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Crystal structure of bis(4-acetylpyridine-κN)bis(ethanol-κO)bis(thiocyanato-κN)-manganese(II)

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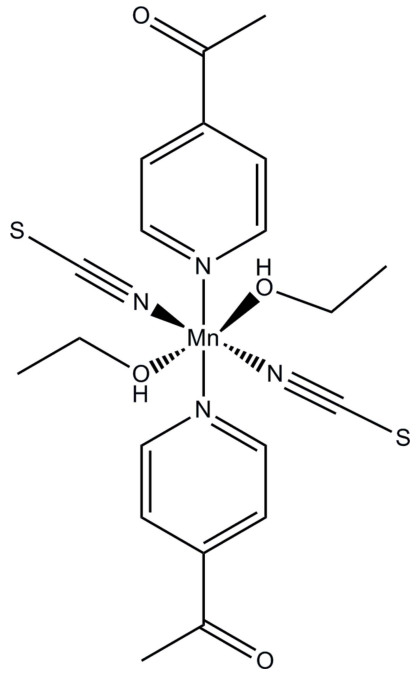
In the crystal structure of the title compound, $[Mn(NCS)_2(C_7H_7NO)_2(C_2H_5OH)_2]$, the Mn^{II} atom is coordinated by two *N*-bonded thiocyanate anions, two 4-acetylpyridine ligands, and two ethanol molecules within a slightly distorted octahedron. The asymmetric unit consits of one manganese cation, located on a centre of inversion, one thiocyanate anion, one 4-acetylpyridine ligand and one ethanol molecule in general positions. The discrete complexes are connected by intermolecular O—H···O hydrogen bonds between the alcohol OH group and the carbonyl O atom into chains parallel to [011].

Keywords: crystal structure; manganese(II); octahedral coordination; hydrogen bonding.

CCDC reference: 1052202

1. Related literature

For a similar structure with thiocyanato ligands in terminal coordination to a manganese(II) atom, see: Li *et al.* (2007).



2. Experimental

2.1. Crystal data

$[Mn(NCS)_2(C_7H_7NO)_2(C_2H_6O)_2]$
M_r = 505.51
Triclinic, $P\overline{1}$
a = 6.9547 (7) Å
b = 9.7733 (9) Å
c = 10.1859 (9) Å
α = 117.449 (10)°
β = 94.978 (11)°
γ = 93.379 (11)°
V = 608.23 (11) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.75 mm^{-1}
T = 200 K
0.04 × 0.03 × 0.02 mm

2.2. Data collection

Stoe IPDS-1 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.966, T_{max} = 0.977
6535 measured reflections
2583 independent reflections
2163 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.088
S = 1.04
2583 reflections
144 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O21—H1*O*1···O11^{i}	0.84	1.95	2.7714 (17)	164

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

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: WM5131).

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supporting information

Acta Cryst. (2015). E**71**, m81–m82 [doi:10.1107/S2056989015004533]

Crystal structure of bis(4-acetylpyridine-κN)bis(ethanol-κO)bis(thiocyanato-κN)manganese(II)

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S1. Synthesis and crystallization

$MnSO_4 \cdot H_2O$ was purchased from Merck; 4-acetylpyridine and $Ba(NCS)_2 \cdot 3H_2O$ were purchased from Alfa Aesar. $Mn(NCS)_2$ was synthesized by stirring 17.97 g (58.44 mmol) $Ba(NCS)_2 \cdot 3H_2O$ and 9.88 g (58.44 mmol) $MnSO_4 \cdot H_2O$ in 400 ml water at room temperature for three hours. The white residue of $BaSO_4$ was filtered off and the solvent evaporated using 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 42.8 mg (0.25 mmol) $Mn(NCS)_2$ and 55.1 μl (0.50 mmol) 4-acetylpyridine in 1.5 ml ethanol at room temperature. After several days, suitable crystals of the title compound were obtained.

S2. Refinement

The C-bound H atoms were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip) and were refined with $U_{iso}(H) = 1.2U_{eq}(C)$ (1.5 for methyl H atoms) using a riding model with C—H = 0.95 Å for aromatic, C—H = 0.99 Å for methylene and C—H = 0.98 Å for methyl H atoms. The O-bound H atom was located in a difference map. Its bond length was set to a value of 0.84 Å and it was refined with $U_{iso}(H) = 1.5U_{eq}(O)$ using a riding model.

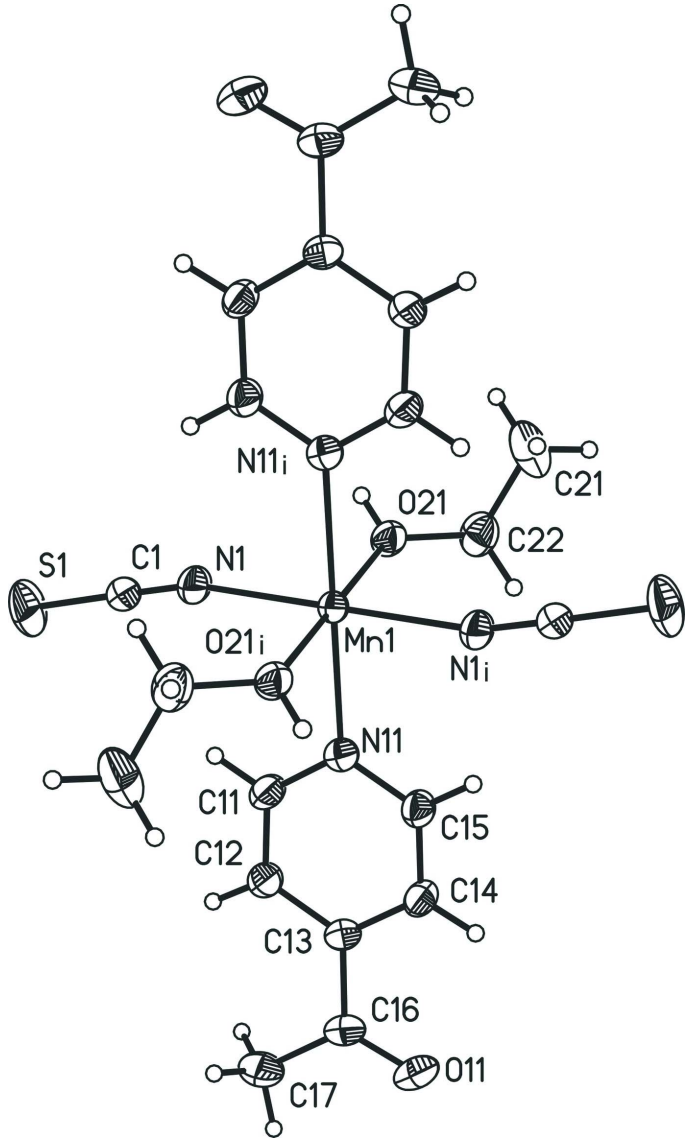

Figure 1

The coordination environment of the Mn^{II} atom in the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) -*x* + 2, -*y* + 1, -*z* + 1.]

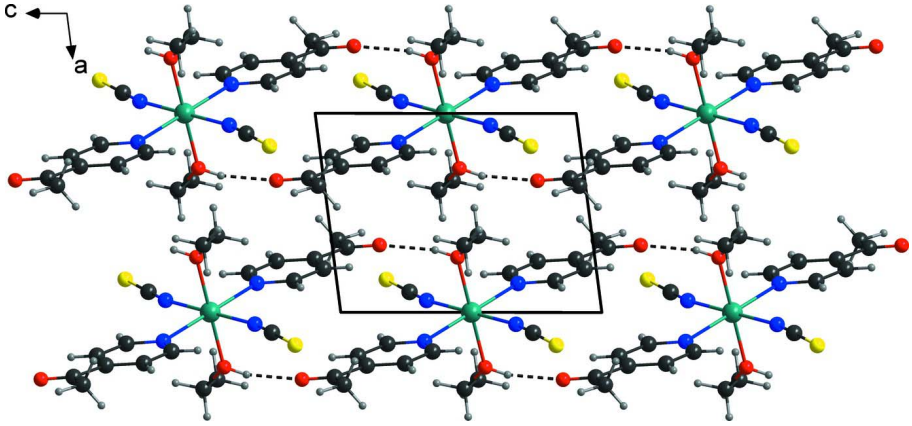

Figure 2

Crystal structure of the title compound in a view along [010]. Hydrogen bonds are indicated by dashed lines.

Bis(ethanol-κ*O*)bis[1-(pyridin-4-yl)ethan-1-one-κ*N*]bis(thiocyanato-κ*N*)manganese(II)

Crystal data

$[Mn(NCS)_2(C_7H_7NO)_2(C_2H_6O)_2]$
$M_r = 505.51$
Triclinic, $P\overline{1}$
$a = 6.9547$ (7) Å
$b = 9.7733$ (9) Å
$c = 10.1859$ (9) Å
$\alpha = 117.449$ (10)°
$\beta = 94.978$ (11)°
$\gamma = 93.379$ (11)°
$V = 608.23$ (11) Å^3
$Z = 1$
$F(000) = 263$
$D_x = 1.380$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6535 reflections
$\theta = 2.4$–27.0°
$\mu = 0.75$ mm^{-1}
$T = 200$ K
Block, colorless
0.04 × 0.03 × 0.02 mm

Data collection

Stoe IPDS-1 diffractometer
phi scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
$T_{min} = 0.966$, $T_{max} = 0.977$
6535 measured reflections
2583 independent reflections
2163 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.4°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.088$
$S = 1.04$
2583 reflections
144 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.069 (8)

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	1.0000	0.5000	0.5000	0.02081 (14)
N1	0.9402 (2)	0.42929 (18)	0.66517 (15)	0.0300 (3)
C1	0.8976 (2)	0.35987 (19)	0.72660 (17)	0.0245 (3)
S1	0.83508 (10)	0.25963 (7)	0.80913 (7)	0.05345 (19)
N11	0.8542 (2)	0.25989 (15)	0.30663 (15)	0.0250 (3)
C11	0.8030 (3)	0.1414 (2)	0.33223 (19)	0.0339 (4)
H11	0.8081	0.1609	0.4329	0.041*
C12	0.7431 (3)	−0.0080 (2)	0.22080 (19)	0.0338 (4)
H12	0.7099	−0.0886	0.2451	0.041*
C13	0.7325 (2)	−0.03842 (18)	0.07316 (17)	0.0241 (3)
C14	0.7813 (3)	0.0837 (2)	0.04487 (18)	0.0276 (4)
H14	0.7738	0.0676	−0.0549	0.033*
C15	0.8413 (3)	0.2296 (2)	0.16300 (18)	0.0278 (4)
H15	0.8749	0.3123	0.1417	0.033*
C16	0.6741 (3)	−0.19848 (19)	−0.05499 (18)	0.0285 (4)
C17	0.6365 (3)	−0.3314 (2)	−0.0243 (2)	0.0417 (5)
H17A	0.6077	−0.4275	−0.1188	0.063*
H17B	0.5254	−0.3158	0.0322	0.063*
H17C	0.7515	−0.3384	0.0341	0.063*
O11	0.6621 (2)	−0.21465 (16)	−0.18155 (14)	0.0409 (3)
C21	0.6624 (4)	0.8271 (3)	0.4935 (3)	0.0549 (6)
H21A	0.5856	0.8597	0.4296	0.082*
H21B	0.6313	0.8831	0.5958	0.082*
H21C	0.8010	0.8504	0.4921	0.082*
C22	0.6154 (3)	0.6558 (2)	0.4366 (2)	0.0367 (4)
H22A	0.4748	0.6326	0.4363	0.044*
H22B	0.6445	0.5999	0.3325	0.044*
O21	0.72349 (18)	0.60072 (14)	0.52571 (12)	0.0291 (3)
H1O1	0.6854	0.6446	0.6101	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0266 (2)	0.01896 (19)	0.01844 (18)	0.00180 (12)	0.00503 (13)	0.00984 (14)
N1	0.0409 (9)	0.0290 (7)	0.0236 (7)	0.0005 (6)	0.0079 (6)	0.0150 (6)
C1	0.0280 (8)	0.0232 (7)	0.0217 (7)	0.0021 (6)	0.0039 (6)	0.0099 (6)
S1	0.0678 (4)	0.0535 (4)	0.0652 (4)	0.0037 (3)	0.0157 (3)	0.0489 (3)
N11	0.0295 (7)	0.0219 (7)	0.0220 (6)	0.0014 (5)	0.0027 (5)	0.0091 (6)
C11	0.0525 (12)	0.0266 (8)	0.0206 (7)	−0.0014 (8)	0.0051 (7)	0.0099 (7)

C12	0.0539 (12)	0.0225 (8)	0.0245 (8)	−0.0016 (8)	0.0056 (8)	0.0111 (7)
C13	0.0237 (8)	0.0241 (7)	0.0219 (7)	0.0041 (6)	0.0051 (6)	0.0080 (6)
C14	0.0323 (9)	0.0305 (8)	0.0194 (7)	0.0012 (7)	0.0039 (6)	0.0114 (7)
C15	0.0338 (9)	0.0267 (8)	0.0240 (7)	−0.0015 (7)	0.0026 (7)	0.0136 (7)
C16	0.0273 (8)	0.0258 (8)	0.0254 (8)	0.0060 (7)	0.0056 (7)	0.0053 (7)
C17	0.0550 (13)	0.0229 (9)	0.0374 (10)	−0.0008 (8)	0.0026 (9)	0.0069 (8)
O11	0.0527 (8)	0.0372 (7)	0.0221 (6)	0.0057 (6)	0.0073 (6)	0.0043 (5)
C21	0.0604 (15)	0.0522 (13)	0.0762 (16)	0.0160 (11)	0.0178 (13)	0.0477 (13)
C22	0.0325 (9)	0.0459 (11)	0.0341 (9)	0.0083 (8)	0.0003 (7)	0.0210 (9)
O21	0.0336 (6)	0.0315 (6)	0.0238 (5)	0.0115 (5)	0.0080 (5)	0.0128 (5)

Geometric parameters (Å, º)

Mn1—N1^{i}	2.1543 (15)	C14—C15	1.385 (2)
Mn1—N1	2.1543 (15)	C14—H14	0.9500
Mn1—O21^{i}	2.1928 (12)	C15—H15	0.9500
Mn1—O21	2.1929 (12)	C16—O11	1.219 (2)
Mn1—N11^{i}	2.3508 (14)	C16—C17	1.484 (3)
Mn1—N11	2.3508 (14)	C17—H17A	0.9800
N1—C1	1.157 (2)	C17—H17B	0.9800
C1—S1	1.6211 (18)	C17—H17C	0.9800
N11—C11	1.334 (2)	C21—C22	1.502 (3)
N11—C15	1.346 (2)	C21—H21A	0.9800
C11—C12	1.383 (2)	C21—H21B	0.9800
C11—H11	0.9500	C21—H21C	0.9800
C12—C13	1.386 (2)	C22—O21	1.435 (2)
C12—H12	0.9500	C22—H22A	0.9900
C13—C14	1.381 (3)	C22—H22B	0.9900
C13—C16	1.505 (2)	O21—H1O1	0.8400
N1^{i}—Mn1—N1	180.0	C13—C14—H14	120.2
N1^{i}—Mn1—O21^{i}	88.59 (5)	C15—C14—H14	120.2
N1—Mn1—O21^{i}	91.41 (5)	N11—C15—C14	123.05 (16)
N1^{i}—Mn1—O21	91.41 (5)	N11—C15—H15	118.5
N1—Mn1—O21	88.59 (5)	C14—C15—H15	118.5
O21^{i}—Mn1—O21	180.0	O11—C16—C17	122.10 (16)
N1^{i}—Mn1—N11^{i}	91.13 (5)	O11—C16—C13	118.40 (17)
N1—Mn1—N11^{i}	88.87 (5)	C17—C16—C13	119.49 (16)
O21^{i}—Mn1—N11^{i}	92.35 (5)	C16—C17—H17A	109.5
O21—Mn1—N11^{i}	87.65 (5)	C16—C17—H17B	109.5
N1^{i}—Mn1—N11	88.87 (5)	H17A—C17—H17B	109.5
N1—Mn1—N11	91.13 (5)	C16—C17—H17C	109.5
O21^{i}—Mn1—N11	87.65 (5)	H17A—C17—H17C	109.5
O21—Mn1—N11	92.35 (5)	H17B—C17—H17C	109.5
N11^{i}—Mn1—N11	180.0	C22—C21—H21A	109.5
C1—N1—Mn1	164.93 (13)	C22—C21—H21B	109.5
N1—C1—S1	178.67 (16)	H21A—C21—H21B	109.5
C11—N11—C15	116.73 (14)	C22—C21—H21C	109.5

C11—N11—Mn1	121.49 (11)	H21A—C21—H21C	109.5
C15—N11—Mn1	121.25 (11)	H21B—C21—H21C	109.5
N11—C11—C12	123.87 (16)	O21—C22—C21	112.19 (18)
N11—C11—H11	118.1	O21—C22—H22A	109.2
C12—C11—H11	118.1	C21—C22—H22A	109.2
C11—C12—C13	118.96 (17)	O21—C22—H22B	109.2
C11—C12—H12	120.5	C21—C22—H22B	109.2
C13—C12—H12	120.5	H22A—C22—H22B	107.9
C14—C13—C12	117.87 (15)	C22—O21—Mn1	130.79 (11)
C14—C13—C16	119.69 (15)	C22—O21—H1O1	105.5
C12—C13—C16	122.44 (16)	Mn1—O21—H1O1	120.2
C13—C14—C15	119.50 (15)		

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O21—H1*O*1···O11^{ii}	0.84	1.95	2.7714 (17)	164

Symmetry code: (ii) *x*, *y*+1, *z*+1.