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Diaquabis[(*E*)-2-(4-oxopentan-2-ylideneamino)-benzoato-*κO*]dipyridinemanganese(II)

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Key indicators

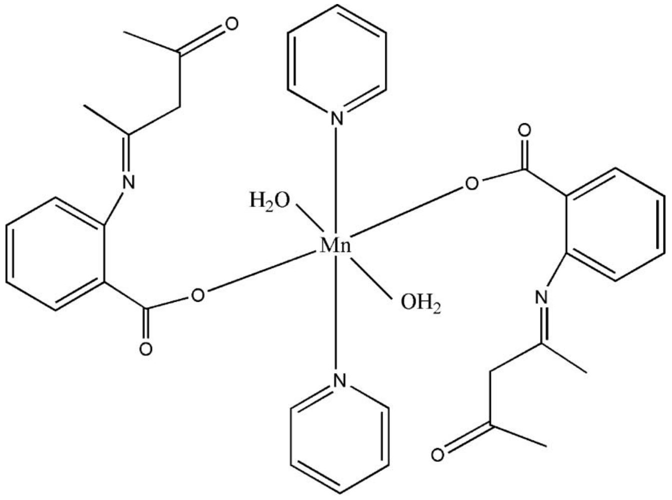
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.116
Data-to-parameter ratio = 17.4

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

The Mn atom in the title compound, $[Mn(C_{12}H_{13}NO_2)_2(C_5H_5N)_2(H_2O)_2]$, is coordinated by two pyridine heterocycles, two water molecules and an O-atom donor in two substituted benzoate groups, in an all-*trans* arrangement with the Mn atom on a center of inversion. The benzoate mean plane is inclined to the coordination plane formed by the four O atoms, with a dihedral angle of 43.4 (1)°. The dihedral angle between the planes of the benzene ring and the Schiff base is 49.5 (1)°.

Comment

The crystal structure and some properties of a substituted benzoate–manganese(II) complex were previously reported by Deng *et al.* (2006) and Xu & Xu (2004). We report here the synthesis and crystal structure of the new title complex, (I), $[Mn(C_{12}H_{13}NO_2)_2(py)_2(H_2O)_2]$, $C_{34}H_{36}MnN_4O_8$, (I), which forms a three-dimensional net structure stabilized by hydrogen-bonding interactions. In this compound, the Mn atom is six-coordinate and located on an inversion center. The coordination environment can be best described as distorted octahedral (Fig. 1). The hydroxyl O atoms of two of the substituted benzoate groups and the O atoms of two water molecules coordinate to the central Mn^{II} ion in the equatorial plane, with the two N atoms of the two pyridine molecules occupying the axial positions.



(I)

The Mn—O distances to O3 and O4 (Table 1) are shorter than those observed in the analogous compounds $[Mn(C_8H_5O_3)_2(C_3H_4N_2)_2(H_2O)_2]$ [2.2180 (10) and 2.2210 (10) Å; Deng *et al.*, 2006] and $[Mn(C_7H_4NO_4)_2$-

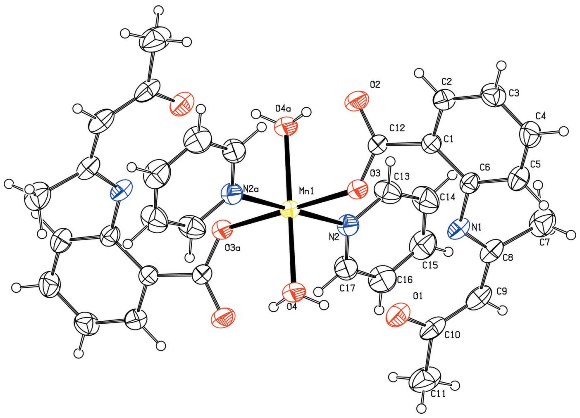


Figure 1
A view of the title mononuclear complex, (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (a) $1 - x, -y, -z$.]

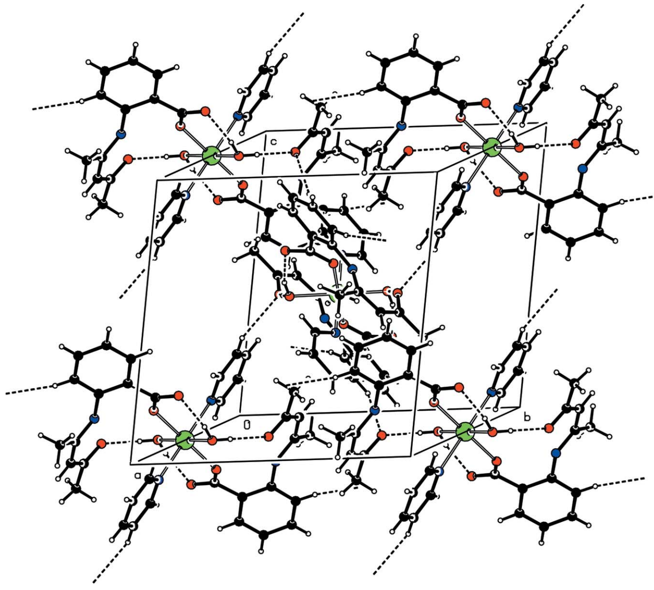


Figure 2
A view of the crystal packing of (I), showing the hydrogen bonds as dashed lines.

$(C_3H_4N_2)_2(H_2O)_2]$ [2.2180 (13) and 2.2376 (12) Å; Xu & Xu, 2004]. The Mn1—N2 distance (Table 1) is much longer than the Mn—N distance in $[Mn(C_8H_5O_3)_2(C_3H_4N_2)_2(H_2O)_2]$ [2.230 (2) Å; Deng *et al.*, 2006] and $[Mn(C_7H_4NO_4)_2(C_3H_4N_2)_2(H_2O)_2]$ [2.2127 (14) Å; Xu & Xu, 2004]. The angles, $O3^i—Mn1—O4^i$, $O3^i—Mn1—N2^i$, $O4^i—Mn1—N2^i$ and $O3^i—Mn1—O4$ [symmetry code as in Table 1], are 90.67 (5), 88.19 (5), 92.39 (6) and 89.33 (5)°, respectively. In the crystal structure, the molecules are stabilized by intramolecular and intermolecular C—H⋯O hydrogen bonds, forming a three-dimensional net structure (Table 2, Fig. 2).

Experimental

To an ethanol solution (30 ml) of *o*-aminobenzoic acid (13.7 g, 0.1 mol), acetylacetone (10 g, 0.1 mol) was slowly added with continuous stirring at reflux for 3 h. The reaction mixture was cooled to room temperature, and the Schiff base molecule, *viz*. (*E*)-2-(4-oxopentan-2-ylideneamino)benzoic acid, precipitated immediately. (*E*)-2-(4-Oxopentan-2-ylideneamino)benzoic acid (1 mmol), $Mn(OAc)_2$ (0.5 mmol), dimethylformamide (15 ml) and pyridine (1 ml) were stirred for 5 h at room temperature. The solution was filtered and allowed to stand at room temperature for three days, whereupon colorless crystals of (I) were obtained.

Crystal data

$[Mn(C_{12}H_{13}NO_2)_2(C_5H_5N)_2(H_2O)_2]$
$M_r = 683.61$
Monoclinic, $P2_1/n$
$a = 9.9611$ (7) Å
$b = 14.5346$ (10) Å
$c = 12.5321$ (8) Å
$\beta = 110.109$ (1)°
$V = 1703.8$ (2) Å^3
$Z = 2$
$D_x = 1.333$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.44$ mm^{-1}
$T = 292$ (2) K
Block, colorless
0.24 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.901$, $T_{max} = 0.957$
10543 measured reflections
3871 independent reflections
2776 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.116$
$S = 0.97$
3871 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

$Mn1—O3^i$	2.1520 (13)	$Mn1—O4^i$	2.1958 (14)
Mn1—O3	2.1521 (13)	$Mn1—N2^i$	2.3093 (15)
Mn1—O4	2.1957 (14)	Mn1—N2	2.3093 (15)
$O3^i—Mn1—O3$	180	$O4—Mn1—N2^i$	87.61 (6)
$O3^i—Mn1—O4$	89.33 (5)	$O4^i—Mn1—N2^i$	92.39 (6)
O3—Mn1—O4	90.67 (5)	$O3^i—Mn1—N2$	91.81 (5)
$O3^i—Mn1—O4^i$	90.67 (5)	O3—Mn1—N2	88.19 (5)
$O3—Mn1—O4^i$	89.33 (5)	O4—Mn1—N2	92.39 (6)
$O4—Mn1—O4^i$	180	$O4^i—Mn1—N2$	87.61 (6)
$O3^i—Mn1—N2^i$	88.19 (5)	$N2^i—Mn1—N2$	180
$O3—Mn1—N2^i$	91.81 (5)		

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

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
$O4—H4A⋯O2^i$	0.82 (2)	1.881 (12)	2.669 (2)	162 (3)
$C16—H16⋯O1^{ii}$	0.93	2.58	3.287 (3)	133
$C5—H5⋯O2^{iii}$	0.93	2.55	3.470 (3)	169
O4—H4B⋯O1	0.82 (2)	1.879 (11)	2.674 (2)	164 (3)

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

After their location in a difference map, all atoms attached to C were positioned geometrically and allowed to ride on the parent C atoms with C—H distances between 0.93 and 0.96 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for the methyl H atoms. The water H atoms were restrained to an O—H distance of 0.82 (2) Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm O})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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References

Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.

Deng, Z.-P., Gao, S. & Ng, S. W. (2006). *Acta Cryst.* E**62**, m2106–m2107.

Sheldrick, G. M. (1997). *SADABS*, *SHELXS97*, and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Xu, T.-G. & Xu, D.-J. (2004). *Acta Cryst.* E**60**, m1462–m1464.