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Crystal structure of bis[4-(dimethylamino)pyridine-κN^1]bis(methanol-κO)-bis(thiocyanato-κN)manganese(II)

Stefan Suckert,* Inke Jess and Christian Näther

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Max-Eyth-Strasse 2, 24118 Kiel, Germany. *Correspondence e-mail: ssuckert@ac.uni-kiel.de

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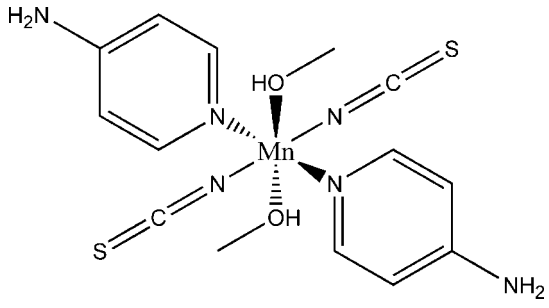
The whole molecule of the title compound, $[Mn(NCS)_2(CH_3OH)_2(C_5H_6N_2)_2]$, is generated by inversion symmetry. The Mn^{II} ion, which is located on an inversion center, is coordinated by two 4-(dimethylamino)pyridine ligands, two methanol ligands and two terminally *N*-bonded thiocyanate anions, forming a slightly distorted octahedron. In the crystal, molecules are linked by O—H···S hydrogen bonds, forming chains extending along the *a*-axis direction.

Keywords: crystal structure; discrete complex; octahedral coordination; hydrogen bonding.

CCDC reference: 1059105

1. Related literature

For the structure of another discrete complex with 4-(dimethylamino)pyridine and thiocyanate ligands, see: Chen *et al.* (2007). For general background to this work, see: Näther *et al.* (2013).



2. Experimental

2.1. Crystal data

$[Mn(NCS)_2(CH_4O)_2(C_5H_6N_2)_2]$
M_r = 479.52
Triclinic, $P\overline{1}$
a = 7.0771 (7) Å
b = 8.1586 (8) Å
c = 10.6491 (10) Å
α = 76.381 (11)°
β = 81.672 (11)°
γ = 79.809 (11)°
V = 584.72 (10) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 180 K
0.16 × 0.10 × 0.04 mm

2.2. Data collection

Stoe IPDS-1 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.903, T_{max} = 0.959
4585 measured reflections
2459 independent reflections
1885 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.100
S = 1.04
2459 reflections
133 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O31—H1*O*···S1^{i}	0.85	2.42	3.2409 (18)	161

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

Data collection: *X-AREA* (Stoe & Cie, 2008); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SJ5446).

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supporting information

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Crystal structure of bis[4-(dimethylamino)pyridine-κN^1]bis(methanol-κO)bis-(thiocyanato-κN)manganese(II)

Stefan Suckert, Inke Jess and Christian Näther

S1. Synthesis and crystallization

$MnSO_4.H_2O$ was purchased from Merck and 4-(dimethylamino)pyridine and $Ba(NCS)_2.3\ H_2O$ were purchased from Alfa Aesar. $Mn(NCS)_2$ was synthesized by stirring 17.97 g (58.44 mmol) $Ba(NCS)_2.3\ H_2O$ and 9.88 g (58.44 mmol) $MnSO_4.H_2O$ in 300 mL H_2O at room temperature for three hours. The white precipitate of $BaSO_4$ was filtered of and the solvent removed with a rotary evaporator. The homogeneity of the product was investigated by X-ray powder diffraction and elemental analysis. The title compound was prepared by the reaction of (0.18 mmol) 30.8 mg $Mn(NCS)_2$ and (0.3 mmol) 36.7 mg 4-(dimethylamino)pyridine in 1.0 mL methanol at room temperature. After a few days colorless plate shaped crystals of the title compound were obtained.

S2. Refinement

The C—H H atoms were positioned with idealized geometry and were refined isotropically with U_{iso}(H) = 1.2 U_{eq}(C) (1.5 for methyl H atoms) using a riding model with C—H = 0.95 Å for aromatic and and C—H = 0.98 Å for methyl H atoms. The O—H H atom was located in a difference map, its bond length set to ideal values of 0.85 Å and refined with U_{iso}(H) = 1.5 U_{eq}(O)using a riding model.

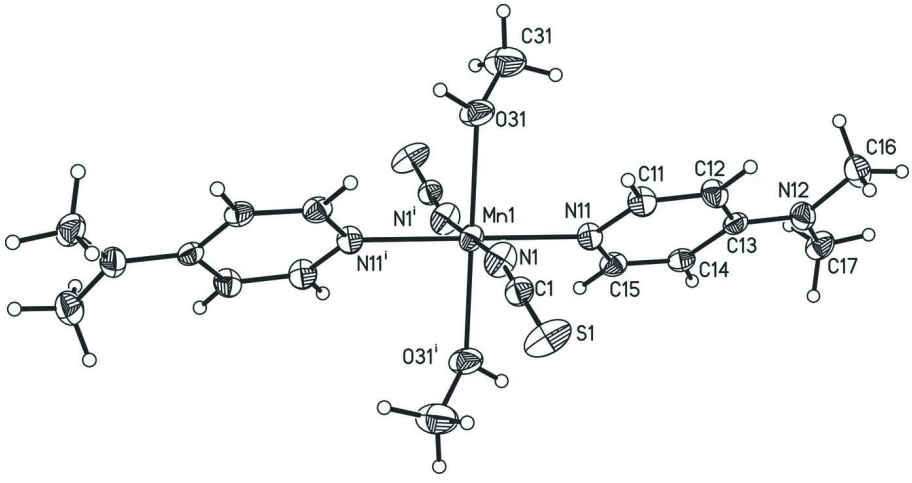

Figure 1

Structure of the title complex with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Symmetry code: i = -x+1,-y,-z+1.

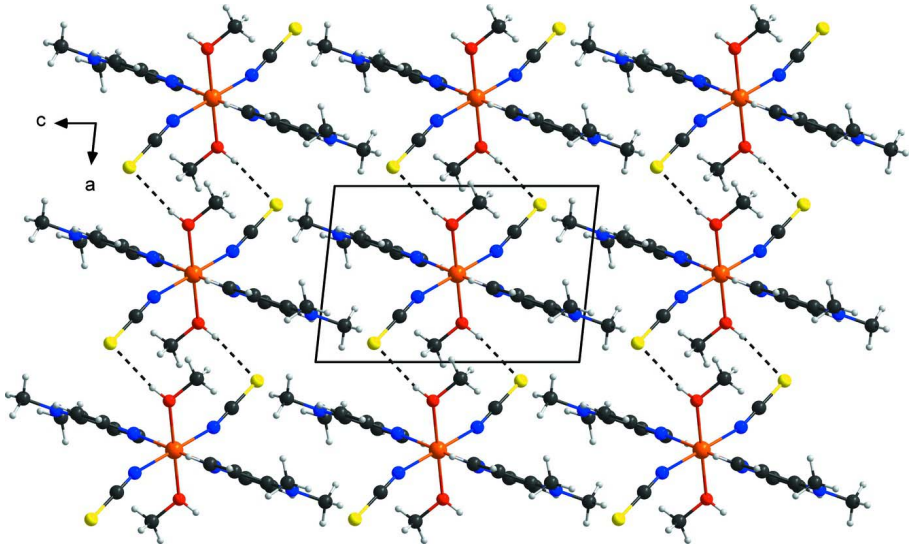

Figure 2

Crystal structure of the title compound viewed perpendicular to the crystallographic *a,c* plane.

Bis[4-(dimethylamino)pyridine-κN^1]bis(methanol-κO)bis(thiocyanato-κN)manganese(II)

Crystal data

$[Mn(NCS)_2(CH_4O)_2(C_5H_6N_2)_2]$
$M_r = 479.52$
Triclinic, $P\overline{1}$
$a = 7.0771$ (7) Å
$b = 8.1586$ (8) Å
$c = 10.6491$ (10) Å
$\alpha = 76.381$ (11)°
$\beta = 81.672$ (11)°
$\gamma = 79.809$ (11)°
$V = 584.72$ (10) Å^3
$Z = 1$
$F(000) = 251$
$D_x = 1.362$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4585 reflections
$\theta = 2.6$–$27.0°$
$\mu = 0.77$ mm^{-1}
$T = 180$ K
Plate, colorless
0.16 × 0.10 × 0.04 mm

Data collection

Stoe IPDS-1 diffractometer
Radiation source: fine-focus sealed tube
phi scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
$T_{min} = 0.903$, $T_{max} = 0.959$
4585 measured reflections
2459 independent reflections
1885 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.6°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.100$
$S = 1.04$
2459 reflections
133 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mn1	0.5000	0.0000	0.5000	0.02111 (15)
N1	0.6294 (3)	0.0926 (3)	0.63997 (19)	0.0312 (5)
C1	0.7395 (3)	0.1199 (3)	0.7004 (2)	0.0236 (5)
S1	0.89299 (9)	0.15954 (10)	0.78630 (6)	0.0394 (2)
O31	0.2205 (2)	0.1737 (2)	0.53900 (16)	0.0339 (4)
H1O	0.1462	0.1444	0.6079	0.051*
C31	0.0945 (4)	0.2643 (4)	0.4439 (3)	0.0486 (7)
H31A	−0.0136	0.3337	0.4847	0.073*
H31B	0.0448	0.1828	0.4074	0.073*
H31C	0.1659	0.3386	0.3743	0.073*
N11	0.5945 (3)	0.2090 (2)	0.33954 (17)	0.0222 (4)
N12	0.7263 (3)	0.6192 (2)	0.03652 (18)	0.0274 (4)
C11	0.5795 (3)	0.3682 (3)	0.3576 (2)	0.0264 (5)
H11	0.5369	0.3866	0.4427	0.032*
C12	0.6210 (3)	0.5063 (3)	0.2624 (2)	0.0252 (5)
H12	0.6057	0.6157	0.2825	0.030*
C13	0.6867 (3)	0.4861 (3)	0.1345 (2)	0.0210 (4)
C14	0.7077 (3)	0.3182 (3)	0.1162 (2)	0.0230 (4)
H14	0.7554	0.2942	0.0334	0.028*
C15	0.6594 (3)	0.1895 (3)	0.2177 (2)	0.0235 (5)
H15	0.6727	0.0783	0.2011	0.028*
C16	0.6858 (4)	0.7919 (3)	0.0569 (3)	0.0353 (6)
H16A	0.7210	0.8713	−0.0243	0.053*
H16B	0.7612	0.8015	0.1244	0.053*
H16C	0.5479	0.8196	0.0844	0.053*
C17	0.7938 (3)	0.5957 (3)	−0.0944 (2)	0.0309 (5)
H17A	0.8140	0.7059	−0.1514	0.046*
H17B	0.6972	0.5482	−0.1267	0.046*
H17C	0.9158	0.5171	−0.0934	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0216 (2)	0.0237 (3)	0.0185 (2)	−0.00575 (18)	0.00127 (17)	−0.00564 (18)
N1	0.0330 (10)	0.0364 (12)	0.0282 (10)	−0.0112 (9)	−0.0016 (8)	−0.0117 (9)
C1	0.0254 (11)	0.0242 (11)	0.0201 (10)	−0.0035 (8)	0.0040 (8)	−0.0067 (9)
S1	0.0261 (3)	0.0703 (5)	0.0288 (3)	−0.0110 (3)	0.0005 (2)	−0.0240 (3)
O31	0.0250 (8)	0.0444 (11)	0.0258 (8)	0.0030 (7)	0.0033 (6)	−0.0046 (8)
C31	0.0373 (15)	0.062 (2)	0.0343 (14)	0.0113 (13)	−0.0022 (11)	−0.0017 (13)

N11	0.0272 (9)	0.0210 (9)	0.0188 (9)	−0.0037 (7)	0.0014 (7)	−0.0070 (7)
N12	0.0300 (10)	0.0241 (10)	0.0248 (10)	−0.0039 (8)	0.0015 (8)	−0.0017 (8)
C11	0.0325 (12)	0.0263 (12)	0.0210 (11)	−0.0043 (9)	0.0028 (8)	−0.0100 (9)
C12	0.0306 (11)	0.0201 (11)	0.0267 (11)	−0.0032 (9)	−0.0011 (9)	−0.0100 (9)
C13	0.0143 (9)	0.0245 (11)	0.0238 (10)	−0.0031 (8)	−0.0019 (7)	−0.0040 (9)
C14	0.0205 (10)	0.0270 (11)	0.0210 (10)	−0.0009 (8)	0.0022 (8)	−0.0090 (9)
C15	0.0222 (10)	0.0242 (11)	0.0258 (11)	−0.0021 (8)	0.0013 (8)	−0.0119 (9)
C16	0.0405 (14)	0.0234 (12)	0.0399 (14)	−0.0064 (10)	−0.0011 (11)	−0.0034 (11)
C17	0.0274 (11)	0.0402 (14)	0.0217 (11)	−0.0070 (10)	−0.0001 (9)	0.0001 (10)

Geometric parameters (Å, º)

Mn1—N1^{i}	2.192 (2)	N12—C16	1.448 (3)
Mn1—N1	2.192 (2)	N12—C17	1.452 (3)
Mn1—N11	2.2302 (17)	C11—C12	1.370 (3)
Mn1—N11^{i}	2.2302 (17)	C11—H11	0.9500
Mn1—O31	2.2676 (17)	C12—C13	1.412 (3)
Mn1—O31^{i}	2.2676 (17)	C12—H12	0.9500
N1—C1	1.160 (3)	C13—C14	1.408 (3)
C1—S1	1.634 (2)	C14—C15	1.368 (3)
O31—C31	1.429 (3)	C14—H14	0.9500
O31—H1O	0.8500	C15—H15	0.9500
C31—H31A	0.9800	C16—H16A	0.9800
C31—H31B	0.9800	C16—H16B	0.9800
C31—H31C	0.9800	C16—H16C	0.9800
N11—C11	1.341 (3)	C17—H17A	0.9800
N11—C15	1.349 (3)	C17—H17B	0.9800
N12—C13	1.355 (3)	C17—H17C	0.9800
N1^{i}—Mn1—N1	180.0	C13—N12—C17	121.3 (2)
N1^{i}—Mn1—N11	89.19 (7)	C16—N12—C17	117.81 (19)
N1—Mn1—N11	90.81 (7)	N11—C11—C12	124.8 (2)
N1^{i}—Mn1—N11^{i}	90.81 (7)	N11—C11—H11	117.6
N1—Mn1—N11^{i}	89.19 (7)	C12—C11—H11	117.6
N11—Mn1—N11^{i}	180.0	C11—C12—C13	120.0 (2)
N1^{i}—Mn1—O31	90.50 (7)	C11—C12—H12	120.0
N1—Mn1—O31	89.50 (7)	C13—C12—H12	120.0
N11—Mn1—O31	89.07 (6)	N12—C13—C14	122.6 (2)
N11^{i}—Mn1—O31	90.93 (6)	N12—C13—C12	122.2 (2)
N1^{i}—Mn1—O31^{i}	89.50 (7)	C14—C13—C12	115.22 (19)
N1—Mn1—O31^{i}	90.50 (7)	C15—C14—C13	120.1 (2)
N11—Mn1—O31^{i}	90.93 (6)	C15—C14—H14	120.0
N11^{i}—Mn1—O31^{i}	89.07 (6)	C13—C14—H14	120.0
O31—Mn1—O31^{i}	180.0	N11—C15—C14	124.7 (2)
C1—N1—Mn1	162.79 (19)	N11—C15—H15	117.6
N1—C1—S1	179.4 (2)	C14—C15—H15	117.6
C31—O31—Mn1	125.44 (16)	N12—C16—H16A	109.5
C31—O31—H1O	105.1	N12—C16—H16B	109.5

Mn1—O31—H1O	119.1	H16A—C16—H16B	109.5
O31—C31—H31A	109.5	N12—C16—H16C	109.5
O31—C31—H31B	109.5	H16A—C16—H16C	109.5
H31A—C31—H31B	109.5	H16B—C16—H16C	109.5
O31—C31—H31C	109.5	N12—C17—H17A	109.5
H31A—C31—H31C	109.5	N12—C17—H17B	109.5
H31B—C31—H31C	109.5	H17A—C17—H17B	109.5
C11—N11—C15	115.11 (18)	N12—C17—H17C	109.5
C11—N11—Mn1	120.90 (14)	H17A—C17—H17C	109.5
C15—N11—Mn1	123.89 (15)	H17B—C17—H17C	109.5
C13—N12—C16	120.6 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O31—H1*O*···S1^{ii}	0.85	2.42	3.2409 (18)	161

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