metal-organic papers

Diaquabis(2,3-dimethylpyrazine 1,4-dioxide-κO)-bis(thiocyanato-κN)manganese(II) dihydrate

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Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.112
Data-to-parameter ratio = 17.2

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

In red crystal of the title compound, [Mn(NCS)$_2$(C$_6$H$_8$-N$_2$O$_2$)$_2$(H$_2$O)$_2$]·2H$_2$O, the Mn^{II} atom lies on an inversion centre and assumes a distorted octahedral coordination geometry. Hydrogen bonds consolidate the crystal structure.

Comment

Pyrazine 1,4-dioxide and its derivatives are useful ligands for the preparation of metal complexes (Sun *et al.*, 2001; Shi *et al.*, 2005). In order to understand the effect of metallic ions on the coordination modes, we have prepared the title Mn^{II} complex, (I), and report its structure here.

(I)
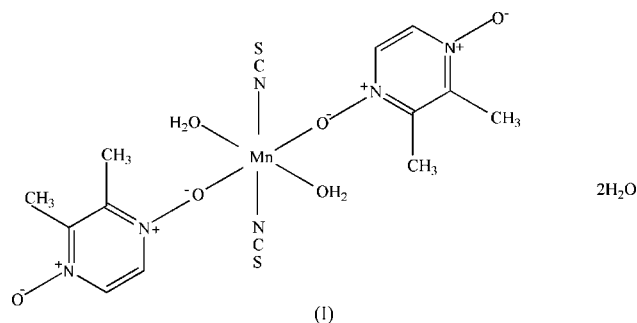
The molecular structure of (I) is shown in Fig. 1. The Mn^{II} atom is located on a inversion centre and assumes a distorted octahedral coordination geometry (Table 1), formed by 2,3-dimethylpyrazine 1,4-dioxide ligands, coordinated water molecules and isothiocyanate anions.

The hydrogen-bonding network, which consolidates the crystal structure, occurs between coordinated water molecules, uncoordinated water molecules and pyrazine 1,4-dioxide ligands (Table 2).

Experimental

An aqueous solution (10 ml) of Mn(ClO$_4$)$_2$·6H$_2$O (0.16 g, 0.44 mmol) was added to an aqueous solution (15 ml) of 2,3-dimethylpyrazine 1,4-dioxide (0.12 g, 0.86 mmol) and NaNCS (0.070 g, 0.86 mmol). The resulting solution was stirred for a few minutes. Red single crystals of (I) were obtained after three weeks.

Crystal data

[Mn(NCS)$_2$(C$_6$H$_8$N$_2$O$_2$)$_2$-(H$_2$O)$_2$]·2H$_2$O	$D_x = 1.533$ Mg m^{-3}
$M_r = 523.47$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1647
$a = 11.766$ (3) Å	reflections
$b = 6.7824$ (19) Å	$\theta = 2.5$–24.3°
$c = 14.429$ (4) Å	$\mu = 0.82$ mm^{-1}
$\beta = 100.099$ (4)°	$T = 298$ (2) K
$V = 1133.6$ (5) Å^3	Prism, red
$Z = 2$	0.25 × 0.12 × 0.08 mm

Data collection

Bruker SMART APEX CCD diffractometer	2470 independent reflections
φ and ω scans	1998 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.033$
($SADABS$; Sheldrick, 1996)	$\theta_{max} = 27.1°$
$T_{min} = 0.822$, $T_{max} = 0.937$	$h = -15 \rightarrow 11$
6562 measured reflections	$k = -8 \rightarrow 8$
	$l = -18 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$+ 0.0694P]$
$wR(F^2) = 0.112$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
2470 reflections	$\Delta\rho_{max} = 0.44$ e Å^{-3}
144 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Mn1—O1	2.2280 (16)	O1—N1	1.325 (2)
Mn1—O3	2.1736 (18)	O2—N2	1.315 (2)
Mn1—N3	2.167 (2)		
N1—O1—Mn1	120.19 (13)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H3$\cdots$O2^i	0.87	1.88	2.735 (2)	167
O3—H4$\cdots$O4^{ii}	0.82	1.87	2.693 (3)	176
O4—H1$\cdots$O1^{iii}	0.88	2.04	2.877 (3)	157
O4—H2$\cdots$O2^{iv}	0.89	1.96	2.843 (3)	174

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

H atoms of water molecules were located in a difference Fourier map and refined as riding in their as-found positions, with $U_{iso}(H) = 1.5U_{eq}(O)$. Methyl H atoms were placed in calculated positions with
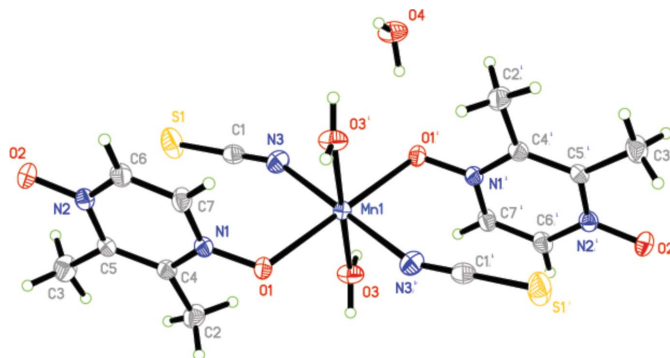

Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (i) $1 - x$, $1 - y$, $1 - z$].

C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed in calculated positions, with C—H = 0.93 Å and refined as riding, with $U_{iso}(H) = 1.2_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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