

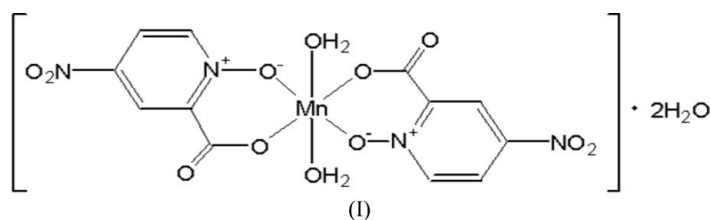
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Key indicators

Single-crystal X-ray study
 $T = 297\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.053
 wR factor = 0.132
Data-to-parameter ratio = 13.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*trans*-Diaquabis(2-carboxylato-4-nitropyridine
1-oxide- $\kappa^2\text{O}^1, \text{O}^2$)manganese(II) dihydrateIn the centrosymmetric title complex, $[\text{Mn}(\text{C}_6\text{H}_3\text{N}_2\text{O}_5)_2(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$, each Mn^{II} ion has a six-coordinate octahedral environment within an O_6 donor set. The presence of $\text{O}-\text{H} \cdots \text{O}$ hydrogen-bonding interactions links adjacent molecules into a two-dimensional array.Received 24 May 2006
Accepted 5 June 2006

Comment

Picolinato *N*-oxide complexes of Mn^{II} were first reported more than 20 years ago (Knuuttila, 1982). However, Mn^{II} complexes containing the closely related 2-carboxylato-4-nitropyridine-1-oxide ligand have not been described. We present here the crystal structure of the title compound, (I).

As shown in Fig. 1, this mononuclear and centrosymmetric complex displays a slightly distorted octahedral MnO_6 coordination geometry defined by the four donor atoms of the chelating ligands and the two aqua ligands, the latter in axial positions. As expected, the $\text{Mn}-\text{O}_{\text{water}}$ bond distances are longer than the other $\text{Mn}-\text{O}$ distances (Table 1). The observed geometric parameters are in good agreement with those observed in related six-coordinate Mn^{II} complexes (Viossat *et al.*, 2003; Shi *et al.*, 2006). The angle subtended by the O atoms at the carboxylate atom C6 of $125.6(2)^\circ$ compares well with the value of $125.15(4)^\circ$ found in the free acid (Knuuttila, 1982). Thus, coordination does not cause any noticeable opening of the carboxylate group angle.

The intermolecular $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 2) involving the coordinated O6-aqua molecule and the neighbouring carboxylate atom O3 link the molecules into one-dimensional chains. Adjacent chains are linked by hydrogen bonds between the solvent water molecules, to form a two-dimensional array (Fig. 2).

Experimental

The 2-carboxylato-4-nitropyridine-1-oxide ligand was synthesized according to the literature procedure of Li *et al.* (1987). The ligand (2 mmol) was dissolved in acetone (20 ml) and added slowly to a solution of $\text{Mn}(\text{OAc})_2$ (2 mmol) in water (20 ml). The solution was filtered after 2 h of stirring at room temperature. The filtrate was

allowed to stand at room temperature for 15 d, yielding deep-red crystals of (I).

Crystal data

$[\text{Mn}(\text{C}_6\text{H}_3\text{N}_2\text{O}_5)_2(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$	$V = 449.4 (8) \text{ \AA}^3$
$M_r = 493.21$	$Z = 1$
Triclinic, $P\bar{1}$	$D_x = 1.822 \text{ Mg m}^{-3}$
$a = 7.566 (6) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.766 (9) \text{ \AA}$	$\mu = 0.82 \text{ mm}^{-1}$
$c = 8.582 (8) \text{ \AA}$	$T = 297 (2) \text{ K}$
$\alpha = 65.57 (4)^\circ$	Prism, red
$\beta = 78.95 (4)^\circ$	$0.15 \times 0.15 \times 0.10 \text{ mm}$
$\gamma = 89.14 (4)^\circ$	

Data collection

Bruker SMART CCD diffractometer	4441 measured reflections
ω scans	2061 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1656 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.884, T_{\max} = 0.921$	$R_{\text{int}} = 0.052$
	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2 + 0.01P]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.97$	$\Delta\rho_{\text{max}} = 0.98 \text{ e \AA}^{-3}$
2061 reflections	$\Delta\rho_{\text{min}} = -0.56 \text{ e \AA}^{-3}$
158 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected geometric parameters (\AA , $^\circ$).

Mn—O1	2.121 (3)	O2—N1	1.311 (3)
Mn—O2	2.133 (2)	O3—C6	1.244 (3)
Mn—O6	2.193 (3)	C1—C6	1.528 (3)
O1—C6	1.250 (3)		
O1—Mn—O2	80.51 (8)	O2—Mn—O6	91.59 (9)
O1—Mn—O6	85.91 (13)	O2—Mn—O6 ⁱ	88.41 (9)
O1—Mn—O2 ⁱ	99.49 (8)	C6—O1—Mn	127.87 (16)
O1—Mn—O6 ⁱ	94.09 (13)	N1—O2—Mn	116.41 (13)

Symmetry code: (i) $-x + 1, -y + 1, -z$.

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O6—H6A \cdots O7	0.87 (4)	1.91 (4)	2.774 (4)	174 (3)
O6—H6B \cdots O3 ⁱⁱⁱ	0.81 (5)	2.11 (5)	2.892 (4)	163 (4)
O7—H7A \cdots O3 ⁱⁱⁱ	0.83 (5)	2.00 (5)	2.819 (4)	171 (4)

Symmetry codes: (ii) $-x + 1, -y, -z$; (iii) $x, y, z + 1$.

The water H atoms were refined freely, while those on C atoms were included in the riding-model approximation, with $C-H = 0.93 \text{ \AA}$, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: SMART (Siemens, 1999); cell refinement: SAINT; data reduction: SAINT (Sheldrick, 2000); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine

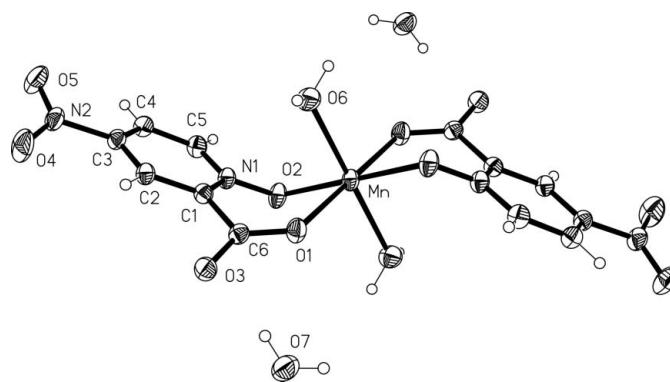


Figure 1

The molecular structure of (I), showing the atom-labelling scheme and with 30% displacement ellipsoids. Unlabelled atoms are related to labelled atoms by the symmetry operation $(1 - x, 1 - y, -z)$.

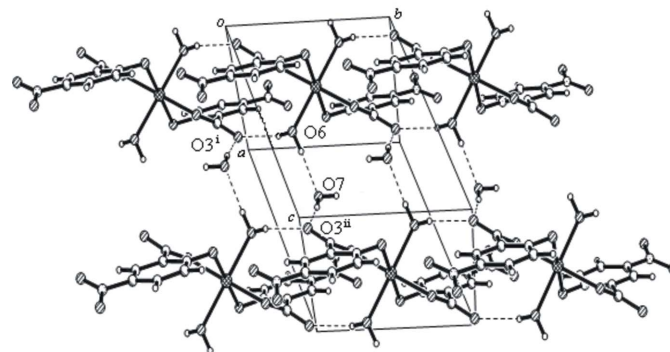


Figure 2

A packing diagram for (I), showing hydrogen bonds as dashed lines.

structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

The authors are grateful for financial support from the Research Foundation of Fujian Province, China (grant No. 2003 F006), and the National Science Foundation of Overseas Chinese Affairs Office of the State Council, China (grant No. 05QZR01).

References

- Knuutila, P. (1982). *Acta Chem. Scand. A*, **36**, 767–772.
- Li, S.-X., Liu, S.-X. & Wu, W.-S. (1987). *Chin. J. Struct. Chem.* **6**, 20–24.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Shi, F.-N., Almeida, F. A. P., Girginova, P. I., Amaral, V. S., Rocha, J., Klinowski, J. & Trindade, T. (2006). *Inorg. Chim. Acta*, **359**, 1147–1158.
- Siemens (1999). *SMART* and *SAINTE*. Version 4.050. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Viossat, V., Lemoine, P., Dayan, E., Dung, N.-H. & Viossat, B. (2003). *Polyhedron*, **22**, 1461–1470.