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Diaquabis(selenocyanato-κ*N*)bis(pyrimidine-κ*N*)manganese(II)

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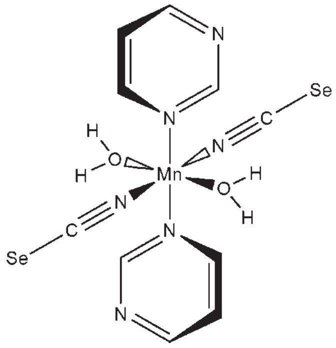
Key indicators: single-crystal X-ray study; T = 170 K; mean σ(C–C) = 0.004 Å; R factor = 0.026; wR factor = 0.064; data-to-parameter ratio = 20.7.

In the crystal structure of the title compound, $[Mn(NCSe)_2(C_4H_4N_2)_2(H_2O)_2]$, the manganese(II) cation is coordinated by two *N*-bonded pyrimidine ligands, two *N*-bonded selenocyanate anions and two *O*-bonded water molecules in a distorted octahedral coordination mode. The asymmetric unit consists of one manganese(II) cation, located on a centre of inversion, as well as one selenocyanate anion, one water molecule and one pyrimidine ligand in general positions. The crystal structure consists of discrete building blocks of composition $[Mn(NCSe)_2(pyrimidine)_2(H_2O)_2]$, which are connected into layers parallel to (101) by strong water–pyrimidine O—H···N hydrogen bonds.

Related literature

For a related pyrimidine structure, see: Lipkowski & Soldatov (1993). For general background to the use of thermal decomposition reactions for the discovery and preparation of new ligand-deficient coordination polymers with defined magnetic properties, see: Wriedt & Näther (2009*a*,*b*); Wriedt *et al.* (2009*a*,*b*).



Experimental

Crystal data

$[Mn(CNSe)_2(C_4H_4N_2)_2(H_2O)_2]$
M_r = 461.12
Monoclinic, $P2_1/n$
a = 9.2402 (7) Å
b = 9.6012 (6) Å
c = 10.2099 (8) Å
β = 111.505 (8)°
V = 842.74 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.11 mm^{-1}
T = 170 K
0.10 × 0.07 × 0.04 mm

Data collection

Stoe IPDS-1 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.653, T_{max} = 0.818
9472 measured reflections
2024 independent reflections
1795 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.064
S = 1.03
2024 reflections
98 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mn1—O1	2.1582 (14)	Mn1—N1	2.3328 (18)
Mn1—N11	2.1840 (19)		
O1—Mn1—O1^i	180.0	N11—Mn1—N1^i	93.23 (7)
O1—Mn1—N11	90.29 (7)	N11^i—Mn1—N1^i	86.77 (7)
O1^i—Mn1—N11	89.71 (7)	O1—Mn1—N1	89.56 (6)
O1—Mn1—N1^i	90.44 (6)		

Symmetry code: (i) $-x+1, -y+2, -z+1$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H2*O*1···N2^{ii}	0.84	1.93	2.748 (2)	164

Symmetry code: (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

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: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *XCIF* in *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2144).

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Acta Cryst. (2010). E**66**, m1014–m1015 Wriedt *et al.* • $[Mn(CNSe)_2(C_4H_4N_2)_2(H_2O)_2]$ **m1015**

supporting information

Acta Cryst. (2010). E**66**, m1014–m1015   [https://doi.org/10.1107/S1600536810028941]

Diaquabis(selenocyanato-*κN*)bis(pyrimidine-*κN*)manganese(II)

Mario Wriedt, Inke Jess and Christian Näther

S1. Comment

Recently, we have shown that thermal decomposition reactions are an elegante route for the discovering and preparation of new ligand-deficient coordination polymers with defined magnetic properties (Wriedt & Näther, 2009*a*, 2009*b*; Wriedt, Sellmer & Näther, 2009*a*, 2009*b*). In our ongoing investigation on the synthesis, structures and properties of such compounds based on paramagnetic transition metal pseudo-halides and N-donor ligands, we have reacted manganese(II) dichloride, potassium selenocyanate and pyrimidine in water. In this reaction single crystals were obtained, which were identified as the title compound by single-crystal X-ray diffraction.

The title compound of composition $[Mn(NCSe)_2(H_2O)_2(pyrimidine)_2]$ (Fig. 1) represents a discrete coordination complex, in which the manganese(II) cation is coordinated by two selenocyanato anions, two water molecules and two pyrimidine ligands in an octahedral coordination mode. The MnN_4O_2 octahedron is slightly distorted with two long $Mn–N_{pyrimidine}$ distances of 2.3328 (18) Å, two short Mn–NCSe distances of 2.1840 (9) Å and two short $Mn—OH_2$ distances of 2.1582 (14) Å, while the angles around the metal center range between 86.77 (7)–93.23 (7) and 180° (Tab. 1). The coordination of the metal center is similar to that in a related structure (Lipkowski & Soldatov, 1993). In the crystal structure the single complexes are connected *via* strong $N_{pyrimidine}\cdots H_{water}$ hydrogen bonds into layers (see Tab. 2), which are located in the crystallographic *a*/*c*-plane (Fig. 2 and 3). The shortest intra- and interlayer Mn···Mn distances amount to 7.2911 (5) and 9.3672 (5) Å, respectively.

S2. Experimental

$MnCl_2$, KNCSe and pyrimidine were obtained from Alfa Aesar. 1 mmol (126 mg) $MnCl_2$, 2 mmol (288 mg) KNCSe, 0.25 mmol (20 mg) pyrimidine and 3 ml water were reacted in a closed snap-vail without stirring. After the mixture was standing for several days at room temperature colorless block shaped single crystals of the title compound were obtained in a mixture with unknown phases.

S3. Refinement

All non-hydrogen atoms were refined anisotropic. The *O*—*H*-hydrogen atoms were located in difference map, where the bond lengths set to ideal values and were refined using a riding model. All other H atoms were located in difference map but were positioned with idealized geometry and were refined isotropic with $U_{eq}(H) = 1.2\ U_{eq}(C)$ of the parent atom using a riding model with C—H = 0.95 Å.

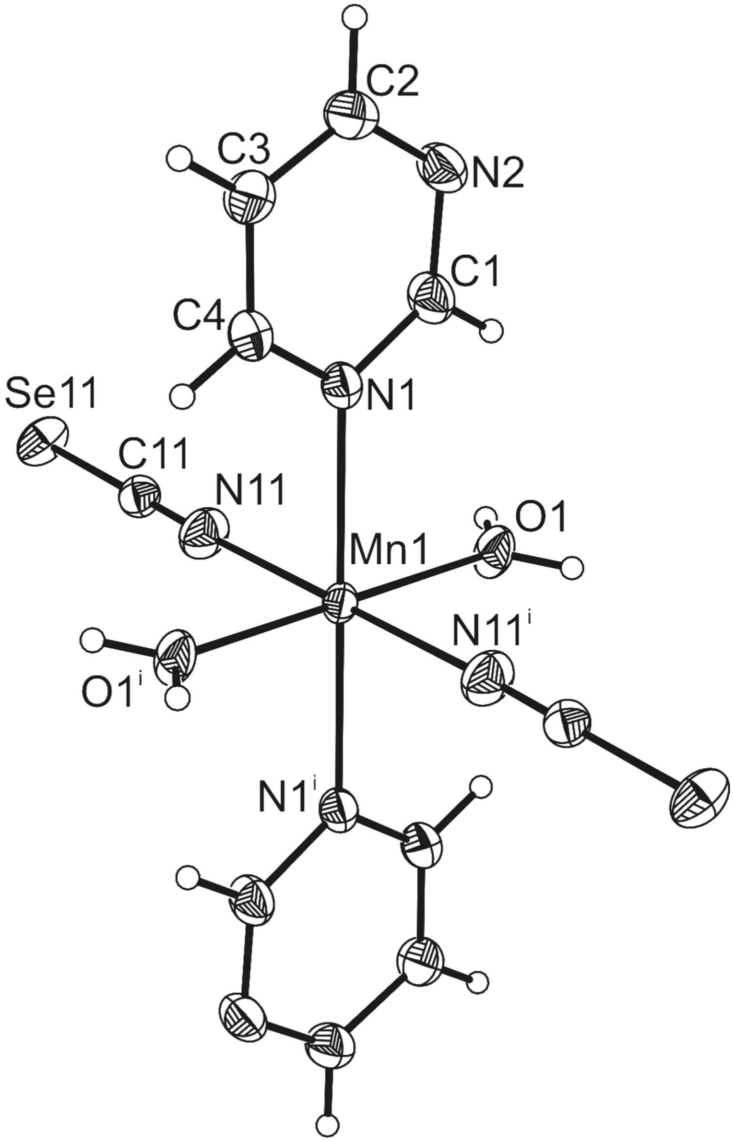

Figure 1

Crystal structure of the discrete title compound with labelling and displacement ellipsoids drawn at the 50% probability level. [Symmetry codes: (i) -*x*+1, -*y*+2, -*z*+1.]

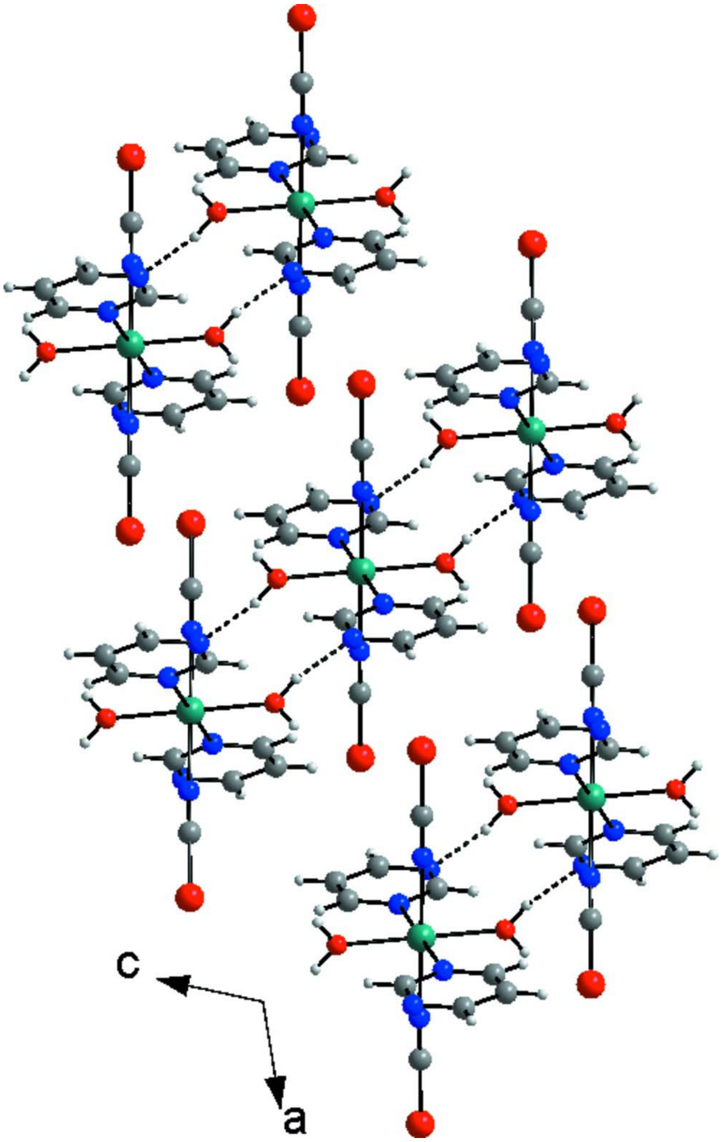

Figure 2

Crystal structure of the title compound with view along the crystallographic *b* axis. The dashed lines indicate N···H—O hydrogen bonding.

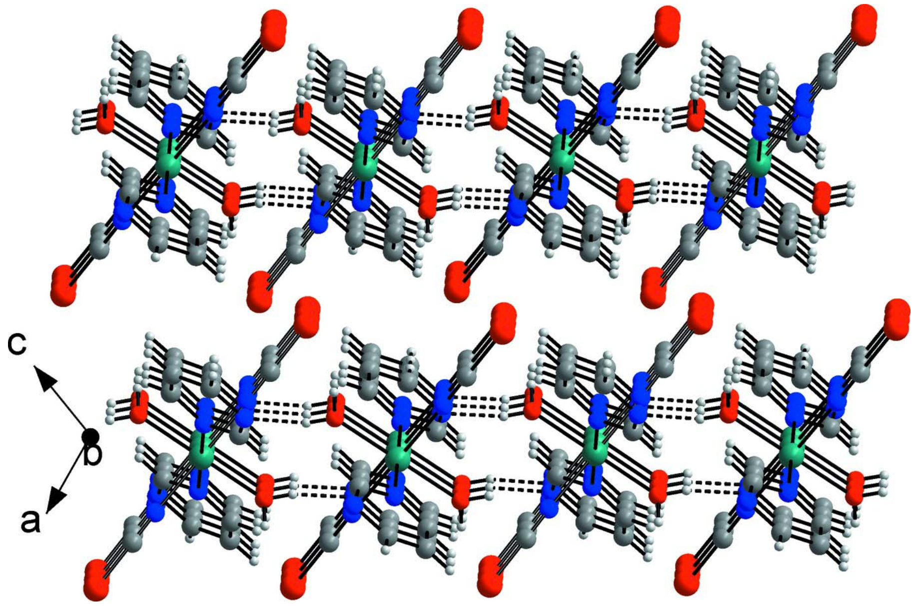

Figure 3

Packing of two single layers with view approximately along the crystallographic *b* axis. The dashed lines indicate N···H—O hydrogen bonding.

Diaquabis(selenocyanato-*κN*)bis(pyrimidine-*κN*)manganese(II)

Crystal data

$[Mn(CNSe)_2(C_4H_4N_2)_2(H_2O)_2]$	$F(000) = 446$
$M_r = 461.12$	$D_x = 1.817$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 9472 reflections
$a = 9.2402$ (7) Å	$\theta = 2.6–28.0°$
$b = 9.6012$ (6) Å	$\mu = 5.11$ mm^{-1}
$c = 10.2099$ (8) Å	$T = 170$ K
$\beta = 111.505$ (8)°	Block, colourless
$V = 842.74$ (11) Å^3	0.10 × 0.07 × 0.04 mm
$Z = 2$	

Data collection

Stoe IPDS-1 diffractometer	9472 measured reflections
Radiation source: fine-focus sealed tube	2024 independent reflections
Graphite monochromator	1795 reflections with $I > 2\sigma(I)$
Phi scans	$R_{\text{int}} = 0.043$
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)	$\theta_{\max} = 28.0°$, $\theta_{\min} = 2.6°$
$T_{\min} = 0.653$, $T_{\max} = 0.818$	$h = -12\rightarrow12$
	$k = -12\rightarrow12$
	$l = -13\rightarrow13$

Refinement

Refinement on F^2	2024 reflections
Least-squares matrix: full	98 parameters
$R[F^2 > 2\sigma(F^2)] = 0.026$	0 restraints
$wR(F^2) = 0.064$	Primary atom site location: structure-invariant direct methods
$S = 1.03$	

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 0.3681P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0110 (14)

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	0.5000	1.0000	0.5000	0.01759 (12)
N1	0.4202 (2)	0.77991 (19)	0.54320 (18)	0.0208 (4)
N2	0.3036 (2)	0.5623 (2)	0.45030 (19)	0.0289 (4)
C1	0.3525 (3)	0.6901 (3)	0.4381 (2)	0.0265 (5)
H1	0.3380	0.7208	0.3456	0.032*
C2	0.3267 (3)	0.5183 (3)	0.5807 (2)	0.0298 (5)
H2	0.2947	0.4271	0.5938	0.036*
C3	0.3961 (3)	0.6023 (3)	0.6969 (2)	0.0299 (5)
H3	0.4128	0.5705	0.7894	0.036*
C4	0.4398 (3)	0.7336 (2)	0.6731 (2)	0.0261 (5)
H4	0.4856	0.7940	0.7511	0.031*
N11	0.7272 (2)	0.9064 (2)	0.5380 (2)	0.0300 (4)
C11	0.8449 (2)	0.8544 (2)	0.5539 (2)	0.0214 (4)
Se11	1.02886 (3)	0.77302 (3)	0.58075 (3)	0.02776 (10)
O1	0.42680 (19)	0.94905 (18)	0.27927 (15)	0.0283 (3)
H1O1	0.4782	0.9061	0.2392	0.042*
H2O1	0.3448	0.9730	0.2134	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0178 (2)	0.0179 (2)	0.0143 (2)	0.00340 (15)	0.00258 (15)	−0.00073 (14)
N1	0.0200 (8)	0.0227 (9)	0.0175 (8)	−0.0008 (7)	0.0044 (7)	−0.0010 (6)
N2	0.0341 (10)	0.0270 (11)	0.0222 (9)	−0.0077 (8)	0.0061 (7)	−0.0039 (7)
C1	0.0300 (11)	0.0291 (12)	0.0185 (10)	−0.0042 (9)	0.0068 (8)	−0.0022 (8)
C2	0.0345 (12)	0.0246 (12)	0.0279 (11)	−0.0045 (9)	0.0086 (9)	0.0002 (9)
C3	0.0381 (12)	0.0285 (12)	0.0200 (10)	−0.0020 (10)	0.0070 (9)	0.0028 (9)
C4	0.0289 (11)	0.0253 (11)	0.0191 (9)	−0.0015 (9)	0.0030 (8)	−0.0021 (8)
N11	0.0230 (9)	0.0297 (11)	0.0367 (10)	0.0060 (8)	0.0101 (8)	0.0036 (8)

C11	0.0218 (9)	0.0218 (10)	0.0205 (9)	−0.0012 (8)	0.0078 (7)	0.0022 (7)
Se11	0.02287 (14)	0.02978 (16)	0.03597 (15)	0.00821 (9)	0.01709 (10)	0.00904 (9)
O1	0.0356 (8)	0.0300 (9)	0.0136 (6)	0.0116 (7)	0.0024 (6)	−0.0026 (6)

Geometric parameters (Å, º)

Mn1—O1	2.1582 (14)	C1—H1	0.9500
Mn1—O1$^{\rm i}$	2.1582 (14)	C2—C3	1.383 (3)
Mn1—N11	2.1840 (19)	C2—H2	0.9500
Mn1—N11$^{\rm i}$	2.1840 (19)	C3—C4	1.372 (3)
Mn1—N1$^{\rm i}$	2.3328 (18)	C3—H3	0.9500
Mn1—N1	2.3328 (18)	C4—H4	0.9500
N1—C1	1.340 (3)	N11—C11	1.153 (3)
N1—C4	1.347 (3)	C11—Se11	1.798 (2)
N2—C1	1.329 (3)	O1—H1O1	0.8400
N2—C2	1.337 (3)	O1—H2O1	0.8400
O1—Mn1—O1$^{\rm i}$	180.0	C1—N2—C2	116.70 (19)
O1—Mn1—N11	90.29 (7)	N2—C1—N1	126.4 (2)
O1$^{\rm i}$—Mn1—N11	89.71 (7)	N2—C1—H1	116.8
O1—Mn1—N11$^{\rm i}$	89.71 (7)	N1—C1—H1	116.8
O1$^{\rm i}$—Mn1—N11$^{\rm i}$	90.29 (7)	N2—C2—C3	121.7 (2)
N11—Mn1—N11$^{\rm i}$	180.0	N2—C2—H2	119.2
O1—Mn1—N1$^{\rm i}$	90.44 (6)	C3—C2—H2	119.2
O1$^{\rm i}$—Mn1—N1$^{\rm i}$	89.56 (6)	C4—C3—C2	117.2 (2)
N11—Mn1—N1$^{\rm i}$	93.23 (7)	C4—C3—H3	121.4
N11$^{\rm i}$—Mn1—N1$^{\rm i}$	86.77 (7)	C2—C3—H3	121.4
O1—Mn1—N1	89.56 (6)	N1—C4—C3	122.4 (2)
O1$^{\rm i}$—Mn1—N1	90.44 (6)	N1—C4—H4	118.8
N11—Mn1—N1	86.77 (7)	C3—C4—H4	118.8
N11$^{\rm i}$—Mn1—N1	93.23 (7)	C11—N11—Mn1	177.7 (2)
N1$^{\rm i}$—Mn1—N1	180.00 (9)	N11—C11—Se11	179.4 (2)
C1—N1—C4	115.56 (19)	Mn1—O1—H1O1	127.1
C1—N1—Mn1	121.24 (15)	Mn1—O1—H2O1	128.3
C4—N1—Mn1	123.19 (14)	H1O1—O1—H2O1	104.5

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H2*O*1···N2$^{\rm ii}$	0.84	1.93	2.748 (2)	164

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