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catena-Poly[[[diaquacalcium(II)]-μ-oxydiacetato-[tetraaquamanganese(II)]-μ-oxydiacetato] dihydrate]

Qian Yang, Dong-Dong Lin and Duan-Jun Xu*

Department of Chemistry, Zhejiang University, People's Republic of China

Correspondence e-mail: xudj@mail.hz.zj.cn

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Key indicators

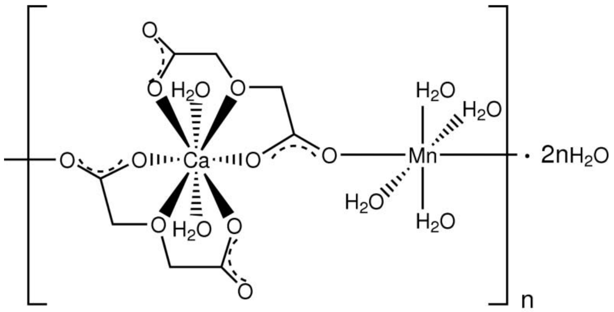
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.154
Data-to-parameter ratio = 18.6

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

In the title complex, $\{[CaMn(C_4H_4O_5)_2(H_2O)_6]\cdot 2H_2O\}_n$, the Ca^{II} and Mn^{II} ions are bridged by oxydiacetate (ODA) dianions to form zigzag polymeric chains. The Ca^{II} ion is located at a twofold axis and is coordinated by two tridentate ODA and two water molecules with a distorted tetragonal antiprism coordination geometry. The Mn^{II} ion is located at another twofold axis, and assumes a distorted octahedral coordination geometry formed by four water molecules and two carboxyl O atoms from monodentate ODA dianions also chelating to the neighboring Ca^{II} atoms.

Comment

As a tridentate ligand, the oxydiacetate (ODA) dianion usually adopts two kinds of chelating conformations, meridional or facial, in metal complexes. To study the factors affecting the chelating conformation of ODA, a series of complexes incorporating ODA has been prepared and their crystal structures determined in our laboratory (Cao *et al.*, 2004; Luo *et al.*, 2004; Gaduo *et al.*, 2005). We present here the crystal structure of the title mixed-metal (Mn^{II} and Ca^{II}) complex, (I), in which the ODA plays the roles of both a chelating and a bridging ligand.



(I)

A segment of the polymeric structure of (I) is illustrated in Fig. 1. The asymmetric unit contains one Ca^{II} ion and one Mn^{II} ion, which are bridged by ODA to form a zigzag polymeric chain which propagates along the crystallographic *a* axis.

The Ca^{II} ion is located at a twofold axis of symmetry and assumes a distorted tetragonal antiprismatic coordination geometry, formed by two tridentate ODA moieties and two water molecules. The Ca—O_c (c = carboxyl) bond distances are much shorter than the Ca—O_e (e = ether) bond distance (Table 1). This distinction in *M*—O bond distances agrees with those found in the reported Mn^{II} complexes of ODA (Grirrane *et al.*, 2004; Luo *et al.*, 2004).

The Mn^{II} ion is located on another twofold axis and assumes a distorted octahedral coordination geometry, formed by four

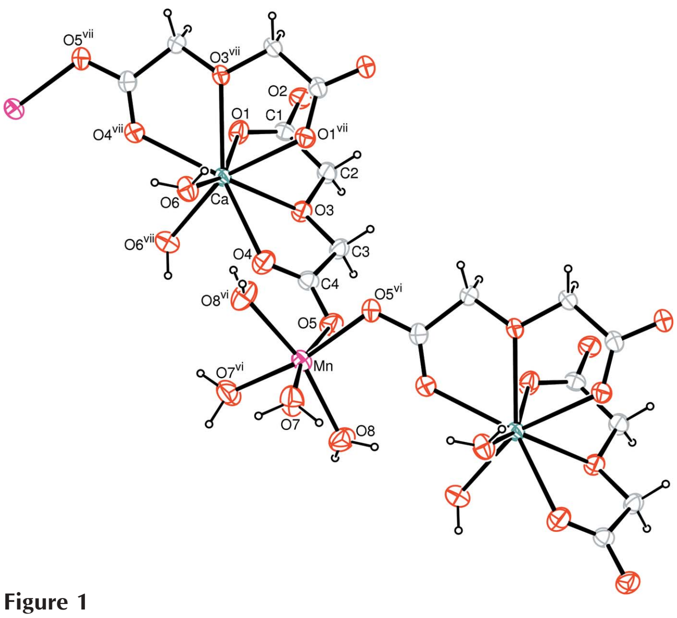


Figure 1
A segment of the polymeric structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry codes: (vi) $\frac{1}{2} - x, \frac{1}{2} - y, z$; (vii) $\frac{3}{2} - x, \frac{1}{2} - y, z$].

water molecules and two carboxyl O atoms from the the ODA dianions also chelating with the neighboring Ca^{II} ion.

The ODA meridionally chelates to the Ca^{II} ion to form two chelating five-membered rings. Each ring displays an envelope conformation. The atoms located in the flap positions are out-of the mean plane formed by the other four atoms by 0.433 (5) (O3) and 0.921 (6) Å (Ca), respectively.

The adjacent polymeric chains are linked together *via* O—H$\cdots$O hydrogen bonding between coordinated water molecules and carboxyl groups of ODA (Table 2). Ether atom O3 is not involved in the hydrogen-bonding network.

Experimental

A hot aqueous solution (10 ml) of H_2ODA (1 mmol) neutralized by Na_2CO_3 (about 1 mmol) was mixed with an aqueous solution (10 ml) of $MnCl_2\cdot4H_2O$ (1 mmol) and $CaCl_2$ (1 mmol). The solution was refluxed for 3 h and then filtered. Colorless single crystals of (I) were obtained from the filtrate after 3 d.

Crystal data

$[CaMn(C_4H_4O_5)_2(H_2O)_6]\cdot2H_2O$
$M_r = 503.29$
Orthorhombic, *Pccn*
$a = 9.9572$ (18) Å
$b = 12.819$ (2) Å
$c = 15.061$ (3) Å
$V = 1922.4$ (6) Å^3
$Z = 4$
$D_x = 1.739$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3866 reflections
$\theta = 3.0$–25.0°
$\mu = 1.04$ mm^{-1}
$T = 295$ (2) K
Prism, colorless
0.40 × 0.38 × 0.30 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.678$, $T_{max} = 0.736$
11775 measured reflections

2379 independent reflections
1341 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 28.3°$
$h = -12 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -20 \rightarrow 10$

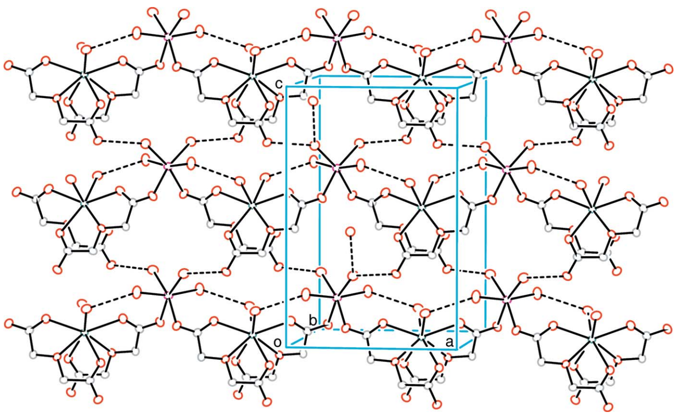


Figure 2
The unit-cell packing digram for (I), showing the hydrogen bonding (dashed lines) between polymeric chains. H atoms have been omitted.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.154$
$S = 0.87$
2379 reflections
128 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0832P)^2 + 3.6036P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Mn—O5	2.181 (3)	Ca—O3	2.493 (2)
Mn—O7	2.177 (3)	Ca—O4	2.390 (3)
Mn—O8	2.207 (3)	Ca—O6	2.415 (3)
Ca—O1	2.425 (3)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H1$A\cdots$O1^{i}	0.87	1.98	2.826 (4)	163
O1W—H1$B\cdots$O5	0.83	1.95	2.756 (4)	166
O6—H6$A\cdots$O2^{ii}	0.82	2.07	2.838 (4)	156
O6—H6$B\cdots$O1W^{iii}	0.85	1.92	2.772 (4)	173
O7—H7$A\cdots$O1W^{ii}	0.88	1.97	2.751 (4)	148
O7—H7$B\cdots$O2^{iv}	0.97	1.84	2.797 (4)	169
O8—H8$A\cdots$O2^{v}	0.92	1.92	2.838 (4)	178
O8—H8$B\cdots$O4^{vi}	0.83	2.29	2.825 (4)	122
O8—H8$B\cdots$O6^{vi}	0.83	2.31	3.094 (4)	158

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

H atoms on water molecules were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{iso}(H) = 1.5U_{eq}(O)$. Other H atoms were placed in calculated positions, with C—H = 0.97 Å, and included in the final cycles of refinement as riding with the constraint $U_{iso}(H) = 1.2U_{eq}(C)$ applied.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia,

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1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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