyl ether and dried, to give a brown powder (0.94 g). Crystals suitable for X-ray structure analysis were obtained via slow evaporation from a water solution.

Experimental details

The H atoms of the two solvent H_2O molecules could neither be located from Fourier difference maps nor added geometrically. The H atoms of the water ligand were localized from Fourier difference maps.

Discussion

The crystal structure of the title compound consists of neutral complex $[Mn(C_{20}H_{22}N_2O_4)Cl(H_2O)]$ and two uncoordinated water molecules. Mn(III) ion is six-coordinated, $MnClN_2O_3$, and the environment around the Mn center is distorted octahedral one (figure, top). Within the equatorial plane, the chelating angles lie in the range of 82.69(11)° – 91.54(9)° and the ∠O2–Mn–O3 bond angle is 94.03(9)°. The two Mn—N bond distances are equivalent (1.984(2) Å) and longer than the Mn—O bond (1.869(2) Å and 1.882(2) Å). The apical ∠Cl–Mn–O5w bond angle is 170.32(6)° and the bond distance between the Mn atom and the O5w atom of the water ligand is considerably long with d(Mn—O5w) = 2.328(2) Å. The complex displays the intermolecular hydrogen bonds between the O5w atom and the O atoms of the chelate ligand with d(O5w···O^i) = 2.852 Å – 3.071 Å (symmetry code i: 1–x,1–y,–z). Moreover, there are intermolecular π-π interactions between the adjacent benzene rings. For Cg1 (the centroid of six-membered ring C3-C8) and Cg2^i (ring C13-C18), the centroid-centroid distance is 3.726 Å and the dihedral angle between the ring planes is 2.6°. Two complex molecules are assembled through these hydrogen-bonding and π-π interactions (figure, bottom).

Table 1. Data collection and handling.

Crystal:	dark red block, size 0.05 × 0.05 × 0.05 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.49 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	56.56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13910, 5229
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4257
$N(param)_{refined}$:	282
Programs:	SHELXS-97 [2], SHELXL-97 [3], ORTEP-III [4], PLATON [5]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(5WA)	4e	0.3522	0.4423	0.0343	0.053
H(5WB)	4e	0.3393	0.4722	–0.0663	0.053
H(1A)	4e	0.8965	0.4938	0.1549	0.105
H(1B)	4e	1.0023	0.5406	0.2293	0.105
H(1C)	4e	0.9097	0.6056	0.1600	0.105
H(2A)	4e	0.8620	0.4921	0.3125	0.066
H(2B)	4e	0.8771	0.6042	0.3190	0.066
H(4)	4e	0.7522	0.5547	0.4158	0.062
H(5)	4e	0.6021	0.5642	0.4951	0.066
H(6)	4e	0.4157	0.5878	0.4083	0.062
H(9)	4e	0.2732	0.5984	0.2568	0.054
H(10A)	4e	0.1225	0.6391	0.1263	0.063
H(10B)	4e	0.1420	0.5600	0.0517	0.063
H(11A)	4e	0.0752	0.6905	–0.0449	0.069
H(11B)	4e	0.1592	0.7603	0.0275	0.069
H(12)	4e	0.1481	0.7089	–0.1808	0.057
H(14)	4e	0.1919	0.7091	–0.3362	0.066
H(15)	4e	0.3184	0.6957	–0.4343	0.072
H(16)	4e	0.5139	0.6649	–0.3661	0.065
H(19A)	4e	0.6914	0.7168	–0.2637	0.073
H(19B)	4e	0.6974	0.6054	–0.2788	0.073
H(20A)	4e	0.8234	0.7044	–0.1122	0.112
H(20B)	4e	0.8783	0.6591	–0.1929	0.112
H(20C)	4e	0.8268	0.5928	–0.1236	0.112

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mn	4e	0.39129(3)	0.63903(3)	0.03455(3)	0.0260(2)	0.0477(3)	0.0412(3)	0.0055(2)	0.0094(2)	0.0039(2)
Cl	4e	0.43007(9)	0.81148(6)	0.09914(9)	0.0637(6)	0.0536(5)	0.0994(8)	–0.0050(4)	0.0242(5)	–0.0125(5)
O(1)	4e	0.7327(2)	0.5627(2)	0.2233(2)	0.030(1)	0.064(1)	0.046(1)	–0.0020(9)	0.0052(8)	0.003(1)
O(2)	4e	0.5213(2)	0.5915(2)	0.1256(1)	0.029(1)	0.056(1)	0.039(1)	0.0062(8)	0.0087(7)	0.0016(9)
O(3)	4e	0.4685(2)	0.6484(2)	–0.0677(2)	0.032(1)	0.059(1)	0.040(1)	0.0069(9)	0.0088(8)	0.0104(9)
O(4)	4e	0.6151(2)	0.6418(2)	–0.1767(2)	0.038(1)	0.067(1)	0.047(1)	0.002(1)	0.0155(9)	0.013(1)
O(5W)	4e	0.3317(2)	0.4829(2)	–0.0094(2)	0.043(1)	0.048(1)	0.042(1)	0.0068(9)	0.0106(9)	0.0018(9)
N(1)	4e	0.2920(2)	0.6195(2)	0.1283(2)	0.032(1)	0.047(1)	0.050(1)	0.001(1)	0.017(1)	–0.002(1)
N(2)	4e	0.2422(2)	0.6799(2)	–0.0543(2)	0.029(1)	0.049(1)	0.057(2)	0.009(1)	0.011(1)	0.005(1)
C(1)	4e	0.9214(3)	0.5474(3)	0.1977(3)	0.033(2)	0.089(3)	0.086(3)	0.002(2)	0.013(2)	0.011(2)
C(2)	4e	0.8519(3)	0.5509(3)	0.2745(3)	0.035(2)	0.062(2)	0.060(2)	–0.002(1)	–0.003(1)	0.002(2)
C(3)	4e	0.6524(3)	0.5706(2)	0.2778(2)	0.038(2)	0.040(1)	0.041(2)	–0.005(1)	0.008(1)	–0.001(1)
C(4)	4e	0.6763(3)	0.5636(2)	0.3794(2)	0.049(2)	0.057(2)	0.044(2)	–0.004(2)	0.001(1)	0.003(1)
C(5)	4e	0.5859(3)	0.5699(2)	0.4270(2)	0.068(2)	0.060(2)	0.036(2)	–0.005(2)	0.010(1)	0.000(2)
C(6)	4e	0.4749(3)	0.5841(2)	0.3754(2)	0.058(2)	0.056(2)	0.046(2)	–0.002(2)	0.022(2)	–0.001(2)
C(7)	4e	0.4487(3)	0.5935(2)	0.2725(2)	0.043(2)	0.043(2)	0.041(2)	–0.000(1)	0.014(1)	–0.001(1)
C(8)	4e	0.5376(2)	0.5866(2)	0.2217(2)	0.037(2)	0.037(1)	0.039(1)	–0.003(1)	0.009(1)	–0.002(1)
C(9)	4e	0.3287(3)	0.6038(2)	0.2202(2)	0.040(2)	0.050(2)	0.050(2)	–0.000(1)	0.024(1)	0.001(1)
C(10)	4e	0.1676(3)	0.6222(3)	0.0795(3)	0.029(2)	0.065(2)	0.066(2)	0.001(1)	0.017(1)	0.002(2)
C(11)	4e	0.1517(3)	0.6964(3)	–0.0007(3)	0.032(2)	0.070(2)	0.072(2)	0.014(2)	0.017(2)	0.008(2)
C(12)	4e	0.2232(3)	0.6920(2)	–0.1476(2)	0.032(2)	0.047(2)	0.060(2)	0.006(1)	0.003(1)	0.008(1)
C(13)	4e	0.3067(3)	0.6821(2)	–0.2055(2)	0.037(2)	0.040(1)	0.048(2)	0.001(1)	0.004(1)	0.005(1)
C(14)	4e	0.2695(3)	0.6953(2)	–0.3082(2)	0.046(2)	0.059(2)	0.051(2)	0.002(2)	–0.003(1)	0.008(2)
C(15)	4e	0.3449(3)	0.6881(3)	–0.3666(3)	0.064(2)	0.071(2)	0.041(2)	–0.001(2)	0.002(2)	0.008(2)
C(16)	4e	0.4623(3)	0.6693(2)	–0.3257(2)	0.059(2)	0.060(2)	0.044(2)	–0.004(2)	0.015(1)	0.006(2)
C(17)	4e	0.5018(3)	0.6573(2)	–0.2252(2)	0.041(2)	0.041(1)	0.043(2)	–0.002(1)	0.010(1)	0.006(1)
C(18)	4e	0.4250(2)	0.6617(2)	–0.1626(2)	0.035(2)	0.039(1)	0.042(2)	0.001(1)	0.007(1)	0.006(1)
C(19)	4e	0.7023(3)	0.6555(3)	–0.2300(3)	0.054(2)	0.075(2)	0.059(2)	–0.007(2)	0.024(2)	0.008(2)
C(20)	4e	0.8180(3)	0.6527(3)	–0.1583(3)	0.041(2)	0.101(3)	0.085(3)	–0.004(2)	0.022(2)	0.013(2)
O(6W)	4e	0.4011(3)	0.1669(3)	0.0474(3)	0.105(3)	0.119(3)	0.105(3)	–0.044(2)	0.006(2)	0.010(2)
O(7W)	4e	0.3086(2)	0.0130(2)	0.1249(2)	0.064(2)	0.103(2)	0.085(2)	0.005(2)	0.029(2)	–0.002(2)

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