metal-organic compounds

trans-Tetrakis(pyridine-κN)bis(thio-cyanato-κN)manganese(II)

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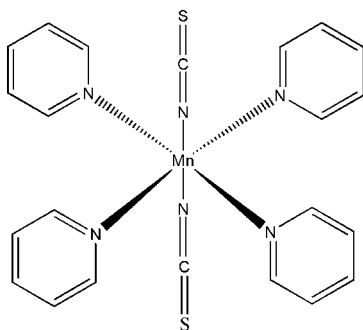Correspondence e-mail: lidacheng@lcu.edu.cn

In the title compound, $[Mn(NCS)_2(C_5H_5N)_4]$, the Mn atom lies on an inversion centre and assumes a distorted octahedral geometry defined by four N atoms from four pyridine molecules and two thiocyanate anions.

Related literature

For the crystal structure of manganese complexes with related ligands, see: Long & Clarke (1978); Deng *et al.* (2006); Cheng *et al.* (2004). For related literature, see: Ma *et al.* (2007).

Experimental

Crystal data

$[Mn(NCS)_2(C_5H_5N)_4]$	$b = 13.1542 (14)$ Å
$M_r = 487.50$	$c = 15.236 (2)$ Å
Monoclinic, $C2/c$	$\beta = 107.413 (2)°$
$a = 12.4982 (12)$ Å	$V = 2390.1 (5)$ Å^3
$Z = 4$	$T = 298 (2)$ K
Mo $K\alpha$ radiation	$0.56 \times 0.42 \times 0.37$ mm
$\mu = 0.75$ mm^{-1}	

Data collection

Siemens SMART CCD area-detector diffractometer	5844 measured reflections
	2107 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1661 reflections with $I > 2\sigma(I)$
$T_{min} = 0.680$, $T_{max} = 0.769$	$R_{int} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$	143 parameters
$wR(F^2) = 0.090$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
2107 reflections	$\Delta\rho_{min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mn1—N1	2.1747 (19)	Mn1—N2	2.3188 (19)
Mn1—N3	2.3057 (19)		
N1—Mn1—N3^i	89.31 (7)	N3—Mn1—N2^i	87.38 (7)
N1—Mn1—N3	90.69 (7)	N1—Mn1—N2	90.41 (7)
N1—Mn1—N2^i	89.59 (7)	N3—Mn1—N2	92.62 (7)

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

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Ma, C. L., Sun, J. S., Zhang, R. F. & Wang, D. Q. (2007). *J. Organomet. Chem.* **692**, 4029–4042.
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supplementary materials

trans-Tetrakis(pyridine-*κN*)bis(thiocyanato-*κN*)manganese(II)

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Comment

A series of complexes of the type MP$_4$X$_2$ (Long & Clarke, 1978; Deng *et al.*, 2006; Cheng *et al.*, 2004)(where *M* is a divalent transition metal, P is a pyridine derivative and *X* is a halide or thiocyanate ion) have been synthesized. We have synthesized the title compound, (I), and characterized it by X-ray diffraction and elemental analysis which is reported in this paper.

In the structure of (I) (Fig. 1), the Mn atom lies on an inversion centre and assumes an octahedral coordination geometry from four N-bonded pyridy molecules and two N-bonded thiocyanate anions. The basal plane consisits of four pyridyl N atoms, with bond lengths in the rang 2.3188 (19)–2.3057 (19) Å. The apical positions are occupied by two thiocyanate N atoms, with equal bond distances. The complex exhibits a one dimensional chain structure *via* short intermolecular contact of the type C—H···S which are consistent with the similar contacts reported earlier (Ma *et al.*, 2007).

The structural features of (I) are very similar to the early results reported for tetrakis(pyridine)metal (Ni, Fe, Co) chloride complexes (Long & Clarke, 1978).

Experimental

A mixtutre of solutions of Mn(OAc)$_2$. 7 H$_2$O (0.0450 g, 0.2 mmol) in MeOH (10 ml) and NaSCN (0.0324 g, 0.4 mmol) in pyridine (10 ml) was stirred for four hours and brown single crystals were obtained after allowing the solution to stand at room temperature for three weeks.

Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H 0.93 Å (pylidyl) [U_{iso}(H) = 1.2U_{eq}(C)].
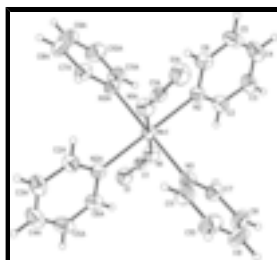
Figures

Fig. 1. A view of the title compound showing the atomic numbering and 30% probability displacement ellipsoids; symmetry code for atoms labelled with A: −*x* + 1/2, −*y* + 1/2, −*z* + 1.

trans-Tetrakis(pyridine-κ*N*)bis(thiocyanato-κ*N*)manganese

Crystal data

[Mn(NCS)$_2$(C$_5$H$_5$N)$_4$]

M_r = 487.50

Monoclinic, $C2/c$

Hall symbol: -C 2yc

a = 12.4982 (12) Å

b = 13.1542 (14) Å

c = 15.236 (2) Å

β = 107.413 (2)°

V = 2390.1 (5) Å^3

Z = 4

F_{000} = 1004

D_x = 1.355 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å

Cell parameters from 2791 reflections

θ = 2.3–26.7°

μ = 0.75 mm^{-1}

T = 298 (2) K

Block, brown

0.56 × 0.42 × 0.37 mm

Data collection

CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 298(2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

T_{min} = 0.680, T_{max} = 0.769

5844 measured reflections

2107 independent reflections

1661 reflections with $I > 2\sigma(I)$

R_{int} = 0.030

θ_{max} = 25.0°

θ_{min} = 2.3°

$h = -14 \rightarrow 14$

$k = -7 \rightarrow 15$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.090$

S = 1.00

2107 reflections

143 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 2.2714P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.28 e Å^{-3}

$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Extinction correction: SHELXL,
Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$

Extinction coefficient: 0.0089 (6)

Special details

Experimental. Elemental analysis: calculated for $C_{22}H_{20}MnN_6S_2$: C 54.20, H 4.14, N 16.42%; found: C 54.21, H 4.13, N 16.40%.

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	0.2500	0.2500	0.5000	0.04435 (19)
N1	0.10819 (16)	0.16743 (16)	0.51970 (15)	0.0601 (6)
N2	0.21099 (15)	0.18519 (15)	0.35223 (13)	0.0498 (5)
N3	0.36836 (16)	0.11699 (15)	0.56271 (13)	0.0524 (5)
S1	−0.07605 (6)	0.14598 (6)	0.58350 (6)	0.0735 (3)
C1	0.03139 (19)	0.15820 (16)	0.54594 (15)	0.0453 (5)
C2	0.1569 (2)	0.0971 (2)	0.32889 (18)	0.0599 (7)
H2	0.1323	0.0633	0.3730	0.072*
C3	0.1355 (2)	0.0536 (2)	0.2437 (2)	0.0711 (8)
H3	0.0991	−0.0088	0.2311	0.085*
C4	0.1684 (2)	0.1034 (3)	0.1779 (2)	0.0797 (9)
H4	0.1541	0.0760	0.1192	0.096*
C5	0.2228 (3)	0.1940 (3)	0.1993 (2)	0.0806 (9)
H5	0.2457	0.2294	0.1553	0.097*
C6	0.2435 (2)	0.2326 (2)	0.28698 (18)	0.0626 (7)
H6	0.2816	0.2940	0.3012	0.075*
C7	0.3356 (2)	0.0206 (2)	0.5493 (2)	0.0664 (7)
H7	0.2613	0.0074	0.5165	0.080*
C8	0.4055 (3)	−0.0603 (2)	0.5813 (2)	0.0847 (10)
H8	0.3795	−0.1266	0.5691	0.102*
C9	0.5135 (3)	−0.0418 (3)	0.6309 (3)	0.0924 (11)
H9	0.5625	−0.0952	0.6541	0.111*
C10	0.5488 (3)	0.0562 (3)	0.6461 (2)	0.0907 (10)
H10	0.6225	0.0709	0.6796	0.109*
C11	0.4744 (2)	0.1328 (2)	0.6113 (2)	0.0701 (8)
H11	0.4994	0.1996	0.6222	0.084*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0405 (3)	0.0446 (3)	0.0497 (3)	−0.0004 (2)	0.0162 (2)	0.0014 (2)

N1	0.0493 (11)	0.0643 (13)	0.0705 (14)	−0.0099 (10)	0.0239 (11)	−0.0002 (11)
N2	0.0461 (10)	0.0537 (12)	0.0503 (11)	0.0016 (9)	0.0154 (9)	0.0013 (10)
N3	0.0487 (11)	0.0514 (12)	0.0582 (12)	0.0034 (9)	0.0178 (10)	0.0047 (10)
S1	0.0721 (5)	0.0691 (5)	0.0963 (6)	−0.0056 (4)	0.0509 (4)	0.0024 (4)
C1	0.0485 (12)	0.0377 (12)	0.0474 (13)	−0.0022 (10)	0.0109 (11)	0.0023 (10)
C2	0.0568 (15)	0.0658 (17)	0.0580 (16)	−0.0058 (13)	0.0188 (12)	−0.0054 (13)
C3	0.0616 (16)	0.082 (2)	0.0677 (18)	−0.0056 (14)	0.0169 (14)	−0.0206 (16)
C4	0.0650 (18)	0.116 (3)	0.0544 (17)	0.0082 (18)	0.0119 (14)	−0.0205 (18)
C5	0.079 (2)	0.115 (3)	0.0547 (17)	0.009 (2)	0.0302 (15)	0.0157 (18)
C6	0.0597 (15)	0.0704 (18)	0.0575 (16)	0.0016 (13)	0.0174 (13)	0.0095 (13)
C7	0.0664 (16)	0.0559 (16)	0.0783 (19)	0.0019 (13)	0.0236 (14)	0.0101 (14)
C8	0.110 (3)	0.0530 (17)	0.103 (2)	0.0173 (17)	0.050 (2)	0.0166 (16)
C9	0.093 (3)	0.089 (3)	0.103 (3)	0.046 (2)	0.041 (2)	0.035 (2)
C10	0.0607 (18)	0.099 (3)	0.103 (3)	0.0258 (18)	0.0096 (17)	0.021 (2)
C11	0.0565 (16)	0.0679 (18)	0.0804 (19)	0.0057 (14)	0.0120 (14)	0.0030 (15)

Geometric parameters (Å, °)

Mn1—N1[i]	2.1747 (19)	C3—H3	0.9300
Mn1—N1	2.1747 (19)	C4—C5	1.362 (5)
Mn1—N3[i]	2.3057 (19)	C4—H4	0.9300
Mn1—N3	2.3057 (19)	C5—C6	1.379 (4)
Mn1—N2[i]	2.3188 (19)	C5—H5	0.9300
Mn1—N2	2.3188 (19)	C6—H6	0.9300
N1—C1	1.151 (3)	C7—C8	1.371 (4)
N2—C2	1.336 (3)	C7—H7	0.9300
N2—C6	1.336 (3)	C8—C9	1.356 (5)
N3—C11	1.327 (3)	C8—H8	0.9300
N3—C7	1.329 (3)	C9—C10	1.360 (5)
S1—C1	1.617 (2)	C9—H9	0.9300
C2—C3	1.369 (4)	C10—C11	1.366 (4)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.360 (4)	C11—H11	0.9300
N1[i]—Mn1—N1	180.00	C4—C3—H3	120.6
N1[i]—Mn1—N3[i]	90.69 (7)	C2—C3—H3	120.6
N1—Mn1—N3[i]	89.31 (7)	C3—C4—C5	118.9 (3)
N1[i]—Mn1—N3	89.31 (7)	C3—C4—H4	120.5
N1—Mn1—N3	90.69 (7)	C5—C4—H4	120.5
N3[i]—Mn1—N3	180.00	C4—C5—C6	119.4 (3)
N1[i]—Mn1—N2[i]	90.41 (7)	C4—C5—H5	120.3
N1—Mn1—N2[i]	89.59 (7)	C6—C5—H5	120.3
N3[i]—Mn1—N2[i]	92.62 (7)	N2—C6—C5	122.5 (3)
N3—Mn1—N2[i]	87.38 (7)	N2—C6—H6	118.7
N1[i]—Mn1—N2	89.59 (7)	C5—C6—H6	118.7
N1—Mn1—N2	90.41 (7)	N3—C7—C8	123.5 (3)
N3[i]—Mn1—N2	87.38 (7)	N3—C7—H7	118.3

N3—Mn1—N2	92.62 (7)	C8—C7—H7	118.3
N2^i—Mn1—N2	180.0	C9—C8—C7	118.7 (3)
C1—N1—Mn1	154.0 (2)	C9—C8—H8	120.6
C2—N2—C6	116.7 (2)	C7—C8—H8	120.6
C2—N2—Mn1	121.34 (16)	C8—C9—C10	118.9 (3)
C6—N2—Mn1	121.90 (17)	C8—C9—H9	120.5
C11—N3—C7	116.5 (2)	C10—C9—H9	120.5
C11—N3—Mn1	121.45 (18)	C9—C10—C11	119.0 (3)
C7—N3—Mn1	122.01 (17)	C9—C10—H10	120.5
N1—C1—S1	179.5 (2)	C11—C10—H10	120.5
N2—C2—C3	123.7 (3)	N3—C11—C10	123.4 (3)
N2—C2—H2	118.2	N3—C11—H11	118.3
C3—C2—H2	118.2	C10—C11—H11	118.3
C4—C3—C2	118.8 (3)		
N3^i—Mn1—N1—C1	−53.0 (4)	N2^i—Mn1—N3—C7	125.3 (2)
N3—Mn1—N1—C1	127.0 (4)	N2—Mn1—N3—C7	−54.7 (2)
N2^i—Mn1—N1—C1	39.6 (4)	C6—N2—C2—C3	1.1 (4)
N2—Mn1—N1—C1	−140.4 (4)	Mn1—N2—C2—C3	−177.5 (2)
N1^i—Mn1—N2—C2	154.24 (18)	N2—C2—C3—C4	−1.6 (4)
N1—Mn1—N2—C2	−25.76 (18)	C2—C3—C4—C5	0.9 (4)
N3^i—Mn1—N2—C2	−115.04 (18)	C3—C4—C5—C6	0.3 (4)
N3—Mn1—N2—C2	64.96 (18)	C2—N2—C6—C5	0.2 (4)
N1^i—Mn1—N2—C6	−24.21 (19)	Mn1—N2—C6—C5	178.7 (2)
N1—Mn1—N2—C6	155.79 (19)	C4—C5—C6—N2	−0.9 (4)
N3^i—Mn1—N2—C6	66.50 (19)	C11—N3—C7—C8	−1.2 (4)
N3—Mn1—N2—C6	−113.50 (19)	Mn1—N3—C7—C8	176.2 (2)
N1^i—Mn1—N3—C11	33.1 (2)	N3—C7—C8—C9	1.4 (5)
N1—Mn1—N3—C11	−146.9 (2)	C7—C8—C9—C10	−0.9 (5)
N2^i—Mn1—N3—C11	−57.3 (2)	C8—C9—C10—C11	0.3 (6)
N2—Mn1—N3—C11	122.7 (2)	C7—N3—C11—C10	0.6 (4)
N1^i—Mn1—N3—C7	−144.2 (2)	Mn1—N3—C11—C10	−176.9 (2)
N1—Mn1—N3—C7	35.8 (2)	C9—C10—C11—N3	−0.1 (5)

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

Fig. 1