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Crystal structure of poly[[(2,2′-bipyridine)manganese(II)]-di-μ-thiocyanato]

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In the crystal structure of the polymeric title compound, $[Mn(NCS)_2(C_{10}H_8N_2)]_n$, the Mn^{II} cations are coordinated by one chelating 2,2′-bipyridine ligand and four thiocyanate anions (two N- and two S-coordinating), forming a distorted $[MnN_4S_2]$ octahedron. The asymmetric unit consists of one manganese cation located on a twofold rotation axis and half of a 2,2′-bipyridine ligand, the other half being generated by the same twofold rotation axis, as well as one thiocyanate anion in a general position. The Mn^{II} cations are linked by two pairs of $\mu_{1,3}$-bridging thiocyanate ligands into chains along the c axis; because the N atoms of the 2,2′-bipyridine ligands, as well as the N and the S atoms of the thiocyanate anions, are each cis-coordinating, these chains show a zigzag arrangement.

1. Related literature

For the magnetic properties of the title compound, see: Dockum *et al.* (1983). For general background to this work, see: Näther *et al.* (2013).
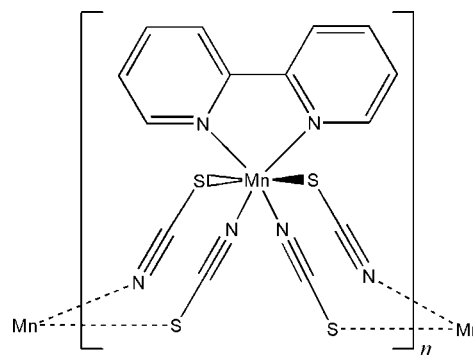
2. Experimental

2.1. Crystal data

$[Mn(NCS)_2(C_{10}H_8N_2)]$	$V = 1317.51 (17)$ Å^3
$M_r = 327.28$	$Z = 4$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 7.6158 (5)$ Å	$\mu = 1.31$ mm^{-1}
$b = 16.2007 (14)$ Å	$T = 180$ K
$c = 10.6784 (7)$ Å	$0.24 \times 0.18 \times 0.11$ mm
$\beta = 90.129 (8)°$	

2.2. Data collection

STOE IPDS-1 diffractometer	5163 measured reflections
Absorption correction: numerical	1424 independent reflections
(*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)	1208 reflections with $I > 2\sigma(I)$
$T_{min} = 0.714$, $T_{max} = 0.825$	$R_{int} = 0.029$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$	87 parameters
$wR(F^2) = 0.065$	H-atom parameters constrained
$S = 1.10$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
1424 reflections	$\Delta\rho_{min} = -0.35$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Mn1—N1^i	2.1318 (13)	Mn1—S1	2.8138 (5)
Mn1—N10	2.2433 (12)		

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: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

data reports

References

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Crystal structure of poly[[(2,2′-bipyridine)manganese(II)]-di-*µ*-thiocyanato]

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S1. Synthesis and crystallization

$MnSO_4 \cdot H_2O$ was purchased from Merck and 2,2′-bipyridine and $Ba(NCS)_2 \cdot 3 H_2O$ 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 300 ml water at RT for three hours. The white precipitate of $BaSO_4$ was filtered off 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 (0.4 mmol) 70.0 mg $Mn(NCS)_2$ and (0.05 mmol) 7.0 mg 2,2′-bipyridine in 1.0 ml acetonitrile at RT. After few days, yellow block-shaped crystals of the title compound were obtained.

S2. Refinement

The H atoms were positioned with idealized geometry and were refined with C—H = 0.93 Å and $U_{eq}(H) = 1.2U_{eq}(C)$ using a riding model.
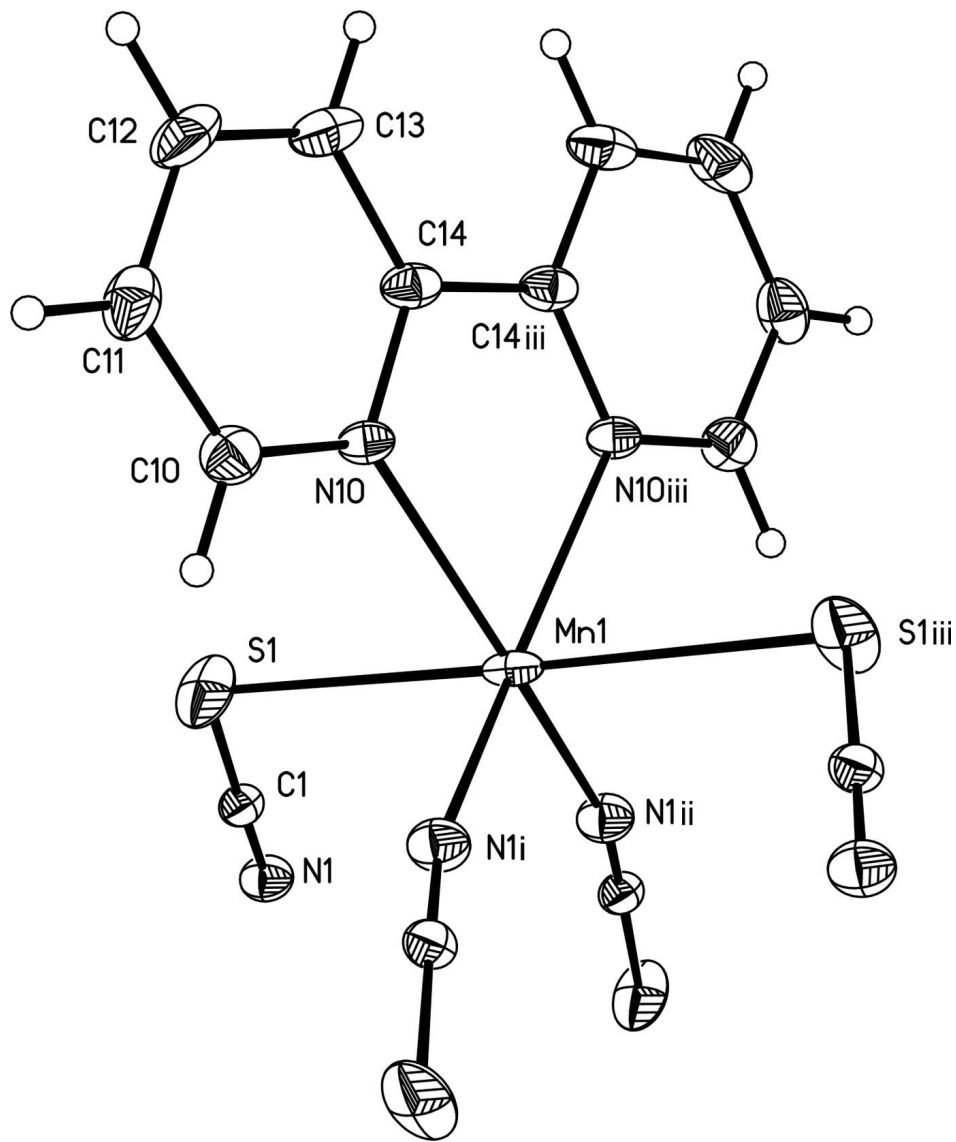
Figure 1

The coordination of the Mn^{II} atom in the title compound with atom labelling and displacement ellipsoids drawn at the 50% probability level. [Symmetry codes: i) x,-y+1,z-1/2; ii) -x,-y+1,-z+1; iii) -x,y,-z+1/2.]
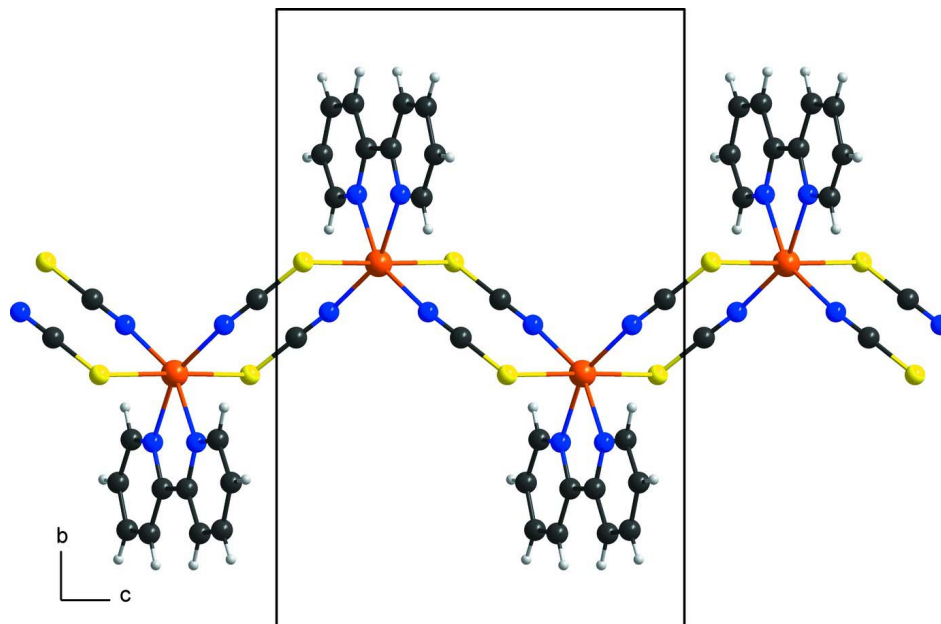
Figure 2

The polymeric arrangement of the chains in the crystal structure of the title compound in a view along the *a* axis. Colour code: Mn orange; N blue; S yellow; C black; H white.

Poly[[(2,2′-bipyridine)manganese(II)]-di-*µ*-thiocyanato]

Crystal data

$[Mn(NCS)_2(C_{10}H_8N_2)]$	$F(000) = 660$
$M_r = 327.28$	$D_x = 1.650$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 5163 reflections
$a = 7.6158$ (5) Å	$\theta = 3.2$–27.0°
$b = 16.2007$ (14) Å	$\mu = 1.31$ mm^{-1}
$c = 10.6784$ (7) Å	$T = 180$ K
$\beta = 90.129$ (8)°	Block, yellow
$V = 1317.51$ (17) Å^3	$0.24 \times 0.18 \times 0.11$ mm
$Z = 4$	

Data collection

STOE IPDS-1 diffractometer	5163 measured reflections
Radiation source: fine-focus sealed tube	1424 independent reflections
Graphite monochromator	1208 reflections with $I > 2\sigma(I)$
phi scans	$R_{int} = 0.029$
Absorption correction: numerical	$\theta_{max} = 27.0°$, $\theta_{min} = 3.2°$
(*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)	$h = -9 \rightarrow 9$
	$k = -20 \rightarrow 20$
$T_{min} = 0.714$, $T_{max} = 0.825$	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$S = 1.10$
Least-squares matrix: full	1424 reflections
$R[F^2 > 2\sigma(F^2)] = 0.026$	87 parameters
$wR(F^2) = 0.065$	0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.0505P]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.22$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ 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.

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.589163 (17)	0.2500	0.01511 (12)
S1	0.26426 (6)	0.59114 (3)	0.43456 (4)	0.02879 (14)
C1	0.1959 (2)	0.53098 (9)	0.54809 (13)	0.0157 (3)
N1	0.14566 (19)	0.48959 (8)	0.62863 (13)	0.0191 (3)
N10	0.15848 (17)	0.70040 (7)	0.19663 (11)	0.0150 (3)
C10	0.3171 (2)	0.69584 (10)	0.14411 (14)	0.0205 (3)
H10	0.3650	0.6440	0.1287	0.025*
C11	0.4133 (2)	0.76537 (11)	0.11149 (15)	0.0246 (4)
H11	0.5240	0.7605	0.0756	0.029*
C12	0.3400 (2)	0.84198 (11)	0.13384 (16)	0.0277 (4)
H12	0.4008	0.8897	0.1122	0.033*
C13	0.1770 (3)	0.84748 (9)	0.18819 (16)	0.0256 (4)
H13	0.1267	0.8988	0.2037	0.031*
C14	0.0878 (2)	0.77516 (9)	0.21990 (13)	0.0175 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0203 (2)	0.00785 (16)	0.01723 (18)	0.000	0.00916 (12)	0.000
S1	0.0261 (3)	0.0358 (3)	0.0244 (2)	−0.01473 (18)	0.00026 (18)	0.01435 (16)
C1	0.0150 (8)	0.0134 (6)	0.0186 (7)	−0.0014 (6)	0.0007 (5)	−0.0002 (5)
N1	0.0218 (7)	0.0167 (6)	0.0187 (6)	−0.0016 (5)	0.0031 (5)	0.0031 (5)
N10	0.0193 (7)	0.0116 (5)	0.0141 (6)	−0.0013 (5)	0.0024 (5)	0.0023 (4)
C10	0.0215 (9)	0.0203 (7)	0.0195 (7)	−0.0010 (6)	0.0016 (6)	0.0026 (6)
C11	0.0196 (9)	0.0319 (8)	0.0222 (8)	−0.0067 (7)	−0.0002 (6)	0.0060 (7)
C12	0.0319 (11)	0.0242 (8)	0.0269 (9)	−0.0145 (7)	−0.0025 (7)	0.0075 (6)
C13	0.0352 (11)	0.0140 (7)	0.0275 (8)	−0.0055 (7)	−0.0005 (7)	0.0002 (6)
C14	0.0254 (9)	0.0133 (7)	0.0137 (6)	−0.0011 (6)	−0.0009 (6)	0.0018 (5)

Geometric parameters (Å, º)

Mn1—N1^i	2.1318 (13)	N10—C14	1.3485 (18)
Mn1—N1^{ii}	2.1318 (13)	C10—C11	1.389 (2)
Mn1—N10^{iii}	2.2433 (12)	C10—H10	0.9300
Mn1—N10	2.2433 (12)	C11—C12	1.382 (3)
Mn1—S1^{iii}	2.8138 (5)	C11—H11	0.9300
Mn1—S1	2.8138 (5)	C12—C13	1.375 (3)
S1—C1	1.6411 (15)	C12—H12	0.9300
C1—N1	1.156 (2)	C13—C14	1.396 (2)
N1—Mn1^{ii}	2.1318 (13)	C13—H13	0.9300
N10—C10	1.335 (2)	C14—C14^{iii}	1.486 (3)
N1^i—Mn1—N1^{ii}	106.48 (7)	C10—N10—C14	119.27 (13)
N1^i—Mn1—N10^{iii}	156.38 (5)	C10—N10—Mn1	123.37 (11)
N1^{ii}—Mn1—N10^{iii}	92.60 (5)	C14—N10—Mn1	117.36 (10)
N1^i—Mn1—N10	92.60 (5)	N10—C10—C11	122.62 (16)
N1^{ii}—Mn1—N10	156.38 (5)	N10—C10—H10	118.7
N10^{iii}—Mn1—N10	73.10 (7)	C11—C10—H10	118.7
N1^i—Mn1—S1^{iii}	87.33 (4)	C12—C11—C10	118.14 (16)
N1^{ii}—Mn1—S1^{iii}	93.46 (4)	C12—C11—H11	120.9
N10^{iii}—Mn1—S1^{iii}	77.55 (3)	C10—C11—H11	120.9
N10—Mn1—S1^{iii}	101.38 (3)	C13—C12—C11	119.78 (15)
N1^i—Mn1—S1	93.46 (4)	C13—C12—H12	120.1
N1^{ii}—Mn1—S1	87.33 (4)	C11—C12—H12	120.1
N10^{iii}—Mn1—S1	101.38 (3)	C12—C13—C14	119.23 (15)
N10—Mn1—S1	77.55 (3)	C12—C13—H13	120.4
S1^{iii}—Mn1—S1	178.69 (2)	C14—C13—H13	120.4
C1—S1—Mn1	106.45 (6)	N10—C14—C13	120.96 (15)
N1—C1—S1	178.83 (15)	N10—C14—C14^{iii}	116.09 (8)
C1—N1—Mn1^{ii}	166.59 (14)	C13—C14—C14^{iii}	122.95 (10)

Symmetry codes: (i) *x*, −*y*+1, *z*−1/2; (ii) −*x*, −*y*+1, −*z*+1; (iii) −*x*, *y*, −*z*+1/2.