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Aqua(*N*,*N*′-disalicylideneethylenediamino-*N*,*N*′,*O*,*O*′)(4,5-imidazoledicarboxylato-*O*)-manganese(III) monohydrate

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Key indicators

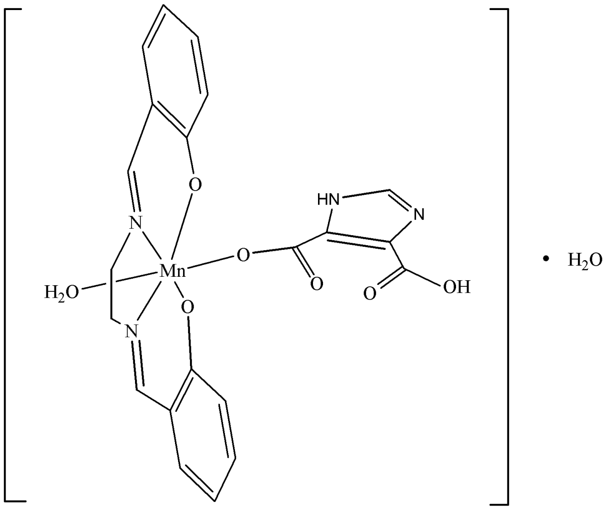
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.130
Data-to-parameter ratio = 11.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, [$\mathrm{Mn^{III}}$(salen)($\mathrm{OCOC_3H_2N_2COOH}$)($\mathrm{H_2O}$)]·$\mathrm{H_2O}$, ($\mathrm{H_2}$salen = *N*,*N*′-disalicylideneethylenediamine, $\mathrm{C_{16}H_{14}N_2O_2}$), two N and two O atoms from salen chelate to the $\mathrm{Mn^{III}}$ ion, forming a distorted octahedral coordination sphere together with two other axial O atoms from a carboxyl group and a water molecule. Extended intermolecular hydrogen bonds are observed to link the complex molecules, forming a one-dimensional chain structure.

Comment

The imidazole N atom of histidine has been described as a functional group (Wieghardt, 1989) in the oxygen-evolving complex (EOC) of photosystem II (PS II). 4,5-Imidazoledicarboxylate was suggested as a bridging ligand (Rypniewski *et al.*, 1995; Tainer *et al.*, 1982) and a dinuclear $\mathrm{Mn^{IV}}$ cluster in 4,5-imidazoledicarboxylate-bridged dimanganese(IV) has been reported (Caudle *et al.*, 1997). It is interesting to study the differing coordination behavior of 4,5-dicarboxyimidazole. We present here a new complex, [Mn(salen)($\mathrm{OCOC_3H_2N_2COOH}$)($\mathrm{H_2O}$)]·$\mathrm{H_2O}$, (I), in which 4,5-imidazoledicarboxylate coordinates to $\mathrm{Mn^{III}}$ as a terminal ligand. The structure of (I) is shown in Fig. 1. The Mn atom is chelated by two N [Mn—N1 1.991 (2) Å and Mn—N2 1.989 (2) Å] and two O atoms [Mn—O1 1.8793 (18) Å and Mn—O2 1.8944 (18) Å] from the salen ligand to form an equatorial plane with a maximum deviation of 0.0052 (10) Å from the least-squares plane, while the opposite axial positions are occupied by another two O atoms from the 4,5-dicarboxylatoimidazole [Mn—O4 2.1815 (19) Å] and the ligated aqua



(I)

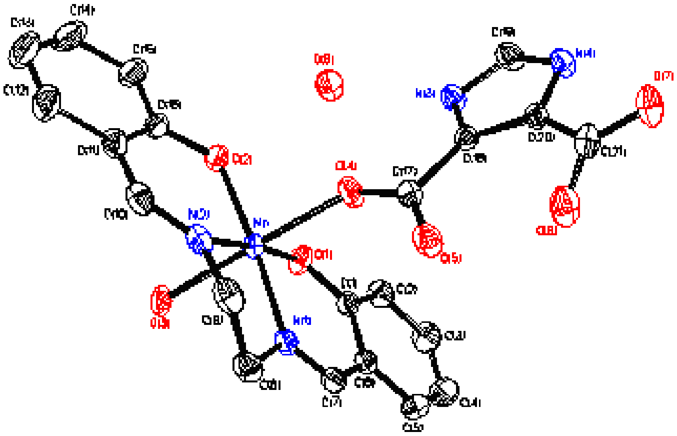


Figure 1
Diagram (*SHELXTL*; Bruker, 1998) of (I) showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level. H atoms have been omitted for clarity.

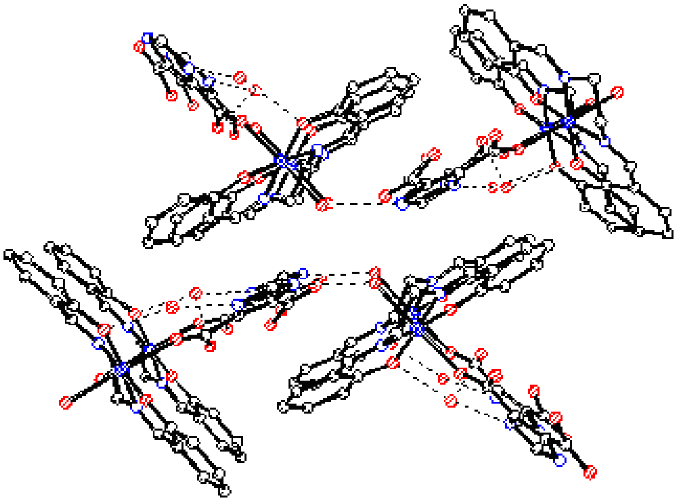


Figure 2
A packing diagram of the title compound.

[Mn—O3 2.249 (2) Å]. The Mn site is surrounded by a distorted octahedral coordination sphere, with the *trans* angles ranging from 172.83 (9) to 178.04 (9)° and the other angles ranging from N2—Mn—N1 of 82.50 (9)° to O2—Mn—O1 of 94.69 (8)°. All the bond lengths and angles are comparable to those of other six-coordinate Mn^{III} Schiff base complexes (Shyu *et al.*, 1999; Watkinson *et al.*, 1999; Oshio *et al.*, 1995), while the Mn—O bonds of (I) are longer than those reported by Caudle *et al.* (1997), where Mn possesses a 4+ oxidation state, higher than that in (I).

A packing diagram of the title compound is presented in Fig. 2 and shows the extensive intermolecular hydrogen-bonding interactions, which contain $N_{imidazo}\cdots H—O_{aqua}$ [2.992 (3) Å], $O_{carboxy}\cdots H—O_{aqua}$ [2.880 (3) Å] and $N_{imidazo}\cdots H—O_{aqua}$ [2.861 (3) Å]. These hydrogen bonds produce a one-dimensional polymeric structure.

Experimental

To a solution of 0.268 g (1 mmol) of H_2salen in 40 ml of CH_2Cl_2/MeCN (*v*/*v*, 1:1), 0.156 g (1 mmol) of 4,5-dicarboxyimidazole was added and the resulting solution stirred. The reaction solution was refluxed for 18 h, accompanied by the addition of 0.245 g (1 mmol) of $Mn(CH_3COO)_2.4H_2O$. After filtration, the filtrate was allowed to stand for several days at room temperature to deposit dark-red crystals of (I), which were recrystallized from ethanol solution to obtain the crystals suitable for X-ray diffraction.

Crystal data

$[Mn(C_5H_3N_2O_4)(C_{16}H_{14}N_2O_2)(H_2O)]\cdot H_2O$
$M_r = 512.36$
Monoclinic, $P2_1/n$
$a = 7.009$ (3) Å
$b = 19.8634$ (4) Å
$c = 16.2811$ (3) Å
$\beta = 101.422$ (1)°
$V = 2221.93$ (6) Å^3
$Z = 4$
$D_x = 1.532$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4856 reflections
$\theta = 1.6$–25.0°
$\mu = 0.65$ mm^{-1}
$T = 293$ (2) K
Block, dark-red
0.76 × 0.54 × 0.34 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.611$, $T_{max} = 0.802$
7277 measured reflections
3856 independent reflections
3101 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 6$
$k = -23 \rightarrow 17$
$l = -19 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.130$
$S = 0.97$
3856 reflections
327 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mn—O1	1.8793 (18)	Mn—N1	1.991 (2)
Mn—O2	1.8944 (18)	Mn—O4	2.1815 (19)
Mn—N2	1.989 (2)	Mn—O3	2.249 (2)
O1—Mn—O2	94.69 (8)	N2—Mn—O4	92.95 (9)
O1—Mn—N2	174.55 (9)	N1—Mn—O4	91.45 (8)
O2—Mn—N2	90.42 (9)	O1—Mn—O3	89.20 (10)
O1—Mn—N1	92.35 (9)	O2—Mn—O3	90.78 (9)
O2—Mn—N1	172.83 (9)	N2—Mn—O3	88.84 (10)
N2—Mn—N1	82.50 (9)	N1—Mn—O3	87.97 (9)
O1—Mn—O4	88.95 (9)	O4—Mn—O3	178.04 (9)
O2—Mn—O4	90.02 (8)		

H atoms were located and included in the structure-factor calculations but their positions were not refined, except for those of the aqua molecule coordinated to Mn.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *SHELXTL XPREP* (Siemens, 1994); program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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