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Crystal structure of poly[(2,2′-bipyridine-κ^2N,N')tetra-μ_2-cyanido-$\kappa^4C:N;\kappa^4N:C$-manganese(II)disilver(I)]

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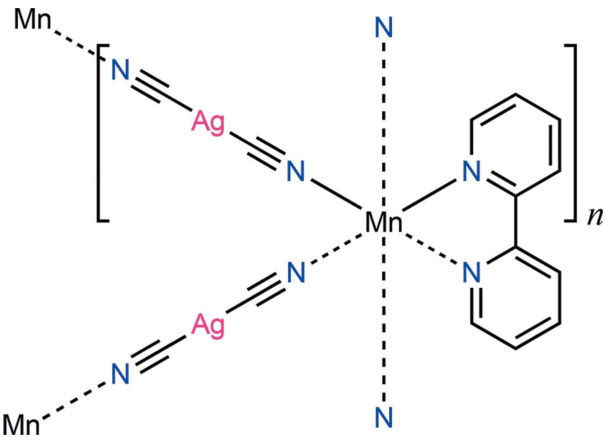
The title compound, $[Ag_2Mn(CN)_4(C_{10}H_8N_2)]_n$ or Mn(bipy){Ag(CN)$_2$}$_2$ (bipy = 2,2′-bipyridine) is isostructural with Cd(bipy){Au(CN)$_2$}$_2$ [Guo *et al.* (2009). *CrystEngComm*, **11**, 61–66]. The Mn^{II} atom has crystallographically imposed twofold symmetry and a distorted octahedral coordination sphere consisting of six N atoms from one bipyridine ligand and four dicyanoargentate(I) anions, $[Ag(CN)_2]^-$, while the Ag^I atom of the complex anion displays the expected linear geometry. Each $[Ag(CN)_2]^-$ unit connects to two neighbouring $[Mn(bipy)]^{2+}$ cations to give an threefold interpenetrating quartz-like three-dimensional framework. No directional interactions beyond van der Waals contacts are observed.

Keywords: crystal structure; dicyanoargentate(I); manganese(II); triple interpenetration.

CCDC reference: 1422937

1. Related literature

For related crystal structures, see: Soma *et al.* (1994); Guo *et al.* (2009). For the use of $[Ag(CN)_2]^-$ as a building block for the construction of cyanide-bridged silver(I)–iron(II) spin-crossover coordination polymers, see: Shorrock *et al.* (2002); Galet *et al.* (2003); Niel *et al.* (2003); Muñoz *et al.* (2007).



2. Experimental

2.1. Crystal data

$[Ag_2Mn(CN)_4(C_{10}H_8N_2)]$
M_r = 530.94
Trigonal, $P3_112$
a = 8.7215 (3) Å
c = 20.9874 (9) Å
V = 1382.52 (13) Å^3
Z = 3
Mo $K\alpha$ radiation
μ = 2.78 mm^{-1}
T = 296 K
0.36 × 0.22 × 0.22 mm

2.2. Data collection

Bruker D8 QUEST CMOS diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker,2014)
T_{min} = 0.484, T_{max} = 0.542
25845 measured reflections
1874 independent reflections
1856 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.015
$wR(F^2)$ = 0.038
S = 1.07
1874 reflections
105 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack x determined using 824 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.037 (6)

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: VN2098).

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supporting information

Acta Cryst. (2015). E**71**, m179–m180 [doi:10.1107/S205698901501676X]

Crystal structure of poly[(2,2′-bipyridine-κ^2N,N')tetra-μ_2-cyanido-$\kappa^4C:N;\kappa^4N:C$-manganese(II)disilver(I)]

Chatphorn Theppitak and Kittipong Chainok

S1. Synthesis and crystallization

$Mn(NO_3)_2{\cdot}6H_2O$ (62 mg, 0.5 mmol) and 2,2′-bipyridine (162 mg, 0.5 mmol) were dissolved in 4 ml of a mixture H_2O/CH_3OH (1:1) to form a bright yellow solution and this was pipetted into one side of the H-tube. $K[Ag(CN)_2]$ (250 mg, 2 mmol) was dissolved in 4 mL of a mixture H_2O/CH_3OH to give a colorless solution and this was pipetted into the other side arm of the H-tube. The H-tube was then carefully filled with a mixture H_2O/CH_3OH. Upon slow diffusion for 3 days, pale-yellow block shaped single crystals of the title compound were formed in the manganese(II)-containing side of the H-tube. Yield: 49 mg, 89% based on manganese source.

S2. Refinement

The C–bound hydrogen atoms were placed in geometrically idealized positions based on chemical coordinations and constrained to ride on their parent atom positions with a C—H distances of 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$ for the aromatic H atoms.

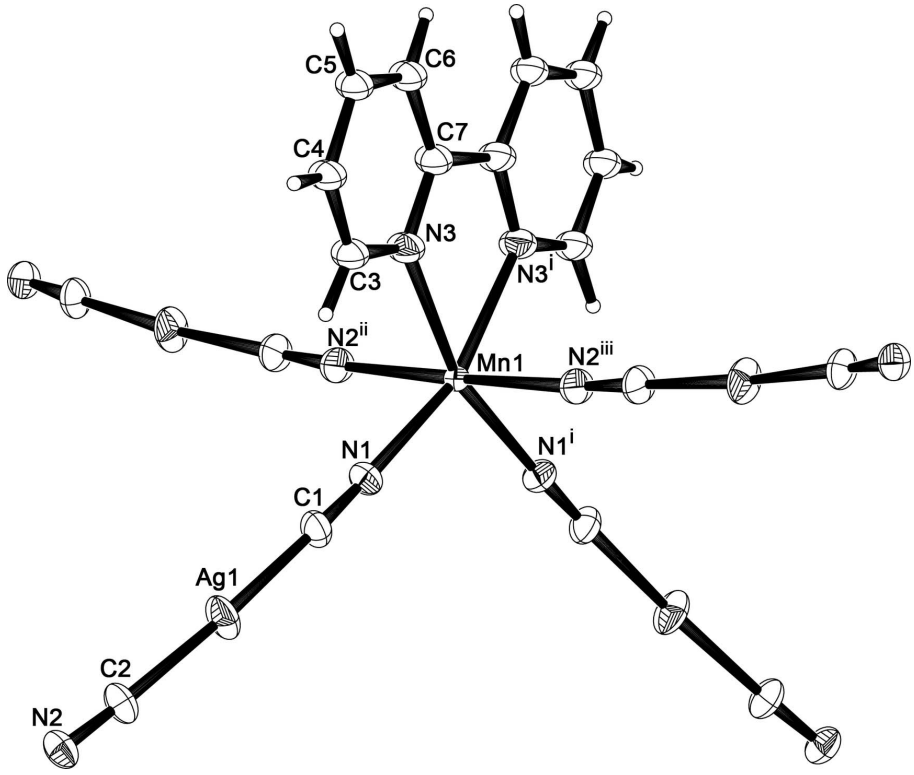

Figure 1

Displacement ellipsoid plot at the 35% probability level of the immediate coordination geometry about the manganese(II) centre in the title compound. The asymmetric unit is labelled. [Symmetry codes: (i) x, -1 + x – y, 2 – z; (ii) 1 – y, x – y, 1/3 + z; (iii) 1 – y, –x, 5/3 – z].

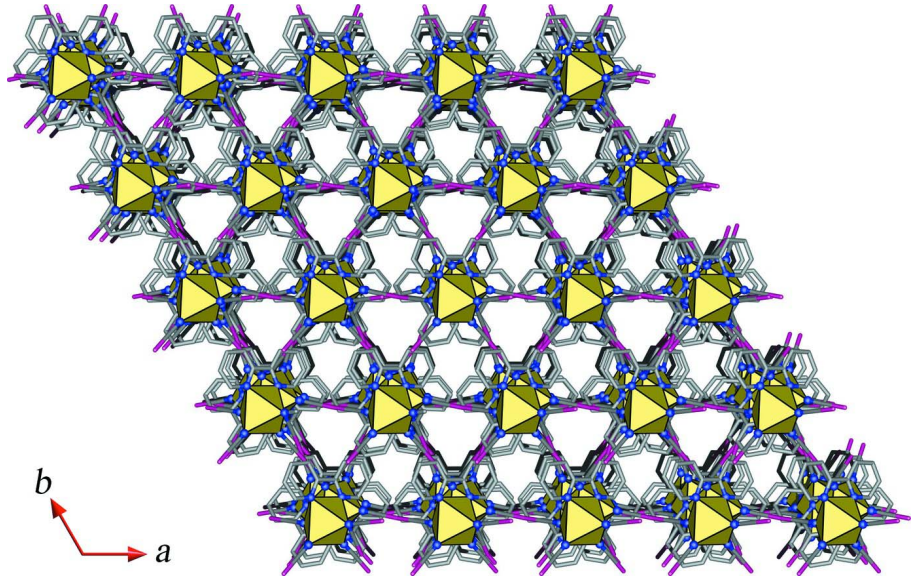

Figure 2

Crystal packing of the title compound viewed along c axis.

Poly[(2,2′-bipyridine-κ^2N,N')tetra-μ_2-cyanido-$\kappa^4C:N;\kappa^4N:C$-manganese(II)disilver(I)]

Crystal data

$[Ag_2Mn(CN)_4(C_{10}H_8N_2)]$
M_r = 530.94
Trigonal, $P3_112$
a = 8.7215 (3) Å
c = 20.9874 (9) Å
V = 1382.52 (13) Å^3
Z = 3
$F(000)$ = 759
D_x = 1.913 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 720 reflections
θ = 3.3–26.3°
μ = 2.78 mm^{-1}
T = 296 K
Block, light yellow
0.36 × 0.22 × 0.22 mm

Data collection

Bruker D8 QUEST CMOS diffractometer
Radiation source: microfocus sealed x-ray tube, Incoatec Iμus
GraphiteDouble Bounce Multilayer Mirror monochromator
Detector resolution: 10.5 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker,2014)
T_{min} = 0.484, T_{max} = 0.542
25845 measured reflections
1874 independent reflections
1856 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.3°, θ_{min} = 3.3°
h = −10→10
k = −10→10
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.015
$wR(F^2)$ = 0.038
S = 1.07
1874 reflections
105 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0231P)^2 + 0.1949P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack x determined using 824 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.037 (6)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Ag1	0.46725 (4)	0.05832 (4)	0.84789 (2)	0.07435 (11)
Mn1	0.89941 (6)	−0.05030 (3)	1.0000	0.03424 (11)
N2	0.2249 (4)	0.0953 (4)	0.74313 (13)	0.0600 (6)
N3	1.1424 (3)	0.1907 (3)	0.96099 (11)	0.0511 (5)
N1	0.7178 (3)	−0.0053 (4)	0.94131 (11)	0.0547 (6)
C7	1.3017 (3)	0.2186 (3)	0.97859 (12)	0.0438 (5)

C4	1.2821 (5)	0.4635 (6)	0.9044 (3)	0.0981 (17)
H4	1.2710	0.5468	0.8803	0.118*
C2	0.3101 (5)	0.0826 (5)	0.78142 (16)	0.0638 (8)
C1	0.6284 (4)	0.0198 (5)	0.90861 (14)	0.0612 (7)
C6	1.4560 (4)	0.3673 (4)	0.95830 (15)	0.0599 (7)
H6	1.5658	0.3828	0.9698	0.072*
C3	1.1344 (5)	0.3099 (6)	0.9239 (2)	0.0848 (13)
H3	1.0238	0.2887	0.9106	0.102*
C5	1.4453 (4)	0.4911 (5)	0.92118 (18)	0.0807 (11)
H5	1.5474	0.5920	0.9077	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0916 (2)	0.1011 (2)	0.05771 (15)	0.06867 (18)	−0.02819 (13)	−0.00888 (13)
Mn1	0.0307 (2)	0.03718 (18)	0.0327 (2)	0.01535 (11)	0.000	−0.00120 (16)
N2	0.0675 (15)	0.0575 (14)	0.0588 (14)	0.0342 (13)	−0.0179 (12)	0.0000 (12)
N3	0.0375 (11)	0.0535 (13)	0.0546 (12)	0.0170 (9)	0.0022 (9)	0.0141 (10)
N1	0.0530 (13)	0.0719 (16)	0.0470 (12)	0.0371 (12)	−0.0084 (10)	−0.0012 (12)
C7	0.0365 (11)	0.0497 (13)	0.0359 (11)	0.0146 (10)	0.0005 (9)	−0.0054 (9)
C4	0.063 (2)	0.080 (3)	0.124 (4)	0.0157 (19)	0.000 (2)	0.056 (3)
C2	0.0752 (19)	0.0685 (19)	0.0578 (17)	0.0435 (17)	−0.0227 (14)	−0.0033 (14)
C1	0.0654 (18)	0.081 (2)	0.0514 (15)	0.0473 (18)	−0.0097 (13)	−0.0030 (14)
C6	0.0363 (13)	0.0621 (17)	0.0594 (16)	0.0082 (12)	−0.0054 (11)	0.0016 (13)
C3	0.0467 (17)	0.082 (2)	0.109 (3)	0.0199 (17)	−0.0021 (18)	0.050 (2)
C5	0.0511 (17)	0.064 (2)	0.088 (2)	−0.0001 (14)	0.0002 (16)	0.0260 (18)

Geometric parameters (Å, º)

Ag1—C2	2.039 (3)	N3—C3	1.328 (4)
Ag1—C1	2.046 (3)	N1—C1	1.139 (4)
Mn1—N2^{i}	2.229 (3)	C7—C7^{iii}	1.485 (5)
Mn1—N2^{ii}	2.229 (3)	C7—C6	1.388 (4)
Mn1—N3^{iii}	2.264 (2)	C4—H4	0.9300
Mn1—N3	2.264 (2)	C4—C3	1.377 (5)
Mn1—N1^{iii}	2.192 (2)	C4—C5	1.365 (6)
Mn1—N1	2.192 (2)	C6—H6	0.9300
N2—Mn1^{iv}	2.229 (3)	C6—C5	1.372 (5)
N2—C2	1.137 (4)	C3—H3	0.9300
N3—C7	1.337 (3)	C5—H5	0.9300
C2—Ag1—C1	174.70 (14)	C3—N3—C7	118.4 (2)
N2^{i}—Mn1—N2^{ii}	177.94 (15)	C1—N1—Mn1	177.0 (3)
N2^{ii}—Mn1—N3	85.78 (10)	N3—C7—C7^{iii}	115.84 (15)
N2^{ii}—Mn1—N3^{iii}	92.55 (10)	N3—C7—C6	121.3 (3)
N2^{i}—Mn1—N3^{iii}	85.78 (10)	C6—C7—C7^{iii}	122.90 (17)
N2^{i}—Mn1—N3	92.55 (10)	C3—C4—H4	120.6
N3^{iii}—Mn1—N3	71.65 (12)	C5—C4—H4	120.6

N1^{iii}—Mn1—N2^{i}	92.34 (11)	C5—C4—C3	118.7 (4)
N1—Mn1—N2^{i}	88.95 (10)	N2—C2—Ag1	178.2 (3)
N1—Mn1—N2^{ii}	92.34 (10)	N1—C1—Ag1	178.1 (3)
N1^{iii}—Mn1—N2^{ii}	88.94 (10)	C7—C6—H6	120.2
N1—Mn1—N3	93.18 (10)	C5—C6—C7	119.6 (3)
N1^{iii}—Mn1—N3	163.66 (9)	C5—C6—H6	120.2
N1^{iii}—Mn1—N3^{iii}	93.18 (10)	N3—C3—C4	123.1 (3)
N1—Mn1—N3^{iii}	163.66 (9)	N3—C3—H3	118.5
N1^{iii}—Mn1—N1	102.49 (15)	C4—C3—H3	118.5
C2—N2—Mn1^{iv}	176.1 (3)	C4—C5—C6	118.9 (3)
C7—N3—Mn1	118.32 (18)	C4—C5—H5	120.6
C3—N3—Mn1	123.2 (2)	C6—C5—H5	120.6
Mn1—N3—C7—C7^{iii}	1.5 (4)	C7—C6—C5—C4	−0.8 (7)
Mn1—N3—C7—C6	−177.6 (2)	C3—N3—C7—C7^{iii}	178.4 (4)
Mn1—N3—C3—C4	174.7 (5)	C3—N3—C7—C6	−0.7 (5)
N3—C7—C6—C5	2.2 (5)	C3—C4—C5—C6	−1.8 (9)
C7—N3—C3—C4	−2.0 (7)	C5—C4—C3—N3	3.3 (10)
C7^{iii}—C7—C6—C5	−176.9 (4)		

Symmetry codes: (i) $-y+1, -x, -z+5/3$; (ii) $-y+1, x-y, z+1/3$; (iii) $x, x-y-1, -z+2$; (iv) $-x+y+1, -x+1, z-1/3$.