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Bis(4,6-dimethyl-2-pyrimidinethiolato-κ^2N,S)bis(methanol-κO)manganese(II)

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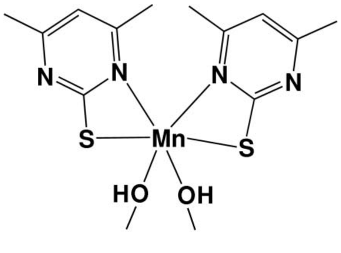
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.071; data-to-parameter ratio = 14.8.

In the title molecular compound, $[\mathrm{Mn}(\mathrm{C_6H_7N_2S})_2(\mathrm{CH_4O})_2]$, the Mn atom is octahedrally coordinated by two N atoms and two S atoms from two 4,6-dimethyl-2-pyrimidinethiolate anions and two O atoms from two methanol molecules. The molecule lies on a twofold rotation axis. The methanol molecule engages in hydrogen bonding with the N atom of an adjacent molecule to form a chain that runs along the c axis.

Related literature

For the chemistry, properties, and medicinal and biological relevance of metal thiolates, see Das & Seth (1997); Halcrow & Christou (1994); Tzeng *et al.* (1999); Zhao *et al.* (2001). For structural studies of metal thiolates, see Castro *et al.* (1995); Constable *et al.* (1991); Costes *et al.* (2004); Cotton & Ilsley (1981); Eichhöfer & Buth (2005); Hadjikakou *et al.* (2005); Han *et al.* (2004); Lang *et al.* (2003); MacGillvray *et al.* (2000); Mikuriya *et al.* (1991); Rodríguez *et al.* (2003); Stephens (1977); Zhang *et al.* (2003).



Experimental

Crystal data

$[\mathrm{Mn}(\mathrm{C_6H_7N_2S})_2(\mathrm{CH_4O})_2]$
M_r = 397.44
Monoclinic, $C2/c$
a = 15.150 (3) Å
b = 8.6713 (17) Å
c = 13.890 (3) Å
β = 99.31 (3)°
V = 1800.7 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.98 mm^{-1}
T = 153 (2) K
0.35 × 0.25 × 0.20 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min}$ = 0.726, $T_{\max}$ = 0.828
8481 measured reflections
1656 independent reflections
1565 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.071
S = 1.09
1656 reflections
112 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.28 e Å^{-3}
$\Delta\rho_{\min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1$^{\mathrm{i}}$	0.800 (16)	1.916 (17)	2.716 (2)	178 (3)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2320).

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supplementary materials

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Bis(4,6-dimethyl-2-pyrimidinethiolato-*κ*^{2}*N*,*S*)bis(methanol-*κO*)manganese(II)

H.-Y. Li, H.-X. Li, X.-Y. Tang, Y. Chen and J.-P. Lang

Comment

Over past decades considerable attention has been paid to the synthesis and characterization of metal complexes of heterocyclic thiones due to their aesthetically interesting structures (Zhao *et al.*, 2001), their luminescence properties (Tzeng *et al.*, 1999), medicinal applications (Das & Seth, 1997) and their potential relevance to active sites in metalloenzymes (Halcrow & Christou, 1994). Among the heterocyclic thiols, pyrimidinethiols, are some of the most versatile sulfur donor ligands as they can act as monodentate, chelating and bridging ligands to yield complexes with variable nuclearity and a wide range of structural geometries (Rodríguez *et al.*, 2003). A lot of mononuclear pyrimidine-2-thiolate complexes of Hg, Sb, Mo (Cotton & Ilsley, 1981; Hadjikakou *et al.*, 2005), oligomeric complexes of Cu, Ag, Au (Han *et al.*, 2004; Zhang *et al.*, 2003) and polmeric structures of Zn(II), Cd(II), Ni(II) (Eichhöfer & Buth, 2005; Lang *et al.*, 2003) have been reported. However, the chemistry of manganese derivatives of this type of ligands, remains undeveloped. The synthesis and structural characterization of only a few manganese complexes such as *trans*-{dichlorotetraquis-[2(lH)-pyridinethione]}manganese(II) (Constable *et al.*, 1991) and [Mn(dmpymt)$_2$phen] [phen is 1,10-phenanthroline; Castro *et al.*, 1995] were described. We report here the crystal structure of the title complex Mn(dmpymt)$_2$(MeOH)$_2$ (I).

An X-ray analysis revealed that complex (I) crystallizes in the monclinic space group *C*2/*c* and the asymmetric unit contains one-half of the discrete molecule Mn(dmpymt)$_2$(MeOH)$_2$. In the structure of (I), the Mn atom is coordinated by two S atoms and two N atoms to form a distorted octahedral geometry (Figure 1). The environment around the manganese center is [MnN$_4$S$_2$], with the sulfur atoms mutually *trans*. Each dmpymt ligand coordinates to one Mn atom in an N,*S*-bidentate fashion, forming a heteroatomic four-membered chelate ring. The chelate bite angle S(1)—Mn(1)—N(2) [64.42 (4)°] is comparable to those found in Mn(phen)(dmpymt)$_2$ [64.6 (2)° and 64.3 (2)°; Castro *et al.*, 1995]. The S(1)—Mn(1)—S(1^i), O(1)—Mn(1)—N(2^i) and O(1^i)-Mn(1)—N(2) [i = $-x$, *y*, $-z + 3/2$] bond angles are 165.47 (3)°, 161.78 (6)° and 161.78 (6)°, respectively. The Mn—S bond distance of 2.6165 (8) Å is longer than the corresponding ones found in Mn(phen)(dmpymt)$_2$ [2.593 (3) Å; Castro *et al.*, 1995], [MnLCl(MeOH)] [2.559 (3) Å; *L* is 2-[2-(2pyridil)ethylamino]ethanethiolate; Mikuriya *et al.*, 1991], but shorter than that observed in *trans*-{dichlorotetraquis-[2(lH)-pyridinethione]}manganese(II) [2.84 (1) Å and 2.656 (1) Å; Constable *et al.*, 1991]. The Mn—N bond length [2.2706 (17) Å] of this title complex is slightly longer than that observed in Mn(phen)(dmpymt)$_2$ [2.258 (2) Å; Castro *et al.*, 1995]. The Mn—O bond distance [2.1951 (16) Å] is shorter than that reported in MnL(CH$_3$OH) [2.277 (3) Å; *L* is *N*-(2-hydroxybenzoyl)-*N*'-(2-hydroxybenzylidene)propane-1,2-diamnine; Costes *et al.*, 2004]. The Mn—N bond distance is 2.2706 (17) Å, which is shorter than that in observed [Mn(acac)$_2$phen] [2.307 (5) Å; acac is 2,4-pentanedionato; Stephens, 1977].

In the crystal of (I), the OH group of each MeOH molecule interacts with atom N(1) in an adjacent molecule to afford pairwise intermolecular C—H···N contacts, thereby forming chains of molecules running along *c* axis (Figure 2 and Table 2).

Experimental

A solution of dmpymtH (0.2861 g, 20 mmol) in MeOH (5 ml) was added to MnAc·$2H_2O$ (0.2455 g, 10 mmol) in MeOH (10 ml). A yellow precipitate was observed to form within an hour. The mixture was stirred at room temperature for ten hours and then filtered. The resulting solid was redissolved in MeOH and CH_2Cl_2 (V: V = 1: 1) and filtered again. Diethyl ether was layered onto the filtrate to form yellow crystals of compound (I) in several days, which was collected by filtration, washed by Et_2O, and dried *in vacuo*. Yield 0.353 g, 89% (based on Mn). Analysis found: C 42.47, H 5.22, N 14.19%; calculated for $C_{14}H_{22}N_4O_2S_2Mn$: C 42.31, H 5.58, N 14.10%.

Refinement

The H atom of the MeOH group was located in a Fourier map, and the O—H distance was fixed at 0.800 (16) Å. All other hydrogen atoms were placed in geometrically idealized positions (C—H = 0.98 Å for methyl groups, and C—H = 0.95 Å for ring CH groups) and constrained to ride on their parent atoms with $U_{iso}(H) = 1.5U_{eq}$ for methyl group and $U_{iso}(H) = 1.2U_{eq}$ for ring CH groups.

Figures

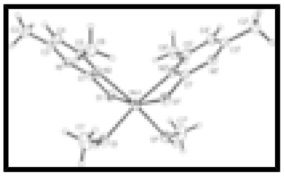

Fig. 1. A perspective view of (I), showing the atom-labling scheme. Displacement elipsoids are drawn at the 30% probability level and H atoms are drawn as spheres of arbitrary radii. [Symmetry code:(i) $-x$, y, $-z + 3/2$.]

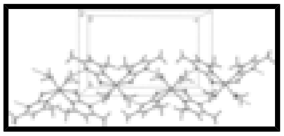

Fig. 2. A packing diagram for (I) viewed down the *c* axis. Dashed lines represent the O—H···N hydrogen bonding interaction.

Bis(4,6-dimethyl-2-pyrimidinethiolato-κ^2*N*,*S*)-\ bis(methanol-κ*O*)manganese(II)

Crystal data

$[Mn(C_6H_7N_2S)_2(CH_4O)_2]$	$F_{000} = 828$
$M_r = 397.44$	$D_x = 1.466$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 3136 reflections
a = 15.150 (3) Å	θ = 3.2–25.4°
b = 8.6713 (17) Å	μ = 0.98 mm^{-1}
c = 13.890 (3) Å	*T* = 153 (2) K
β = 99.31 (3)°	Block, yellow
V = 1800.7 (6) Å^3	0.35 × 0.25 × 0.20 mm
Z = 4	

Data collection

Rigaku Mercury diffractometer	1656 independent reflections
Radiation source: fine-focus sealed tube	1565 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.026$
$T = 153(2)$ K	$\theta_{\text{max}} = 25.3^\circ$
ω scans	$\theta_{\text{min}} = 3.2^\circ$
Absorption correction: multi-scan (Jacobson, 1998)	$h = -18 \rightarrow 18$
$T_{\text{min}} = 0.726$, $T_{\text{max}} = 0.828$	$k = -10 \rightarrow 8$
8481 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 3.1898P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1656 reflections	$\Delta\rho_{\text{max}} = 0.28$ e Å^{-3}
112 parameters	$\Delta\rho_{\text{min}} = -0.25$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Mn1	0.0000	0.03331 (5)	0.7500	0.01898 (14)
S1	0.10027 (3)	0.07146 (6)	0.61515 (4)	0.02198 (15)
O1	−0.08155 (9)	−0.14465 (17)	0.66532 (10)	0.0228 (3)

H1	−0.0602 (16)	−0.181 (3)	0.6212 (15)	0.037 (8)*
N1	0.00580 (11)	0.25958 (19)	0.48392 (12)	0.0204 (4)
N2	−0.05222 (11)	0.20439 (19)	0.63054 (11)	0.0190 (4)
C1	0.01101 (13)	0.1891 (2)	0.57136 (14)	0.0184 (4)
C2	−0.07078 (16)	0.4251 (3)	0.35684 (16)	0.0302 (5)
H2A	−0.0124	0.4683	0.3497	0.045*
H2B	−0.1155	0.5077	0.3503	0.045*
H2C	−0.0883	0.3474	0.3061	0.045*
C3	−0.06489 (14)	0.3517 (2)	0.45536 (14)	0.0229 (5)
C4	−0.12950 (14)	0.3754 (3)	0.51330 (15)	0.0256 (5)
H4	−0.1780	0.4437	0.4933	0.031*
C5	−0.12206 (13)	0.2972 (2)	0.60133 (14)	0.0220 (4)
C6	−0.19153 (14)	0.3138 (3)	0.66618 (16)	0.0290 (5)
H6A	−0.2458	0.2584	0.6375	0.043*
H6B	−0.2055	0.4233	0.6729	0.043*
H6C	−0.1687	0.2707	0.7306	0.043*
C7	−0.17601 (15)	−0.1366 (3)	0.63694 (17)	0.0381 (6)
H7A	−0.2017	−0.0735	0.6839	0.057*
H7B	−0.2013	−0.2408	0.6354	0.057*
H7C	−0.1900	−0.0902	0.5720	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0182 (2)	0.0232 (2)	0.0156 (2)	0.000	0.00279 (16)	0.000
S1	0.0182 (3)	0.0266 (3)	0.0213 (3)	0.0034 (2)	0.0036 (2)	0.0024 (2)
O1	0.0206 (8)	0.0267 (8)	0.0212 (8)	−0.0014 (6)	0.0044 (6)	−0.0027 (6)
N1	0.0233 (9)	0.0183 (8)	0.0193 (8)	−0.0012 (7)	0.0023 (7)	−0.0008 (7)
N2	0.0180 (8)	0.0201 (9)	0.0187 (8)	−0.0007 (7)	0.0020 (7)	−0.0010 (7)
C1	0.0194 (10)	0.0166 (10)	0.0183 (9)	−0.0028 (8)	0.0009 (8)	−0.0028 (8)
C2	0.0402 (14)	0.0260 (12)	0.0227 (11)	0.0035 (10)	−0.0003 (10)	0.0044 (9)
C3	0.0298 (11)	0.0175 (10)	0.0193 (10)	−0.0016 (9)	−0.0019 (8)	−0.0012 (8)
C4	0.0264 (11)	0.0249 (11)	0.0237 (11)	0.0071 (9)	−0.0018 (9)	−0.0010 (9)
C5	0.0207 (10)	0.0233 (11)	0.0207 (10)	0.0003 (8)	−0.0002 (8)	−0.0053 (8)
C6	0.0222 (11)	0.0376 (13)	0.0267 (11)	0.0058 (10)	0.0029 (9)	−0.0044 (10)
C7	0.0207 (12)	0.0606 (17)	0.0329 (13)	−0.0064 (11)	0.0041 (10)	−0.0050 (12)

Geometric parameters (Å, °)

Mn1—O1	2.1957 (15)	C2—H2A	0.9800
Mn1—O1^{i}	2.1957 (15)	C2—H2B	0.9800
Mn1—N2^{i}	2.2706 (17)	C2—H2C	0.9800
Mn1—N2	2.2706 (17)	C3—C4	1.379 (3)
Mn1—S1	2.6165 (8)	C4—C5	1.386 (3)
Mn1—S1^{i}	2.6165 (8)	C4—H4	0.9500
S1—C1	1.724 (2)	C5—C6	1.499 (3)
O1—C7	1.423 (3)	C6—H6A	0.9800
O1—H1	0.800 (16)	C6—H6B	0.9800

N1—C3	1.343 (3)	C6—H6C	0.9800
N1—C1	1.351 (3)	C7—H7A	0.9800
N2—C5	1.339 (3)	C7—H7B	0.9800
N2—C1	1.366 (3)	C7—H7C	0.9800
C2—C3	1.499 (3)		

O1—Mn1—O1$^{\rm i}$	90.69 (8)	C3—C2—H2B	109.5
O1—Mn1—N2$^{\rm i}$	161.81 (6)	H2A—C2—H2B	109.5
O1$^{\rm i}$—Mn1—N2$^{\rm i}$	88.19 (6)	C3—C2—H2C	109.5
O1—Mn1—N2	88.19 (6)	H2A—C2—H2C	109.5
O1$^{\rm i}$—Mn1—N2	161.81 (6)	H2B—C2—H2C	109.5
N2$^{\rm i}$—Mn1—N2	98.41 (9)	N1—C3—C4	121.55 (19)
O1—Mn1—S1	92.69 (4)	N1—C3—C2	116.69 (19)
O1$^{\rm i}$—Mn1—S1	97.51 (4)	C4—C3—C2	121.8 (2)
N2$^{\rm i}$—Mn1—S1	105.46 (4)	C3—C4—C5	118.54 (19)
N2—Mn1—S1	64.42 (4)	C3—C4—H4	120.7
O1—Mn1—S1$^{\rm i}$	97.51 (4)	C5—C4—H4	120.7
O1$^{\rm i}$—Mn1—S1$^{\rm i}$	92.69 (4)	N2—C5—C4	120.56 (19)
N2$^{\rm i}$—Mn1—S1$^{\rm i}$	64.42 (4)	N2—C5—C6	118.22 (18)
N2—Mn1—S1$^{\rm i}$	105.46 (4)	C4—C5—C6	121.22 (19)
S1—Mn1—S1$^{\rm i}$	165.47 (3)	C5—C6—H6A	109.5
C1—S1—Mn1	79.29 (7)	C5—C6—H6B	109.5
C7—O1—Mn1	124.58 (14)	H6A—C6—H6B	109.5
C7—O1—H1	108.6 (18)	C5—C6—H6C	109.5
Mn1—O1—H1	115.0 (19)	H6A—C6—H6C	109.5
C3—N1—C1	117.55 (17)	H6B—C6—H6C	109.5
C5—N2—C1	118.22 (17)	O1—C7—H7A	109.5
C5—N2—Mn1	141.17 (14)	O1—C7—H7B	109.5
C1—N2—Mn1	100.41 (12)	H7A—C7—H7B	109.5
N1—C1—N2	123.54 (18)	O1—C7—H7C	109.5
N1—C1—S1	120.88 (15)	H7A—C7—H7C	109.5
N2—C1—S1	115.58 (14)	H7B—C7—H7C	109.5
C3—C2—H2A	109.5		

Symmetry codes: (i) $-x, y, -z+3/2$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1$^{\rm ii}$	0.800 (16)	1.916 (17)	2.716 (2)	178 (3)

Symmetry codes: (ii) $-x, -y, -z+1$.

Fig. 1

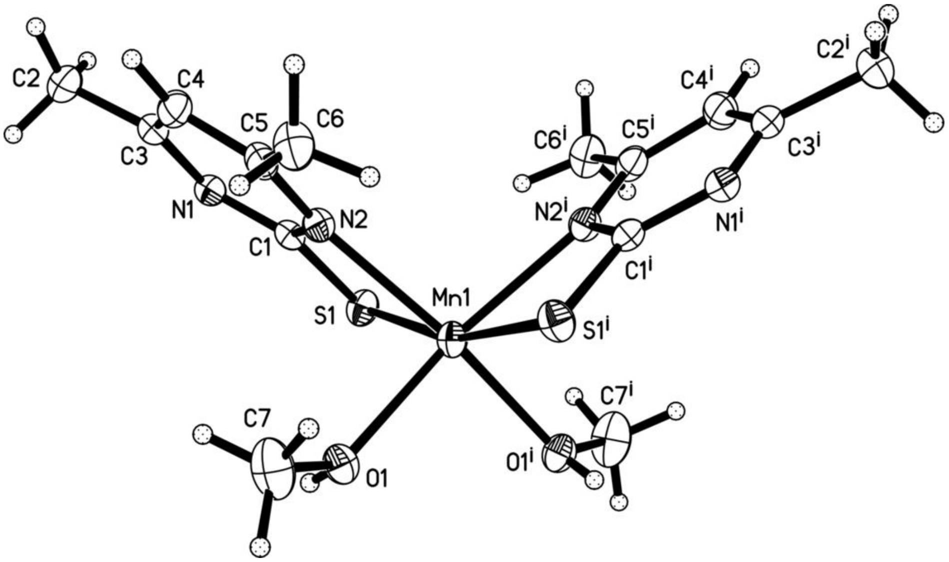
C2
C4
C5
C6
C3
N1
N2
C1
S1
Mn1
C7
O1
C6^i
C5^i
C4^i
C2^i
C3^i
N2^i
N1^i
C1^i
S1^i
C7^i
O1^i

Fig. 2