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Tetraaquaphenanthrolinemanganese(II) sulfate dihydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.069
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.

In the title compound, $[Mn(C_{12}H_8N_2)(H_2O)_4]SO_4\cdot 2H_2O$, the Mn^{II} atom is coordinated by two N atoms of 1,10-phenanthroline and four water O atoms in a distorted octahedral geometry. The aqua ligands are connected to the sulfate anion and water molecules of crystallization *via* O—H···O hydrogen bonds.

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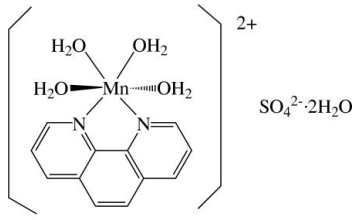
Comment

5-Fluorouracil (5-FU) possesses an antitumor activity (Heidelderger, 1957). 1,10-Phenanthroline (phen) also has biological activity, such as a sterilizing effect (Husseini, 1981). In the course of the preparation of manganese complexes with mixed ligands, we obtained the title compound, (I), which is composed of Mn^{II}, phen, water molecules and a sulfate anion. 5-FU was not incorporated in the crystal structure, but it may have had some effect on the formation of the title mononuclear Mn^{II} complex.



(I)

In (I), the Mn atom is coordinated by two N atoms from a phen ligand and four O atoms of the aqua ligands in a distorted octahedral geometry (Fig. 1 and Table 1). Atoms N1/

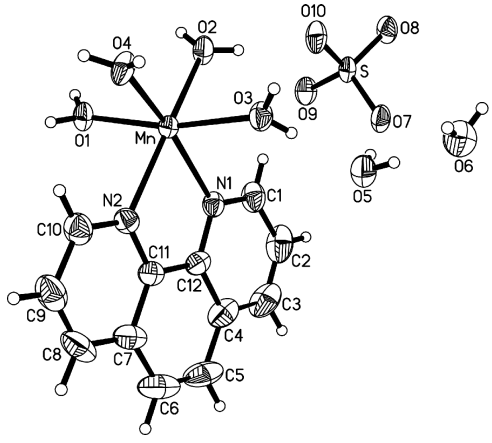


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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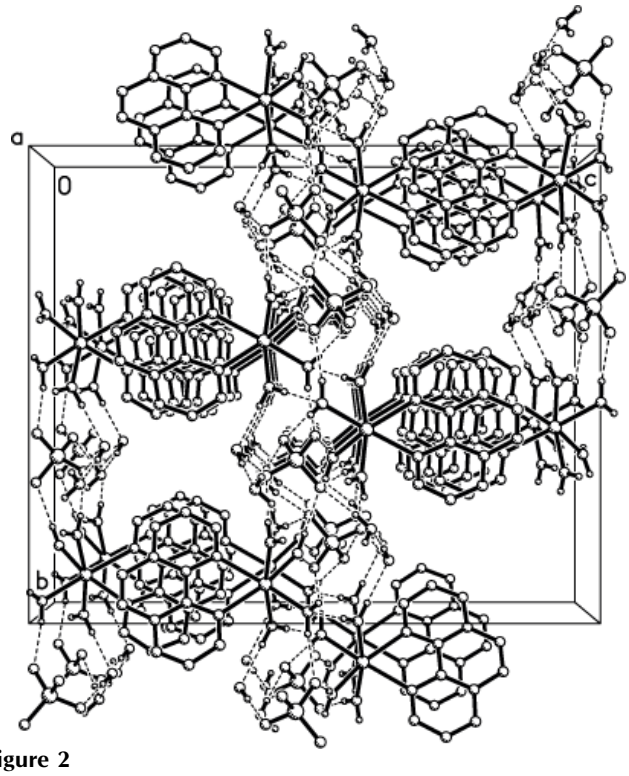

Figure 2
The crystal structure of (I). Dashed lines indicate hydrogen bonds.

C12/C11/N2 are coplanar (plane 1), with atom Mn deviating from the plane by 0.0126 (8) Å. The dihedral angles between plane 1 and the planes of the pyridine rings are 2.77 (13) and 1.97 (12)° for N1/C1–C4/C12 and N2/C7–C11, respectively. A sulfate anion and the two water molecules of crystallization are involved in the formation of intermolecular O—H···O hydrogen bonds with aqua ligands (Fig. 2 and Table 2).

Experimental

The title compound, (I), was prepared by reacting $MnSO_4·H_2O$ with 1,10-phenanthroline and 5-FU (1:1:1) in ethanol (pH = 4). Single crystals of (I) suitable for X-ray study were obtained by slow evaporation of an aqueous ethanol solution at 293 K for a month.

Crystal data

$[Mn(C_{12}H_8N_2)(H_2O)_4]SO_4·2H_2O$
M_r = 439.30
Orthorhombic, *Pbca*
a = 8.877 (1) Å
b = 18.508 (3) Å
c = 22.098 (3) Å
V = 3630.6 (9) Å^3
Z = 8
D_x = 1.607 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 31 reflections
θ = 4.1–13.6°
μ = 0.90 mm^{-1}
T = 295 (2) K
Prism, yellow
0.56 × 0.44 × 0.40 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.608, T_{max} = 0.699
4394 measured reflections
3568 independent reflections
2501 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.0°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 22$
$l = -1 \rightarrow 27$
3 standard reflections
every 97 reflections
intensity decay: 7.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.069
S = 0.89
3568 reflections
284 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0076 (3)

Table 1
Selected geometric parameters (Å, °).

Mn—O4	2.1530 (16)	Mn—O2	2.2018 (17)
Mn—O1	2.1580 (16)	Mn—N1	2.2513 (19)
Mn—O3	2.1829 (17)	Mn—N2	2.2747 (19)
O4—Mn—O1	87.56 (7)	O3—Mn—N1	86.76 (7)
O4—Mn—O3	86.83 (7)	O2—Mn—N1	98.39 (7)
O1—Mn—O3	168.33 (7)	O4—Mn—N2	91.60 (8)
O4—Mn—O2	98.01 (8)	O1—Mn—N2	87.22 (7)
O1—Mn—O2	86.40 (7)	O3—Mn—N2	103.16 (7)
O3—Mn—O2	84.25 (7)	O2—Mn—N2	168.22 (7)
O4—Mn—N1	161.69 (8)	N1—Mn—N2	73.22 (7)
O1—Mn—N1	101.53 (7)		
N2—C11—C12—N1	−2.9 (3)	C7—C11—C12—C4	−1.9 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O7^{i}	0.818 (9)	1.860 (11)	2.671 (2)	171 (2)
O1—H1B···O8^{ii}	0.823 (9)	1.958 (11)	2.775 (2)	172 (3)
O2—H2B···O9^{ii}	0.829 (9)	1.873 (10)	2.691 (2)	169 (3)
O2—H2A···O10	0.826 (10)	1.866 (12)	2.684 (2)	171 (4)
O3—H3A···O5	0.813 (9)	1.870 (11)	2.679 (3)	173 (3)
O3—H3B···O2^{iii}	0.814 (9)	2.187 (10)	2.999 (3)	175 (2)
O4—H4B···O8^{i}	0.815 (9)	1.985 (9)	2.793 (2)	171 (2)
O4—H4A···O10^{iii}	0.823 (9)	1.924 (11)	2.740 (2)	172 (3)
O5—H5A···O6	0.822 (9)	1.958 (10)	2.771 (3)	170 (2)
O5—H5B···O7	0.827 (9)	2.021 (9)	2.847 (2)	176 (2)
O6—H6B···O8^{iv}	0.818 (10)	2.080 (16)	2.852 (3)	157 (4)
O6—H6A···O9^{v}	0.816 (10)	2.090 (10)	2.905 (3)	177 (3)

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

The water H atoms were located in difference Fourier syntheses and refined isotropically. The H atoms of 1,10-phenanthroline were placed in geometrically calculated positions, with C—H distances of 0.93 Å and U_{iso}(H) values of 1.2U_{eq}(C).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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