Crystal structure of triaqua-(1,10-phenanthroline-*N*,*N*')sulfato-manganese(II), $Mn(C_{12}H_8N_2)(H_2O)_3SO_4$

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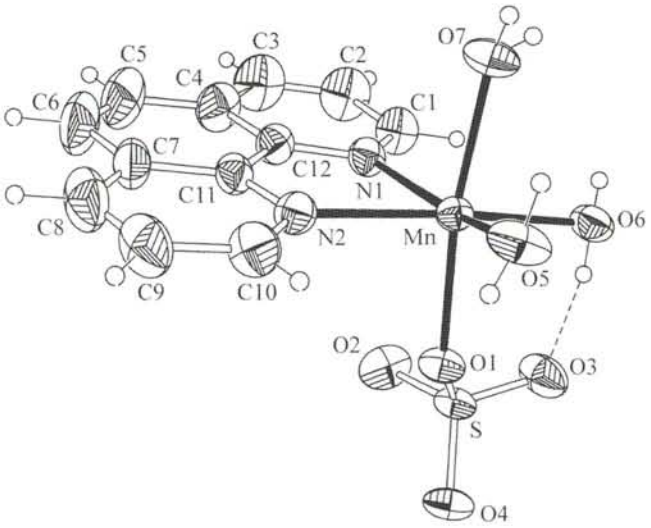

Abstract

$C_{12}H_{14}MnN_2O_7S$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 8.081(1)$ Å, $b = 9.689(1)$ Å, $c = 19.338(2)$ Å, $V = 1514.1$ Å^3, $Z = 4$, $R_{gt}(F) = 0.031$, $wR_{ref}(F^2) = 0.078$, $T = 293$ K.

Source of material

0.225 g (1.33 mmol) $MnSO_4 \cdot H_2O$, 0.100 g (1.33 mmol) H_2NCH_2COOH, 0.264 g (1.33) 1,10-phenanthroline monohydrate were dissolved in 20 ml CH_3OH-H_2O (1:1 v/v) under stirring. After few minutes, a yellowish precipitate was formed. By slow evaporationof the filtrate, pale redish plate-like crystals grew.

Discussion

$[Mn(C_{12}H_8N_2)(H_2O)_3SO_4]$ complex molecules are interlinked into a 3D network by hydrogen bonds. Each Mn atom is octahedrally coordinated by two N atoms of the bidentate chelating phenanthroline ligand and by four O atoms of one sulfate group and three water molecules with d(Mn—N) = 2.263(3) Å – 2.266(3) Å and d(Mn—O) = 2.163(3) Å – 2.202(2) Å. The principal O–Mn–N and O–Mn–O axes with angles of 161.6(1)° – 170.0(1)° indicate strong distortion of the octahedral coordination around the manganese atom. The S—O bond length for the coordinated O1 atom is 1.496(2) Å and slightly longer than the others averaged at 1.463(1) Å. The plane of the phenanthroline ligand is slightly distorted with a maximal deviation of 0.049(3) Å. One coordinating water molecule forms an intramolecular hydrogen bond to sulfate O3 with d(O···O) = 2.698 Å and ∠(O–H···O) = 168° and an intermolecular hydrogen bond to the sulfate O1 of an adjacent complex molecule. The remaining water molecules form intermolecular hydrogen bonds to three neighboring complex molecules with d(O···O) = 2.652 Å – 2.784 Å and ∠(O–H···O) = 167° – 176°.

Table 1. Data collection and handling.

Crystal:	pale redish, thin plate, size 0.18 × 0.22 × 0.47 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.48 cm^{-1}
Diffractometer, scan mode:	Bruker P4, θ/2θ
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2666, 2477
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2240
$N(param)_{refined}$:	210
Programs:	SHELXS-97 [1], SHELXL-97 [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.5076	1.2750	0.1668	0.065(5)
H(2)	4*a*	0.5801	1.4300	0.0812	0.065
H(3)	4*a*	0.5773	1.3593	–0.0325	0.065
H(5)	4*a*	0.5282	1.1691	–0.1194	0.065
H(6)	4*a*	0.4521	0.9522	–0.1428	0.065
H(8)	4*a*	0.3558	0.7178	–0.0996	0.065
H(9)	4*a*	0.2882	0.5780	–0.0072	0.065
H(10)	4*a*	0.2843	0.6694	0.1027	0.065
HA(O5)	4*a*	0.0942	0.8141	0.2306	0.05
HB(O5)	4*a*	0.2270	0.7064	0.2217	0.05
HA(O6)	4*a*	0.5077	1.0633	0.2881	0.05
HB(O6)	4*a*	0.3949	1.1680	0.2782	0.05
HA(O7)	4*a*	0.0242	1.0615	0.1781	0.05
HB(O7)	4*a*	0.1276	1.1532	0.1979	0.05

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mn	4*a*	0.35914(6)	0.96043(5)	0.18412(2)	0.0228(2)	0.0252(2)	0.0277(2)	–0.0003(2)	0.0002(2)	0.0023(2)
S	4*a*	0.75540(9)	0.92740(7)	0.22794(4)	0.0203(3)	0.0213(3)	0.0422(4)	–0.0005(3)	–0.0046(3)	–0.0030(3)
N(1)	4*a*	0.4580(4)	1.1178(3)	0.1079(1)	0.030(1)	0.034(2)	0.031(1)	–0.002(1)	0.005(1)	0.002(1)
N(2)	4*a*	0.3556(4)	0.8555(3)	0.0796(1)	0.034(1)	0.034(1)	0.035(1)	–0.001(2)	–0.003(1)	–0.003(1)
C(1)	4*a*	0.5046(5)	1.2461(4)	0.1210(2)	0.047(2)	0.039(2)	0.044(2)	–0.010(2)	0.003(2)	0.002(2)
C(2)	4*a*	0.5494(6)	1.3404(5)	0.0697(2)	0.065(3)	0.041(2)	0.063(3)	–0.019(2)	0.006(2)	0.010(2)
C(3)	4*a*	0.5474(7)	1.2982(5)	0.0024(2)	0.072(3)	0.053(3)	0.056(2)	–0.018(3)	0.015(2)	0.020(2)
C(4)	4*a*	0.5003(6)	1.1632(5)	–0.0142(2)	0.049(2)	0.056(2)	0.040(2)	–0.006(2)	0.010(2)	0.013(2)
C(5)	4*a*	0.4973(7)	1.1113(6)	–0.0833(2)	0.081(4)	0.088(4)	0.033(2)	–0.008(3)	0.011(2)	0.015(2)
C(6)	4*a*	0.4516(7)	0.9828(6)	–0.0972(2)	0.075(3)	0.094(4)	0.028(2)	–0.003(3)	0.010(2)	–0.001(2)
C(7)	4*a*	0.4011(5)	0.8898(5)	–0.0431(2)	0.045(2)	0.065(3)	0.031(2)	0.004(2)	0.001(2)	–0.010(2)
C(8)	4*a*	0.3570(7)	0.7528(5)	–0.0548(2)	0.057(3)	0.073(3)	0.051(2)	–0.005(3)	–0.004(3)	–0.030(2)
C(9)	4*a*	0.3156(6)	0.6702(5)	–0.0002(2)	0.059(3)	0.050(2)	0.065(3)	–0.007(2)	–0.005(2)	–0.025(2)
C(10)	4*a*	0.3149(5)	0.7260(4)	0.0660(2)	0.049(2)	0.038(2)	0.049(2)	–0.008(2)	–0.004(2)	–0.007(2)
C(11)	4*a*	0.4023(4)	0.9370(4)	0.0261(2)	0.027(2)	0.041(2)	0.030(2)	–0.000(2)	0.002(1)	–0.004(2)
C(12)	4*a*	0.4546(4)	1.0771(4)	0.0406(2)	0.029(2)	0.041(2)	0.031(2)	–0.002(2)	0.004(1)	0.003(2)
O(1)	4*a*	0.5942(3)	0.8619(2)	0.2088(1)	0.019(1)	0.026(1)	0.045(1)	–0.0027(9)	–0.003(1)	–0.001(1)
O(2)	4*a*	0.8182(3)	1.0047(3)	0.1687(2)	0.033(1)	0.052(2)	0.060(2)	–0.012(1)	–0.002(1)	0.017(1)
O(3)	4*a*	0.7298(3)	1.0211(3)	0.2863(2)	0.042(1)	0.049(1)	0.059(2)	0.009(1)	–0.012(1)	–0.025(1)
O(4)	4*a*	0.8712(3)	0.8170(2)	0.2463(1)	0.024(1)	0.030(1)	0.059(2)	0.005(1)	–0.003(1)	0.004(1)
O(5)	4*a*	0.2088(3)	0.8003(3)	0.2302(2)	0.028(1)	0.029(1)	0.076(2)	0.001(1)	0.012(1)	0.012(1)
O(6)	4*a*	0.4025(3)	1.0753(2)	0.2807(1)	0.036(1)	0.031(1)	0.043(1)	0.009(1)	–0.001(1)	–0.005(1)
O(7)	4*a*	0.1330(3)	1.0809(2)	0.1753(1)	0.024(1)	0.030(1)	0.066(2)	0.001(1)	–0.000(1)	–0.001(1)

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References

1. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Larger Structures. Acta Crystallogr. A**46** (1990) 467-473.
2. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.