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Redetermination of *catena*-poly[[diaquamanganese(II)]-μ-iminodiacetato-$\kappa^4N,O,O':O''$]

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Key indicators

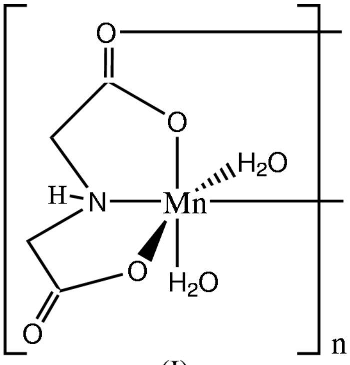
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.021
wR factor = 0.051
Data-to-parameter ratio = 13.4

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

The redetermined structure of the title compound, $[Mn(C_4H_5NO_4)(H_2O)_2]_n$, including the full hydrogen-bonding scheme, is reported; the original report was by Zhang & Lu [*Acta Cryst.* (2004). E**60**, m1189–m1190]. The structure contains polymeric chains of Mn^{II} cations bridged by iminodiacetate (IDA) dianions. The manganese species assumes a distorted octahedral coordination geometry, formed by two IDA molecules (one tridentate and one monodentate) and two water molecules. The tridentate IDA chelates to the Mn atom in the facial configuration; one chelating five-membered ring is almost planar and the other adopts an envelope configuration. A significant difference of 0.118 (2) Å in Mn—$O_{carboxyl}$ bond distances is observed within the chelating IDA moiety.

Comment

Iminodiacetate (IDA), oxydiacetate (ODA) and thiodiacetate (TDA) dianions have been widely observed as tridentate chelating ligands in metal complexes. A search of the Cambridge Structure Database (Version 5.25; Allen, 2002) revealed that they can display meridional (*mer*) or facial (*fac*) configurations. In the title complex, (I), IDA chelates to the Mn^{II} atom in a *fac* configuration. Zhang & Lu (2004) recently reported this structure but did not locate all the H atoms.



(I)

The structure of (I) is illustrated in Fig. 1. The complex is polymeric, and the repeat unit contains an Mn^{II} atom, an IDA dianion and two coordinated water molecules. Each IDA ligand bridges two neighbouring Mn atoms, and thus the Mn^{II} atom is surrounded by two IDA (one tridentate and one monodentate) and two water molecules in a distorted octahedral coordination geometry. Two carboxyl O atoms (O1 and O3) of the same IDA ligand and two water molecules form the equatorial coordination plane, while the bridging O atom (O2^i;

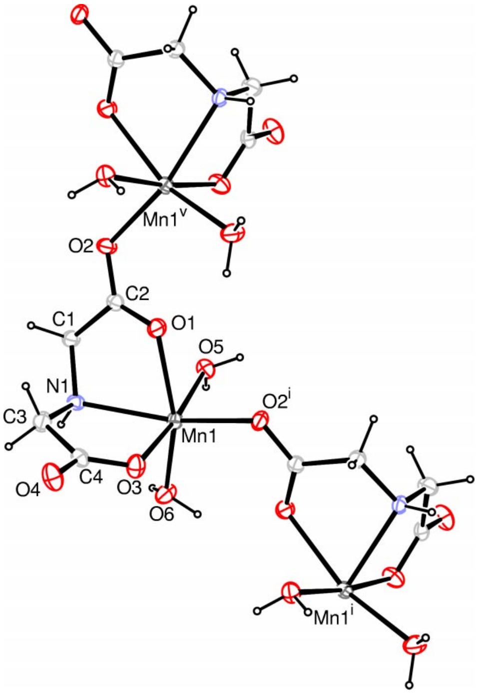


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids. [Symmetry codes: (i) $-x+1, -y+1, z+\frac{1}{2}$; (v) $-x+1, -y+1, z-\frac{1}{2}$].

Fig. 1) from the neighbouring IDA molecule and the imine N atom occupy the axial sites, with a resulting O2^{i}—Mn—N1 bond angle of 155.88 (6)°.

IDA chelates to the Mn^{II} atom in a facial configuration, resulting in two chelating five-membered rings that show distinctly different conformations. The atoms of the O3-containing chelate ring are nearly coplanar, the maximum atomic deviation being 0.0726 (12) Å for N1, whereas the O1-containing ring displays an envelope conformation, with the Mn1 atom in the flap position and out of the mean plane formed by the other four atoms by 1.092 (3) Å. The Mn1—O1 bond is 0.118 (2) Å longer than the corresponding Mn1—O3 bond (Table 1). This bond-length difference is similar to that [0.096 (6) Å] found in the Ni^{II} analogue (Wu *et al.*, 2003) of (I), and may result from the significant deviation [0.650 (3) Å] of atom O1 from the coordination plane formed by atoms O3, O5, O6 and Mn1. The C2 carboxyl group of IDA bridges to a neighbouring Mn^{II} atom to form a polymeric chain along the *c* axis (Fig. 1). Atom O4 of the C4 carboxyl group does not coordinate to the Mn1 atom but acts as an acceptor for a hydrogen bond from a coordinated water molecule of a neighbouring complex (Table 2), resulting in the overall crystal structure (Fig. 2).

Experimental

An aqueous solution (20 ml) containing H_2IDA (0.13 g, 1 mmol), NaOH (0.08 g, 2 mmol) and $MnCl_2\cdot4H_2O$ (0.20 g, 1 mmol) was refluxed for 6 h. After cooling to room temperature, the solution was filtered. Single crystals of (I) were obtained from the filtrate after three weeks.

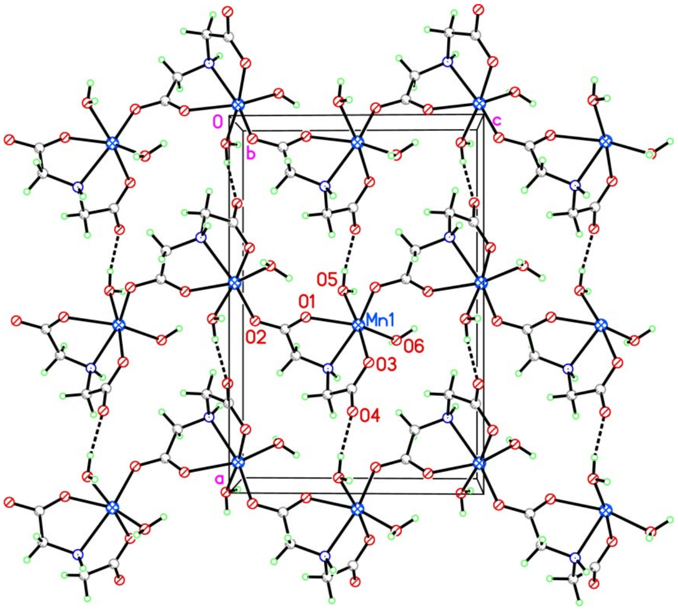


Figure 2
The unit-cell packing for (I), showing hydrogen bonding between polymeric complex chains as dashed lines.

Crystal data

$[Mn(C_4H_5NO_4)(H_2O)_2]$
$M_r = 222.06$
Orthorhombic, $Pca2_1$
$a = 14.5818$ (15) Å
$b = 5.3508$ (6) Å
$c = 9.7958$ (11) Å
$V = 764.31$ (14) Å^3
$Z = 4$
$D_x = 1.930$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3836 reflections
$\theta = 3.0$–26.0°
$\mu = 1.72$ mm^{-1}
$T = 295$ (2) K
Prism, colourless
0.36 × 0.32 × 0.26 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.540$, $T_{max} = 0.635$
4295 measured reflections
1466 independent reflections
1440 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.1°$
$h = -18 \rightarrow 18$
$k = -6 \rightarrow 6$
$l = -12 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.051$
$S = 1.06$
1466 reflections
109 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2 + 0.14P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Absolute structure: Flack (1983), 573 Friedel Pairs
Flack parameter = 0.030 (18)

Table 1
Selected interatomic distances (Å).

Mn1—O1	2.2766 (18)	Mn1—O5	2.2441 (16)
Mn1—O2^{i}	2.1195 (15)	Mn1—O6	2.1872 (17)
Mn1—O3	2.1585 (16)	Mn1—N1	2.2760 (18)

Symmetry code: (i) $-x+1, -y+1, z+\frac{1}{2}$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O4^{ii}	0.90	2.04	2.940 (2)	174
O5—H5$A\cdots$O2^{iii}	0.86	1.96	2.822 (2)	174
O5—H5$B\cdots$O4^{iv}	0.84	1.82	2.646 (2)	170
O6—H6$A\cdots$O3^{ii}	0.85	1.96	2.762 (2)	158
O6—H6$B\cdots$O5^{iii}	0.88	2.18	2.975 (2)	150

Symmetry codes: (ii) $x, 1+y, z$; (iii) $1-x, 2-y, \frac{1}{2}+z$; (iv) $x-\frac{1}{2}, 1-y, z$.

H atoms on C atoms were placed in calculated positions, with C—H = 0.97 Å, and included in the final cycles of refinement in a riding model, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(carrier atom). Other H atoms were located in a difference Fourier map and refined as riding in their as-found positions relative to O or N atoms, with a fixed $U_{\rm iso}$(H) value of 0.05 Å^2.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *XP* (Siemens, 1994); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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