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Aquabis(*p*-methoxybenzoato)(1,10-phenanthroline)manganese(II) *p*-methoxybenzoic acid

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The title compound, $[Mn(C_8H_7O_3)_2(C_{12}H_8N_2)(H_2O)]\cdot C_8H_8O_3$, has been synthesized by the reaction of $MnCl_2\cdot 6H_2O$ with *p*-anisic acid and 1,10-phenanthroline in an alcohol–water solution. The Mn atom is six-coordinated by two N atoms and four O atoms, forming a distorted octahedral geometry. The Mn—O and Mn—N distances range from 2.113 (3) to 2.410 (3) Å.

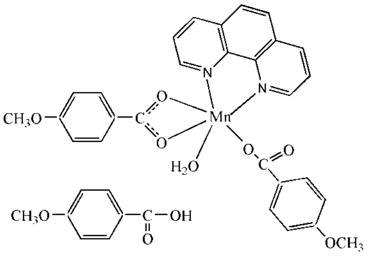
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.127
Data-to-parameter ratio = 12.6

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

Comment

In order to study the role of manganese ions and the structures of the active sites in manganese enzymes, small-molecule complexes of Mn are often used as model systems (Shi *et al.*, 2000). In recent years, Mn–phen (phen is 1,10-phenanthroline) complexes have been studied extensively as model compounds for simulating the oxygen-evolving center (OEC) of photosystem II (PSII) (Manchanda *et al.*, 1994). In this paper, we report the synthesis and structure of aquabis(*p*-methoxybenzoato)(1,10-phenanthroline)manganese(II) *p*-methoxybenzoic acid, (I) (Fig. 1).



(I)

The Mn atom in (I) is located in a very distorted octahedral environment containing two N atoms from a chelating phen ligand, three O atoms from monodentate and bidentate carboxylate groups and one O atom from a water molecule. The Mn—O bond lengths (Table 1) in (I) are in the range 2.118 (2)–2.324 (2) Å, and the Mn—N bond lengths are 2.254 (2) and 2.246 (2) Å. The phenanthroline ligand exhibits the usual short N···N bite distance [N1···N2 = 2.696 (2) Å] and acute N—Mn—N angle [N1—Mn1—N2 = 73.61 (8)°]; these are very close to those found in $[Mn(phen)_2Cl_2]$ (McCann *et al.*, 1998), $[Mn(phen)_2(NCS)_2]$ (Holleman *et al.*, 1994) and $[Mn(phen)_2(N_3)_2]$ (Shen *et al.*, 1999). The dihedral angles between the chelating phen plane and chelating carboxylate planes are 106.1 (3) and 73.9 (2)°. There exist strong O—H···O hydrogen bonds between the carboxylic acid group of *p*-methoxybenzoic acid and the water molecule (Table 2). These hydrogen bonds produce dimeric clusters (Fig. 2). The dimers interact by way of π–π stacking interactions in the sequence benz–phen–phen–benz (Fig. 3), with the neighbouring phen planes separated by 3.447 Å.

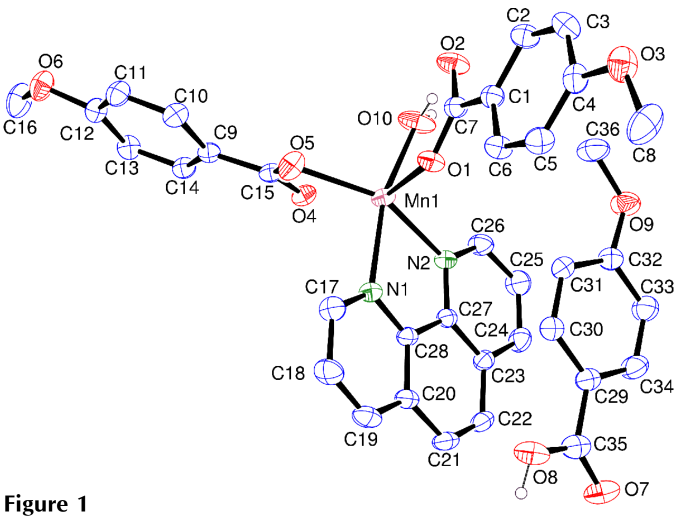


Figure 1
View of (I), showing 30% probability displacement ellipsoids. H atoms attached to C have been omitted for clarity.

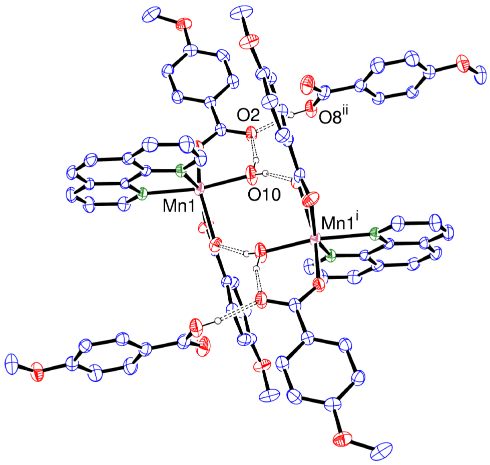


Figure 2
Hydrogen bonding (dashed lines) in the dimeric cluster of (I) (30% probability displacement ellipsoids). [Symmetry codes: (i) $2 - x, 1 - y, z$; (ii) $1 + x, y, z$.]

Experimental

The reaction of *p*-anisic acid (0.456 g, 3 mmol) and NaOH (0.12 g, 3 mmol) in a 1:1 molar ratio in a water–alcohol (3:1) solution (40 ml) at room temperature for 30 min produced a colorless solution, to which $MnCl_2 \cdot 6H_2O$ (0.198 g, 1 mmol) and 1,10-phenanthroline (0.198 g, 1 mmol) were added, yielding a yellow solution. The reaction solution was kept at room temperature after stirring for an hour. Yellow crystals were obtained after a few days; they were washed with water and ethanol several times and then dried in air.

Crystal data

$[Mn(C_8H_7O_3)_2(C_{12}H_8N_2)(H_2O)] \cdot C_8H_8O_3$
$M_r = 707.58$
Triclinic, $P\overline{1}$
$a = 10.7353$ (6) Å
$b = 11.3832$ (2) Å
$c = 16.0107$ (1) Å
$\alpha = 76.41$ (3)°
$\beta = 71.56$ (2)°
$\gamma = 62.855$ (18)°
$V = 1642.1$ (3) Å^3
$Z = 2$
$D_x = 1.431$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5737 reflections
$\theta = 2.0$–25.0°
$\mu = 0.47$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.55 × 0.40 × 0.30 mm

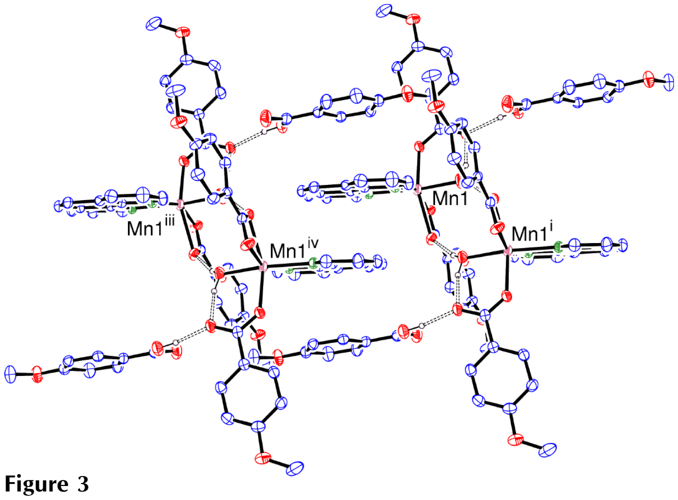


Figure 3
Diagram showing the π–π stacking interactions in (I) (30% probability displacement ellipsoids). [Symmetry codes: (i) $2 - x, 1 - y, z$; (iii) $-x, 1 - y, z$; (iv) $1 - x, 1 - y, z$.]

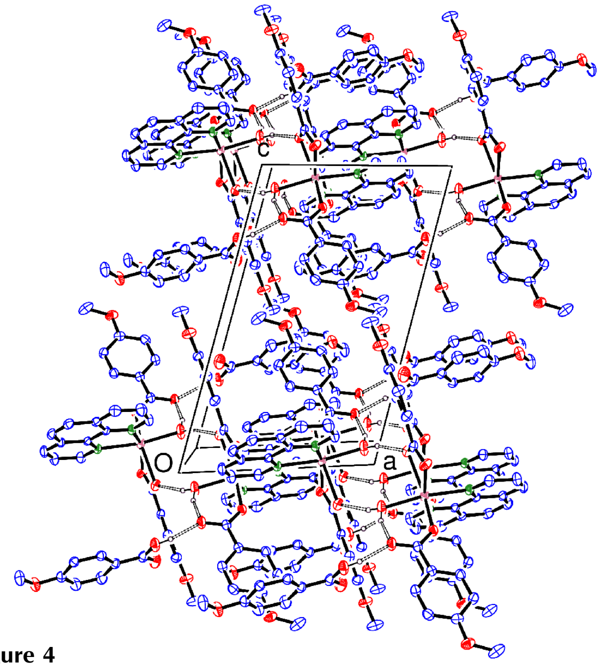


Figure 4
Packing diagram of (I), viewed down [010], showing hydrogen bonds as dashed lines (30% probability displacement ellipsoids).

Data collection

Mercury diffractometer
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
$T_{min} = 0.784$, $T_{max} = 0.873$
10 120 measured reflections
5737 independent reflections
4803 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.0°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -19 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.127$
$S = 1.10$
5737 reflections
454 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.3137P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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Table 1
Selected geometric parameters (Å).

Mn1—O1	2.118 (2)	Mn1—N2	2.246 (2)
Mn1—O10	2.156 (2)	Mn1—N1	2.254 (2)
Mn1—O5	2.181 (2)	Mn1—O4	2.324 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O10—H10$B\cdots$O2	0.84 (4)	1.85 (2)	2.649 (3)	159 (4)
O10—H10$C\cdots$O4^{i}	0.84 (4)	1.91 (2)	2.726 (3)	165 (4)
O8—H8$D\cdots$O2^{v}	0.85 (4)	1.77 (2)	2.615 (3)	171 (4)

Symmetry codes: (i) $2 - x, 1 - y, -z$; (v) $x - 1, y, z$.

Hydroxyl H atoms were located in difference maps and freely refined, while H atoms attached to C were placed in idealized positions (C—H = 0.93–0.96 Å) and refined as riding, with U_{iso} = 1.2 or $1.5U_{eq}$ of the parent atom.

Data collection: *Crystal Clear* (Rigaku, 2000); cell refinement: *Crystal Clear*; data reduction: *Crystal Clear*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

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