

Poly[[[aqua(2,2'-bipyridine- κ^2N,N')-manganese(II)]- μ -croconato- $\kappa^4O,O':-O'',O'''$] monohydrate]: a one-dimensional coordination polymer connected by hydrophilic–hydrophilic and lipophilic–lipophilic interactions at 135 K

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Received 23 December 2009

Accepted 23 February 2010

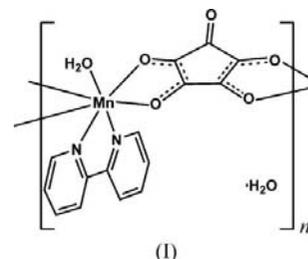
Online 6 March 2010

In the title one-dimensional coordination polymer, $\{[\text{Mn}(\text{C}_5\text{O}_5)(\text{C}_{10}\text{H}_8\text{N}_2)(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}\}_n$, each Mn^{II} ion is seven-coordinated by four O atoms from two croconate ligands, two N atoms from a 2,2'-bipyridine (2,2'-bipy) ligand and one O atom from an aqua ligand. The croconate ligand bridges the Mn^{II} ions in a bis-bidentate chelation mode, forming an extended $[\text{Mn}(\text{C}_5\text{O}_5)]_n$ chain running parallel to the [001] direction, with the lipophilic 2,2'-bipy ligands lying along one side and the hydrophilic water molecules along the opposite side. Coordinated water and solvent water molecules are arranged in the hydrophilic layer, which is characterized by $\text{O}—\text{H}\cdots\text{O}$ hydrogen bonds between croconate ligands. Meanwhile, 2,2'-bipy ligands from adjacent chains partially overlap and exhibit π – π interactions to form a lipophilic layer. The hydrophilic and lipophilic layers are arranged alternately to build a layer structure.

Comment

The chemistry of the croconate dianion ($\text{C}_5\text{O}_5^{2-}$) dates from 1825 (Gmelin, 1825). In recent years, the coordination chemistry of the croconate ligand has attracted much attention because of its ability to construct various one- to three-dimensional frameworks. One or two croconate ligands can chelate in a bidentate manner a metal ion to form $[\text{M}(\text{C}_5\text{O}_5)]$ (Brouca-Cabarrecq & Trombe, 1992*a,b*) or $[\text{M}(\text{C}_5\text{O}_5)_2]$ complexes (Chen *et al.*, 2005). Various croconate–metal frameworks with three to five O atoms involved in coordination and

bridging have also been formed (Glick & Dahl, 1966; Brouca-Cabarrecq & Trombe, 1992*a,b*; Cornia *et al.*, 1993; Maji *et al.*, 2003). We have previously reported several mixed-ligand complexes formulated as $[\text{M}(\text{C}_5\text{O}_5)(\text{phen})_2]$ (phen is 1,10-phenanthroline). Within this series, $[\text{Mn}(\text{C}_5\text{O}_5)(\text{phen})_2]$ (Chen, Chen *et al.*, 2008) and $[\text{Cu}(\text{C}_5\text{O}_5)(\text{phen})_2]$ (Chen *et al.*, 2007) crystallize in the space group $C2/c$, while $[\text{Co}(\text{C}_5\text{O}_5)(\text{phen})_2]$ and $[\text{Ni}(\text{C}_5\text{O}_5)(\text{phen})_2]$ (Chen *et al.*, 2007) crystallize in the space group $Pbcn$. These complexes do not show polymeric network structures.



2,2'-Bipyridine (2,2'-bipy) also has excellent chelating and π -conjugation ability. However, in comparison with phen, it has greater structural flexibility and has accordingly been used to build supramolecular architectures (Sun *et al.*, 2005). We therefore chose 2,2'-bipy in place of phen to develop new mixed-ligand complexes and $[\text{Ni}(\text{C}_5\text{O}_5)(2,2'\text{-bipy})_2]$ was thus obtained (Chen, Fang & Yu, 2008). This complex is isostruc-

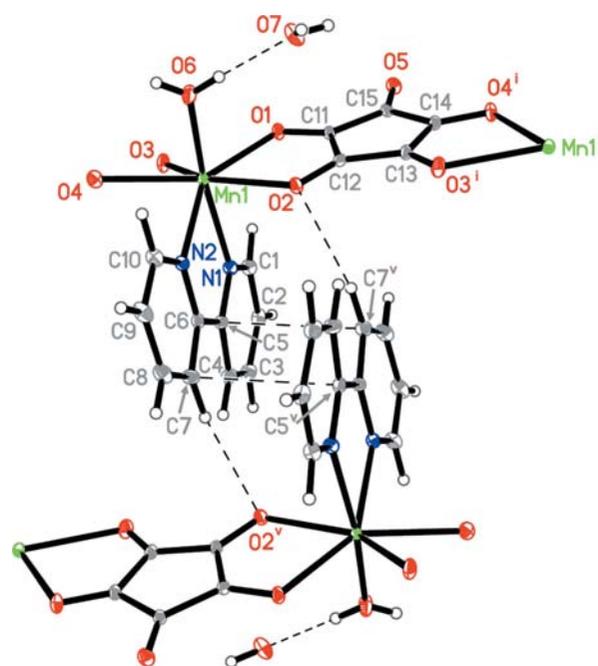
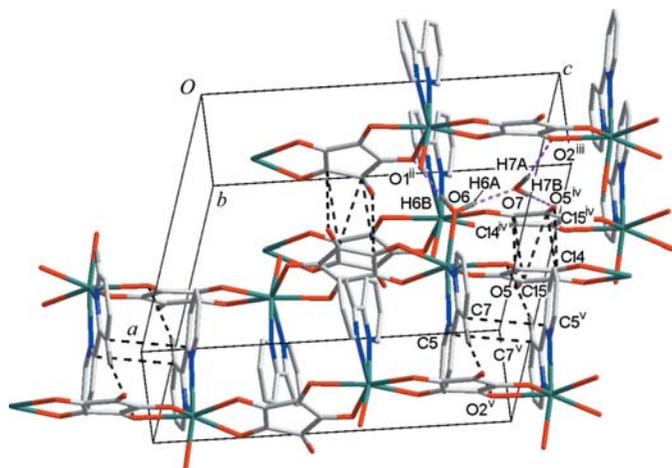


Figure 1

The unique $[\text{Mn}(\text{C}_5\text{O}_5)(2,2'\text{-bipy})(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$ unit of (I) at 135 K, together with another 'molecule' to show the π – π interactions and hydrogen bonds (dashed lines). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (v) $-x + 2, -y, -z + 2$.]


Figure 2

A side view of several $[\text{Mn}(\text{C}_5\text{O}_5)]_n$ chains in (I), showing the chain structure and the hydrophilic-hydrophilic and lipophilic-lipophilic interactions at 135 K. Dashed lines denote unique $\text{O}-\text{H}\cdots\text{O}$ (red lines in the electronic version of the paper) and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and intermolecular $\text{C}\cdots\text{C}$ and $\text{C}\cdots\text{O}$ short contacts. [Symmetry codes: (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y, -z+2$; (iv) $-x+1, 1-y, -z+2$; (v) $-x+2, -y, -z+2$.]

tural with $[\text{Ni}(\text{C}_5\text{O}_5)(\text{phen})_2]$ and also shows an isolated structure. Recently, we synthesized the title mixed-ligand complex, $\{[\text{Mn}(\text{C}_5\text{O}_5)(2,2'\text{-bipy})(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}\}_n$ (I), which shows a bis-bidentate bridging mode and a one-dimensional polymeric structure. We report here its crystal structure at 135 K. The structure at 291 K has also been determined; it is essentially the same as that at 135 K, and the data have been deposited in the supplementary material. Note that in all of our previous or present mixed-ligand complexes, the neutral phen or 2,2'-bipy ligands are essentially lipophilic, while the croconate anion is prone to be hydrophilic, and its alkali metal salts can easily dissolve in water.

As shown in Fig. 1, the asymmetric unit of (I) comprises an Mn^{II} ion and three different ligands, with a solvent water molecule linking to the aqua ligand *via* a hydrogen bond. The seven coordinating atoms make up a severely distorted pentagonal bipyramid. Atoms O6 and N1 are in axial positions, with the $\text{O6}-\text{Mn1}-\text{N1}$ moiety being essentially linear [$170.14(3)^\circ$] and with the $\text{Mn1}-\text{O6}(\text{aqua})$ bond length [$2.1484(8) \text{ \AA}$] being the shortest in the MnO_5N_2 coordination polyhedron. Among the five equatorial atoms, O1 and N2 are displaced by $-0.800(1)$ and $1.582(1) \text{ \AA}$, respectively, on opposite sides of the plane defined by atoms O2, O3 and O4. The terminal $\text{C15}-\text{O5}$ bond shows the 'ketonic' $\text{C}=\text{O}$ bond length [$1.2360(10) \text{ \AA}$], while the other four $\text{C}-\text{O}$ bonds involved in coordination show longer $\text{C}-\text{O}$ distances.

The croconate ligand is bridging, and exhibits a bis-bidentate chelation mode through four O atoms to two Mn^{II} ions. In this way, an extended one-dimensional coordination chain is formed along the [001] direction (Fig. 2), in which the zigzag-arranged Mn^{II} ions are the nodes connecting a series of planar croconate ligands. The planes of two neighbouring croconate ligands in the chain have a dihedral angle of $22.7(1)^\circ$ and the two carbonyl groups point to opposite sides of the chain.

Along the $[\text{Mn}(\text{C}_5\text{O}_5)]_n$ chain, 2,2'-bipy ligands are attached to the same side, forming the lipophilic side of the chain. The least-squares plane of the 2,2'-bipy ligand is roughly perpendicular to those of the two croconate ligands coordinated to the same Mn^{II} ion, with dihedral angles of $89.3(1)$ and $84.6(1)^\circ$. The aqua ligands, which lie *trans* to the 2,2'-bipy ligands, form the hydrophilic side of the $[\text{Mn}(\text{C}_5\text{O}_5)]_n$ chain. The $[\text{Mn}(\text{C}_5\text{O}_5)]_n$ chains run parallel to *c*-glide planes, with the croconate ligands lying across these glide planes. The dihedral angle between the glide plane and the plane of the croconate ligand is $81.5(1)^\circ$, and that between the glide plane and the plane of the 2,2'-bipy ligand is $77.9(1)^\circ$.

To date, several polymeric mixed-ligand croconate complexes have been reported. In the one-dimensional coordination polymer $\{[\text{Co}_2(\text{C}_5\text{O}_5)_2(\text{bpds})_2(\text{H}_2\text{O})_4]\cdot 3\text{H}_2\text{O}\}_n$ (bpds is 4,4'-bipyridyl disulfide), the terminal-bidentate croconate ligand is at the side of the $[\text{Co}(\text{bpds})]_n$ main chain (Manna *et al.*, 2007). In contrast to 2,2'-bipy, 4,4'-bipyridine (4,4'-bipy) is a bridging ligand that is useful for the construction of two-dimensional frameworks. The $[\text{Cd}_2(\text{C}_5\text{O}_5)_2(4,4'\text{-bipy})(\text{H}_2\text{O})]_n$ complex (Wang *et al.*, 2003) and the isostructural $\{[\text{Cd}_2(\text{C}_5\text{O}_5)_2(\text{bipy})_2(\text{H}_2\text{O})]\cdot 2\text{H}_2\text{O}\}_n$ [bipy is 1,2-bis(4-pyridyl)ethylene; Wang, Tseng *et al.*, 2007] complexes exhibit a tightly-bonded bilayer two-dimensional framework, which is characterized by a tris-bidentate bridging mode in the $[\text{Cd}(\text{C}_5\text{O}_5)]_n$ chain. The $\{[\text{Cu}_2(\text{C}_5\text{O}_5)_2(\text{bipy})_2]\cdot \text{H}_2\text{O}\}_n$ complex shows an undulating monolayer two-dimensional framework (Ghoshal *et al.*, 2005), while $\{[\text{Zn}_2(\text{C}_5\text{O}_5)_2(\text{bipy})_2]\cdot \text{H}_2\text{O}\}_n$ (Wang, Tseng *et al.*, 2007) exhibits a brick-wall-like two-dimensional structure. Similar bilayer two-dimensional frameworks can be found in the group of isostructural complexes $[\text{M}(\text{C}_5\text{O}_5)(\text{bipy})]_n$ [$\text{M} = \text{Mn, Fe, Co, Cd}$; bipy is 1,2-bis(4-pyridyl)ethane; Wang, Dai *et al.*, 2007]. Small ligands such as pyrazine (Maji *et al.*, 2004) or polydentate *N,N'*-bis(3-aminopropyl)-oxamide (Castro *et al.*, 2001) are also involved in two-dimensional mixed-ligand frameworks. One could consider that the 2,2'-bipyrimidine (bipym) ligand combines the properties of bidentate 2,2'-bipy and bridging 4,4'-bipy, and it has thus been employed to enhance dimensionality. However, the structure of $[\text{Cd}_2(\text{C}_5\text{O}_5)_2(\text{bipym})(\text{H}_2\text{O})_2]_n$ also shows a two-dimensional framework (Wang, Kuo *et al.*, 2007), in which the $[\text{Cd}(\text{C}_5\text{O}_5)]_n$ chain is similar to the $[\text{Mn}(\text{C}_5\text{O}_5)]_n$ chain of (I).

Fig. 3 shows a projection along a bundle of polymeric coordination chains of (I) (along the *c* direction), which are crosslinked by hydrogen bonding and $\pi-\pi$ interactions. Coordinated water and solvent water molecules from neighbouring chains come together to form a hydrophilic layer, and 2,2'-bipy ligands from adjacent chains form a lipophilic layer. The hydrophilic and lipophilic layers are stacked alternately to build a layer structure.

The detailed hydrophilic-hydrophilic interactions are exhibited in Fig. 2 and Table 2, and involve four unique $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. Both the coordinated and solvent water molecules act as double hydrogen-bond donors in these hydrogen bonds, and the latter also acts as a single hydrogen-bond acceptor. Besides hydrogen bonding, there are several short intermolecular $\text{C}\cdots\text{O}$ and $\text{C}\cdots\text{C}$ contacts involving the

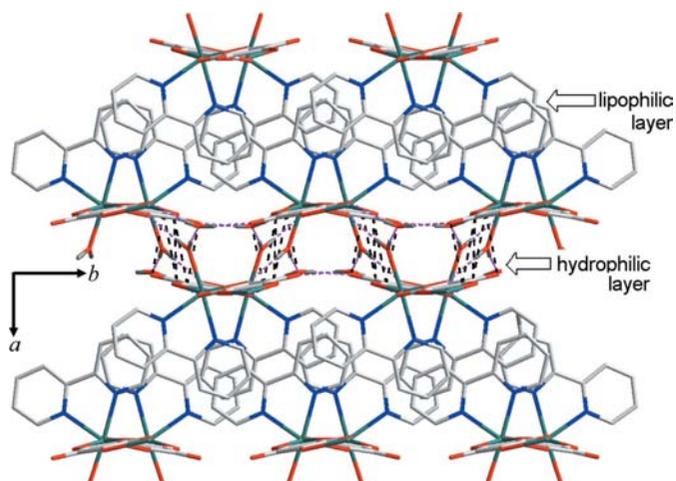


Figure 3

A projection of the bundle of $[\text{Mn}(\text{C}_5\text{O}_5)_n]$ chains along the chain direction (c direction) at 135 K, showing the alternate hydrophilic and lipophilic layers. Dashed lines denote $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (red lines in the electronic version of the paper) and intermolecular $\text{C}\cdots\text{C}$ and $\text{C}\cdots\text{O}$ short contacts (for a clearer visualization, see Fig. 2).

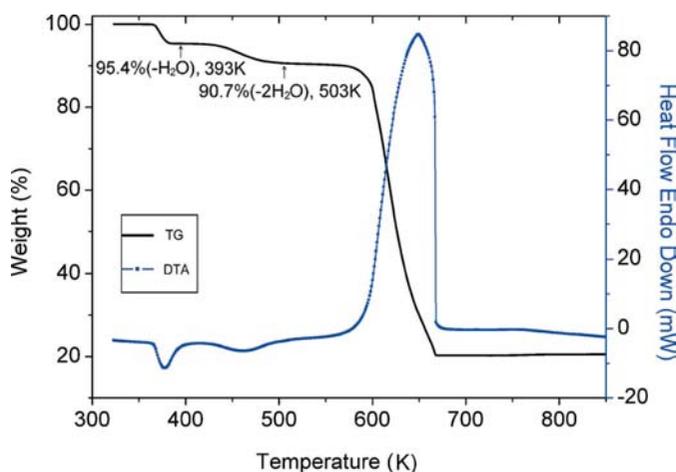


Figure 4

The thermogravimetric (TG) curve of (I), together with the differential thermal analysis (DTA) curve, at a heating rate of 10 K min^{-1} under a nitrogen atmosphere.

$\text{C}=\text{O}$ group in the hydrophilic layer: the $\text{C}14\cdots\text{O}5^{\text{iv}}$, $\text{C}14\cdots\text{C}15^{\text{iv}}$ and $\text{C}15\cdots\text{C}15^{\text{iv}}$ distances are 2.9002 (11), 3.1888 (12) and 3.1170 (12) Å, respectively [symmetry code: (iv) $-x + 1, -y + 1, -z + 2$]. These interactions may also be considered to be π - π interactions between the $\text{C}=\text{O}$ group and an adjacent croconate C_5 ring, as these two entities are parallel.

As shown in Fig. 2, the lipophilic layer is characterized by π - π interactions in 2,2'-bipy pairs between neighbouring $[\text{Mn}(\text{C}_5\text{O}_5)_n]$ chains. The crosslinking of the chains through these π - π interactions is further assisted by the $\text{C}7-\text{H}7\cdots\text{O}2(-x + 2, -y, -z + 2)$ interaction. The 2,2'-bipy ligand is basically planar, with a small dihedral angle of $9.4(1)^\circ$ between its two pyridine rings as a result of the bidentate chelation balance and intra-ligand $\text{H}\cdots\text{H}$ repulsion. The two

2,2'-bipy ligands in the π - π interaction are approximately half-overlapped, with the interplanar spacing being 3.429 (5) Å (the spacing is based on the least-squares plane of the 2,2'-bipy ligand). The $\text{C}5\cdots\text{C}7(-x + 2, -y, -z + 2)$ distance of 3.2664 (13) Å in the 2,2'-bipy pair is slightly shorter than the interplanar spacing and is nearly perpendicular [$85.1(1)^\circ$] to these planes. Thus, the structural flexibility of the 2,2'-bipy ligand is manifested in (I). Using the rigid phen ligand and similar synthetic methods, the resultant $[\text{Mn}(\text{C}_5\text{O}_5)(\text{phen})_2]$ complex does not show a network structure (Chen, Chen *et al.*, 2008; Chen *et al.*, 2007). Note that $[\text{Ni}(\text{C}_5\text{O}_5)(2,2'\text{-bipy})_2]$ also fails to form a network structure (Chen, Fang & Yu, 2008). The half-empty d shell and the relatively large size of the Mn^{II} cation, which accommodates higher coordination numbers and has longer $M-X$ bond lengths, may account for this difference. For example, the $\text{Mn}-\text{N}$ distances in (I) (Table 1) are much longer than the average $\text{Ni}-\text{N}$ length [2.063 (3) Å] in $[\text{Ni}(\text{C}_5\text{O}_5)(2,2'\text{-bipy})_2]$ at the same temperature (*i.e.* room temperature).

The unit-cell dimensions of compound (I) at 291 K are $a = 11.3911(2)$ Å, $b = 9.4023(1)$ Å and $c = 14.4361(2)$ Å, $\beta = 104.740(1)^\circ$ and $V = 1495.26(3)$ Å³ (see supplementary material). The cell dimensions contract anisotropically with decreasing temperature, with the largest contractions being perpendicular to the direction of the rigid chain (c). Adjacent chains may be able to nestle closer to one another at lower temperature, thus increasing the strength of the intermolecular interactions.

The thermogravimetry (TG) curve (Fig. 4) of the title compound exhibits three weight-loss steps. The first weight loss begins at 366 K and the weight approaches 95.4% of the original weight at 393 K, corresponding to the loss of a solvent water molecule. The differential thermal analysis (DTA) curve gives an enthalpy change of 46.8 kJ mol^{-1} for this dehydration process, indicating the strong hydrogen bonding. The second weight loss begins at 422 K and the weight approaches 90.7% of the original weight at 503 K, which can be assigned to the release of the coordinated water molecule. In the DTA curve, the corresponding second valley suggests an enthalpy change of 43.1 kJ mol^{-1} . The strength of hydrogen bonding here is comparable with the aqua coordination of the water molecule.

In conclusion, the one-dimensional molecular fibres of (I) have been woven into a three-dimensional structure by alternating layers of strong hydrophilic-hydrophilic and lipophilic-lipophilic interactions.

Experimental

$[\text{K}_2(\text{C}_5\text{O}_5)]$ (0.032 g, 0.15 mmol) and $\text{MnCl}_2\cdot 4\text{H}_2\text{O}$ (0.044 g, 0.22 mmol) were separately dissolved in water (10 ml), and 2,2'-bipy (0.036 g, 0.23 mmol) was dissolved in ethanol (10 ml). These three solutions were then mixed. The mixture was filtered, giving a green-yellow solution. Pale-green prismatic crystals of (I) were obtained by slow evaporation from this solution at room temperature over a period of several weeks. Analysis found: C 45.97, N 7.45, H 3.05%; calculated for $[\text{Mn}(\text{C}_5\text{O}_5)(\text{C}_{10}\text{H}_8\text{N}_2)(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$: C 46.53, N 7.23, H 3.12%.

Crystal data

$[\text{Mn}(\text{C}_5\text{O}_5)(\text{C}_{10}\text{H}_8\text{N}_2)(\text{H}_2\text{O})]\cdot\text{H}_2\text{O}$	$V = 1477.84 (4) \text{ \AA}^3$
$M_r = 387.21$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 11.3363 (2) \text{ \AA}$	$\mu = 0.94 \text{ mm}^{-1}$
$b = 9.3622 (1) \text{ \AA}$	$T = 135 \text{ K}$
$c = 14.4053 (2) \text{ \AA}$	$0.26 \times 0.20 \times 0.11 \text{ mm}$
$\beta = 104.8460 (6)^\circ$	

Data collection

Bruker APEXII CCD area-detector diffractometer	33771 measured reflections
Absorption correction: multi-scan (APEX2; Bruker, 2005)	5570 independent reflections
$T_{\min} = 0.790$, $T_{\max} = 0.904$	5142 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$	275 parameters
$wR(F^2) = 0.064$	All H-atom parameters refined
$S = 0.99$	$\Delta\rho_{\text{max}} = 0.50 \text{ e \AA}^{-3}$
5570 reflections	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (Å).

Mn1—N1	2.2991 (8)	Mn1—O6	2.1484 (8)
Mn1—N2	2.2990 (7)	O1—C11	1.2605 (10)
Mn1—O1	2.2990 (6)	O2—C12	1.2574 (10)
Mn1—O2	2.3719 (7)	C13—O3 ⁱ	1.2484 (11)
Mn1—O3	2.3508 (7)	C14—O4 ⁱ	1.2428 (10)
Mn1—O4	2.3499 (7)	O5—C15	1.2360 (10)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O6—H6A \cdots O7	0.820 (17)	1.919 (18)	2.7373 (12)	175.0 (17)
O6—H6B \cdots O1 ⁱⁱ	0.779 (19)	2.137 (19)	2.9153 (10)	177.2 (18)
O7—H7A \cdots O2 ⁱⁱⁱ	0.80 (2)	2.20 (2)	2.9838 (11)	169.1 (19)
O7—H7B \cdots O5 ^{iv}	0.81 (2)	2.06 (2)	2.8755 (11)	176.8 (19)
C7—H7 \cdots O2 ^v	0.984 (15)	2.487 (15)	3.4425 (12)	163.8 (12)

Symmetry codes: (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $-x + 1, -y, -z + 2$; (iv) $-x + 1, -y + 1, -z + 2$; (v) $-x + 2, -y, -z + 2$.

All H atoms were located in difference Fourier maps, and their positions and isotropic displacement parameters were refined freely. The C—H distances are in the range 0.941 (16)–0.984 (15) Å. There are relatively large differences between the anisotropic displacements along the Mn—O bonds [diff(Mn—O)] involving the two chelating rings. These may be due to the nature of the structure itself. The dominant ionic Mn—O coordination bonds are not as strong or as directional as a typical covalent bond. The Mn^{II} ion has seven Mn—X bonds and the MnO₅N₂ coordination polyhedron is totally asymmetric. The weak and multicoordinate bonding in $M(-X)_m$ ($m > 4$) may be responsible for some deviations from the Hirshfeld rigid-bond postulate (Hirshfeld, 1976). Lutz & Spek (2009) reported that certain Zn—O(carboxylate) coordination bonds fail the Hirshfeld rigid-bond postulate, which was attributed to steric strain in the chelate ring. Similar steric strain may also be present in (I). The five

atoms in the equatorial position of the distorted MnO₅N₂ pentagonal bipyramid may be a little crowded. We note that diff(Mn1—N2) (8.7 s.u.) involving the equatorial N2 atom is larger than diff(Mn1—N1) (5.7 s.u.) involving axial atom N1.

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL97 (Sheldrick, 2008) and DIAMOND (Brandenburg, 2008); software used to prepare material for publication: WinGX (Farrugia, 1999).

This work was supported by the National Natural Science Foundation of China (grant Nos. 50673054, 50811130359 and 20972089). We thank Professor Todd B. Marder, Durham University, England, for assistance in editing the text of the manuscript.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN3137). Services for accessing these data are described at the back of the journal.

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