Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

cis-Diaquabis(1,10-phenanthroline)manganese(II) 3-carboxylato-4-hydroxybenzenesulfonate tetrahydrate

Sai-Rong Fan,[a] Long-Guan Zhu,[a]* Hong-Ping Xiao[b] and Seik Weng Ng[c]

[a]Department of Chemistry, Zhejiang University, Hangzhou 310007, People's Republic of China, [b]School of Chemistry and Materials Science, Wenzhou Normal College, Wenzhou 325027, People's Republic of China, and [c]Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

Correspondence e-mail: chezlg@zju.edu.cn

Received 17 January 2005
Accepted 14 February 2005
Online 19 February 2005

The cation interacts with the anion in the title compound, $[Mn(C_{12}H_8N_2)_2(H_2O)_2](C_7H_4O_6S)\cdot 4H_2O$, through hydrogen bonding with the coordinated water molecules in a hydrogen-bonded sheet structure. The Mn atom is in an octahedral environment in which the two water molecules occupy *cis* positions.

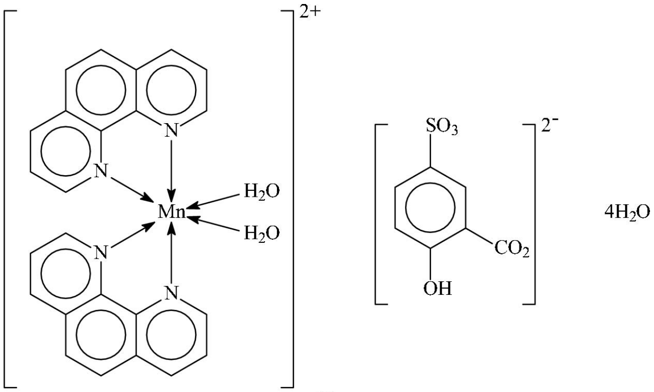
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.065
wR factor = 0.148
Data-to-parameter ratio = 11.9

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

Comment

A study on a heterocyclic complex of cobalt(II) 3-carboxylato-4-hydroxybenzenesulfonate has documented the presence of two covalent bonds between the metal and the dianion, one with the carboxylate portion and the other with the sulfonate portion (Fan *et al.*, 2005). With the manganese analog, a similar synthesis has yielded a salt in which the 3-carboxylato-4-hydroxybenzenesulfonate dianion is not directly linked to the metal atom.



(I)

The title compound, (I), exists as a tetrahydrate (Fig. 1), in which the uncoordinated water molecules interact with the cation and anion to give a sheet structure (Fig. 2). The bis-phenanthroline-chelated Mn atom shows an octahedral coordination and the two coordinated water molecules are aligned *cis* with respect to each other.

Experimental

Manganese sulfate hydrate (0.035 g, 0.2 mmol) and 5-sulfosalicylic acid dihydrate (0.051 g, 0.2 mmol) were dissolved in water (15 ml). The solution was mixed with a solution of 1,10-phenanthroline (0.040 g, 0.2 mmol) in water (4 ml) and methanol (1 ml). After 2 d, pale-yellow crystals of the hydrate separated from solution. The yield was 64% based on the metal salt.

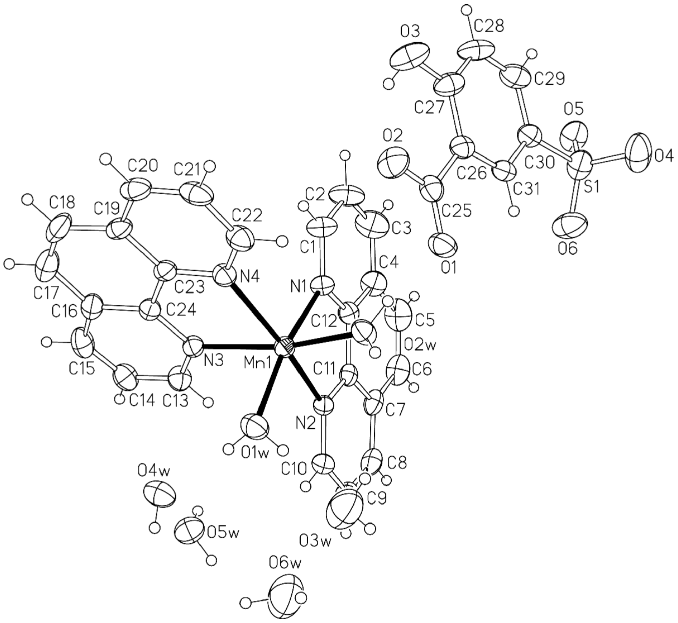


Figure 1

ORTEPII plot (Johnson, 1976) of (I). Displacement ellipsoids are drawn at the 50% probability level.

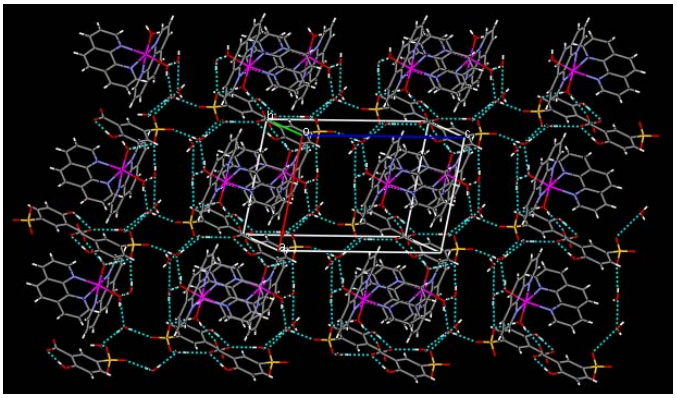


Figure 2

Packing diagram of (I), showing the sheet-like structure. Dashed lines indicate hydrogen bonds.

Crystal data

$[Mn(C_{12}H_8N_2)_2(H_2O)_2]\cdot C_7H_4O_6S\cdot 4H_2O$
$M_r = 739.61$
Triclinic, $P\overline{1}$
$a = 10.0639$ (6) Å
$b = 12.2445$ (7) Å
$c = 13.9291$ (8) Å
$\alpha = 96.985$ (1)°
$\beta = 101.406$ (1)°
$\gamma = 98.851$ (1)°
$V = 1641.90$ (17) Å^3
$Z = 2$
$D_x = 1.496$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4195 reflections
$\theta = 2.4$–24.3°
$\mu = 0.54$ mm^{-1}
$T = 295$ (2) K
Prism, pale yellow
0.33 × 0.23 × 0.19 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.859$, $T_{max} = 0.905$
11924 measured reflections
5737 independent reflections
5163 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.0°$
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.148$
$S = 1.20$
5737 reflections
481 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 1.2662P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Mn1—O2*w*	2.125 (3)	Mn1—N3	2.281 (3)
Mn1—O1*w*	2.138 (3)	Mn1—N4	2.266 (3)
Mn1—N1	2.252 (3)	S1—O4′	1.408 (7)
Mn1—N2	2.260 (3)		
O1*w*—Mn1—O2*w*	91.6 (1)	O2*w*—Mn1—N1	91.2 (1)
O1*w*—Mn1—N1	166.2 (1)	O2*w*—Mn1—N2	101.7 (1)
O1*w*—Mn1—N2	92.4 (1)	O2*w*—Mn1—N3	166.5 (1)
O1*w*—Mn1—N3	89.0 (1)	O2*w*—Mn1—N4	93.5 (1)
O1*w*—Mn1—N4	96.1 (1)		

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*o*···O2	0.85	1.78	2.512 (5)	143
O1*w*—H1*w*1···O3*w*	0.85	1.89	2.74 (1)	177
O1*w*—H1*w*2···O4*w*	0.85	1.86	2.687 (4)	166
O2*w*—H2*w*1···O1	0.85	1.76	2.601 (4)	170
O2*w*—H2*w*2···O4^{i}	0.84	1.94	2.776 (5)	175
O2*w*—H2*w*2···O4$'^{i}$	0.84	1.94	2.685 (13)	147
O3*w*—H3*w*1···O4^{i}	0.88	1.96	2.84 (1)	174
O4*w*—H4*w*1···O5^{ii}	0.85	1.94	2.791 (5)	180
O4*w*—H4*w*1···O5$'^{ii}$	0.85	2.11	2.91 (2)	156
O4*w*—H4*w*2···O5*w*	0.85	1.90	2.748 (4)	172
O5*w*—H5*w*1···O6^{iii}	0.85	2.07	2.908 (6)	168
O5*w*—H5*w*1···O4$'^{iii}$	0.85	2.02	2.78 (1)	148
O5*w*—H5*w*2···O2^{iv}	0.85	1.94	2.791 (4)	175
O6*w*—H6*w*1···O5^{ii}	0.86	2.28	3.098 (7)	160
O6*w*—H6*w*1···O5$'^{ii}$	0.86	2.20	2.89 (2)	137
O6*w*—H6*w*2···O3*w*	0.85	2.13	2.61 (1)	116

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

The sulfonate group was disordered over two positions; the occupancies refined to a 0.81 (1):0.19 (1) ratio when the S—O distances were restrained to within 0.01 Å of each other. The aromatic H atoms were positioned geometrically and were included in the refinement in the riding-model approximation [C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$], as was the hydroxyl H atom [O—H = 0.85 Å and $U_{iso}(H) = 1.2U_{eq}(O)$]. The water H atoms were placed at chemically sensible positions but were not refined [$U_{iso}(H) = 1.2U_{eq}(O)$]. Atom O3*w* was disordered over two sites, but the H atoms were placed on the major component only; for the O3*w* and O3*w*′ atoms, the displacement parameters were restrained to be nearly isotropic. When the H atoms are positioned in these sites, the shortest H···H contact exceeds 2 Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve

m564 Fan *et al.* • $[Mn(C_{12}H_8N_2)_2(H_2O)_2]\cdot C_7H_4O_6S\cdot 4H_2O$ *Acta Cryst.* (2005). E**61**, m563–m565

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

We thank the National Natural Science Foundation of China (No. 50073019) and the University of Malaya for supporting this study.

References

Bruker (2002). *SADABS, SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

Fan, S.-R., Zhu, L.-G., Xiao, H.-P. & Ng, S. W. (2005). *Acta Cryst.* E**61**, m435–m436.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

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

Acta Cryst. (2005). E**61**, m563–m565 Fan *et al.* • $[Mn(C_{12}H_8N_2)_2(H_2O)_2]\cdot C_7H_4O_6S\cdot 4H_2O$ **m565**