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Double interpenetrated structure of poly[tetra-μ_2-cyanido-bis(μ_2-1,2-di-4-pyridylethane)manganese(II)disilver(I)]

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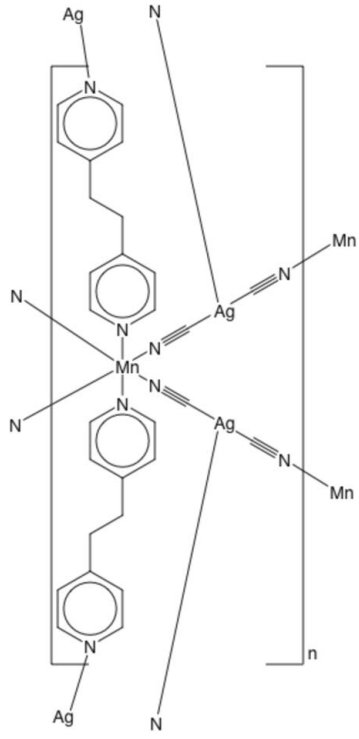
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.026; wR factor = 0.066; data-to-parameter ratio = 20.2.

In the title compound, $[Ag_2Mn(CN)_4(C_{12}H_{12}N_2)_2]$, the Mn atom is located on an inversion center and is linked to adjacent Mn atoms by four μ-$[Ag(CN)_2]^-$ units. Each $[Ag(CN)_2]^-$ unit connects two Mn atoms, defining the edges of a large $\{Mn_4[Ag(CN)_2]_4\}$ mesh. The Mn^{II} atom is further coordinated by two 1,2-di-4-pyridylethane ligands in a *trans* arrangement to attain an MnN_6 octahedral coordination. The Ag^I atoms are three-coordinate, with two C atoms from CN^- and one N atom from a 1,2-di-4-pyridylethane ligand. The doubly interpenetrating three-dimensional framework structure is built by the stacking of $\{Mn[Ag(CN)_2]_2\}$ network sheets and 1,2-di-4-pyridylethane bridges from an Mn atom in one network, penetrating through the $\{Mn_4[Ag(CN)_2]_4\}$ meshes of the adjacent networks, to two Ag atoms in adjacent networks.

Related literature

For related structures, see: Soma *et al.* (1994); Soma & Iwamoto (1997); Niel *et al.* (2002); Dong *et al.* (2003); Maher & Sykora (2007); McElearney *et al.* (1979); Defotis *et al.* (1990); Batten *et al.* (1999).



Experimental

Crystal data

$[Ag_2Mn(CN)_4(C_{12}H_{12}N_2)_2]$
M_r = 743.23
Monoclinic, $P2_1/n$
a = 9.1784 (11) Å
b = 13.2446 (16) Å
c = 11.9536 (14) Å
β = 91.550 (2)°
V = 1452.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.80 mm^{-1}
T = 298 K
0.25 × 0.11 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.663, T_{max} = 0.827
10581 measured reflections
3598 independent reflections
2884 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.066
S = 1.04
3598 reflections
178 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ag1—C1	2.093 (2)	Mn1—N1^{ii}	2.212 (2)
Ag1—C2	2.074 (2)	Mn1—N2	2.2418 (19)
Ag1—N4^{i}	2.468 (2)	Mn1—N3	2.3049 (18)
C1—Ag1—C2	156.57 (9)	N1^{iii}—Mn1—N2	92.02 (8)
C2—Ag1—N4^{i}	106.49 (8)	N1^{iii}—Mn1—N3	90.47 (7)
C1—Ag1—N4^{i}	94.36 (8)	N2—Mn1—N3	90.12 (8)
N1—C1—Ag1	167.4 (2)	C1—N1—Mn1^{iv}	164.15 (19)
N2—C2—Ag1	170.2 (2)	C2—N2—Mn1	158.1 (2)

Symmetry codes: (i) $-x-\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z-\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{5}{2}, z+\frac{1}{2}$; (iv) $-x+\frac{1}{2}, y+\frac{1}{2}, -z-\frac{1}{2}$.

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Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2295).

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supplementary materials

Acta Cryst. (2007). E**63**, m2215-m2216 [**doi:10.1107/S1600536807034897**]

Double interpenetrated structure of poly[tetra-μ_2-cyanido-bis(μ_2-1,2-di-4-pyridylethane)manganese(II)disilver(I)]

T. Kawasaki and T. Kitazawa

Comment

Many of the bimetallic doubly interpenetrated three-dimensional coordination polymers of formula $\{M(L)_2[Ag(CN)_2]_2\}$ [*M* = Cd; *L* = 4,4'-bpy (Soma *et al.*, 1994), *M* = Cd; *L* = dppn (Soma & Iwamoto, 1997), *M* = Fe; *L* = 4,4'-bpy, *M* = Fe; *L* = bpe (Niel *et al.*, 2002), *M* = Mn; *L* = 4,4'-bpy (Dong *et al.*, 2003), *M* = Cu; *L* = 4,4'-bpy (Maher & Sykora, 2007)] [4,4'-bpy = 4,4'-bipyridyl, dppn = 1,3-bis(4-pyridyl)-propane, bpe = *trans*-1,2-bis(4-pyridyl)-ethylene] were reported. In 2002, the spin-crossover behavior was reported in $\{Fe(4,4'\text{-bpy})_2[Ag(CN)_2]_2\}$ at high pressure, and the spin-crossover phenomena with a large hysteresis loop at about 95 K was discovered in $\{Fe(bpe)_2[Ag(CN)_2]_2\}$ (Niel *et al.*, 2002). We report herein the synthesis and crystal structure of a new bimetallic double interpenetrated three-dimensional coordination polymer of formula $\{Mn(edp)_2[Ag(CN)_2]_2\}$ (I) [edp = 1,2-bis(4-pyridyl)-ethane].

In the title compound, (I), Mn1 is located on an inversion center and each Mn^{II} is linked by four μ-$[Ag(CN)_2]^-$. Each $[Ag(CN)_2]^-$ unit connects two manganese atoms defining the edges of a large $\{Mn_4[Ag(CN)_2]_4\}$ mesh. The Mn···Mn distance through the Mn—NC—Ag—CN—Mn edge is 9.958 Å, whereas the Mn···Mn separations through the diagonals of the mesh are 13.245 and 14.873 Å. The puckered meshwork structure extends across [1 0 − 1] plane of the cell to form a 2-D layer of molecular brick wall composed of Mn^{II} and $[Ag(CN)_2]^-$ in a 1:2 ratio. A similar sheet structure was found for $[Mn(SCN)_2(C_2H_5OH)_2]$ (McElearney *et al.*, 1979; Defotis *et al.*, 1990) and $[Mn(dca)_2(C_2H_5OH)_2](CH_3)_2CO$ (Batten *et al.*, 1999).

The Mn^{II} atom in the network coordinates to two edp lignads in a *trans* arrangement to attain the inversion center of a MnN_6 octahedral coordination. The dihedral angle between the pyridine rings of the edp ligand is 82.4 (1)°. Different Mn—Ag meshworks are connected through the edp ligands to form the three-dimensional structure. Ag^I atom is three-coordinate, with two carbon atoms from CN^- and one nitrogen atom from edp ligand, consequently, the C1—Ag1—C2 moiety is bent [C1—Ag1—C2 = 156.57 (9)°]. The crystal structure of (I) is the 3-D interpenetrating double framework formed from two three-dimensional molecules interpenetrating each other, similar to that of $\{M(4,4'\text{-bpy})_2[Ag(CN)_2]_2\}$ [*M* = Cd (Soma *et al.*, 1994), *M* = Fe (Niel *et al.*, 2002), *M* = Mn (Dong *et al.*, 2003), *M* = Cu (Maher & Sykora, 2007)] and $\{Fe(bpe)_2[Ag(CN)_2]_2\}$ (Niel *et al.*, 2002).

Experimental

$MnCl_2{\cdot}4H_2O$ (0.5 mmol) and edp (0.5 mmol) were dissolved in water (15 ml), then obtained red precipitation were filtered off. The filtrate and 15 ml aqueous solution containing $K[Ag(CN)_2]$ (1.0 mmol) were slowly mixed in a H-shaped tube. A few day later, colorless crystals of $\{Mn(edp)_2[Ag(CN)_2]_2\}$ were obtained. Elemental analysis calculated for $C_{28}H_{24}Ag_2MnN_8$: C 45.24%, H 3.25%, N 15.08%; found, C 45.10%, H 3.43%, N 14.85%. IR spectrum data (nujol technique): ν_{CN} 2142 cm^{-1}.

Refinement

H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for aromatic and methylene H atoms, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

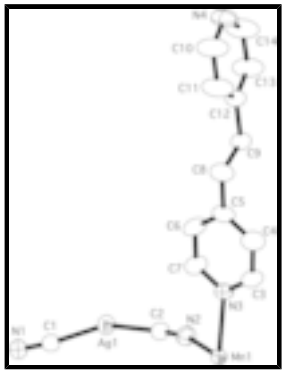

Fig. 1. The asymmetric unit of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are omitted for clarity.

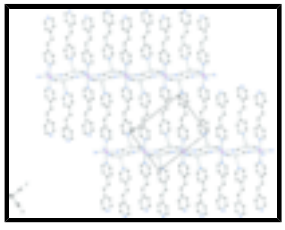

Fig. 2. One of the three-dimensional frameworks observed in (I); viewed along *b* axis; the unit cell is shown as dashed lines. Mn: pink balls, Ag: white balls, C: black balls, N: bule balls.

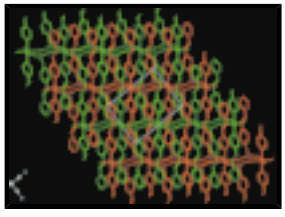

Fig. 3. View of the interpenetrating double three-dimensional framework structure of (I).

poly[tetra-μ$_2$-cyanido-bis(μ$_2$-1,2-di-4- pyridylethane)manganese(II)disilver(I)]

Crystal data

$[Ag_2Mn(CN)_4(C_{12}H_{12}N_2)_2]$	F_{000} = 734
M_r = 743.23	D_x = 1.699 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 4339 reflections
a = 9.1784 (11) Å	θ = 2.3–28.2º
b = 13.2446 (16) Å	μ = 1.80 mm^{-1}
c = 11.9536 (14) Å	T = 298 K
β = 91.550 (2)º	Block, colorless
V = 1452.6 (3) Å^3	0.25 × 0.11 × 0.11 mm

$Z = 2$

Data collection

Bruker SMART CCD area-detector diffractometer	3598 independent reflections
Radiation source: fine-focus sealed tube	2884 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
Detector resolution: 8.366 pixels mm^{-1}	$\theta_{max} = 28.3°$
$T = 298$ K	$\theta_{min} = 2.3°$
φ and ω scans	$h = -11 \rightarrow 12$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -17 \rightarrow 17$
$T_{min} = 0.663$, $T_{max} = 0.827$	$l = -15 \rightarrow 7$
10581 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.026$	H-atom parameters constrained
$wR(F^2) = 0.066$	$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 0.3909P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.003$
3598 reflections	$\Delta\rho_{max} = 0.58$ e $Å^{-3}$
178 parameters	$\Delta\rho_{min} = -0.46$ e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.0000	0.03002 (11)
Ag1	0.241172 (19)	1.336662 (14)	−0.134524 (15)	0.04139 (7)
N1	0.1379 (2)	1.46406 (16)	−0.35068 (17)	0.0432 (5)

N2	0.4096 (2)	1.15716 (14)	−0.00741 (19)	0.0436 (5)
N3	0.3127 (2)	0.95122 (16)	0.11267 (16)	0.0408 (4)
N4	−0.4954 (2)	0.84446 (18)	0.5562 (2)	0.0578 (6)
C1	0.1871 (2)	1.42740 (18)	−0.2729 (2)	0.0392 (5)
C2	0.3545 (2)	1.22646 (18)	−0.0448 (2)	0.0393 (5)
C3	0.3207 (3)	0.8754 (2)	0.1835 (3)	0.0573 (7)
H3	0.4106	0.8441	0.1948	0.069*
C4	0.2044 (3)	0.8393 (2)	0.2424 (3)	0.0614 (8)
H4	0.2169	0.7844	0.2902	0.074*
C5	0.0705 (3)	0.8843 (2)	0.2303 (2)	0.0455 (6)
C6	0.0629 (3)	0.9656 (2)	0.1596 (3)	0.0628 (8)
H6	−0.0246	1.0003	0.1497	0.075*
C7	0.1837 (3)	0.9963 (2)	0.1029 (3)	0.0598 (8)
H7	0.1745	1.0516	0.0553	0.072*
C8	−0.0620 (3)	0.8473 (2)	0.2895 (2)	0.0510 (7)
H8A	−0.0505	0.7760	0.3059	0.061*
H8B	−0.1469	0.8550	0.2401	0.061*
C9	−0.0876 (2)	0.90353 (18)	0.3971 (2)	0.0415 (5)
H9A	−0.0852	0.9754	0.3817	0.050*
H9B	−0.0078	0.8887	0.4494	0.050*
C10	−0.4551 (3)	0.7921 (3)	0.4693 (3)	0.0711 (10)
H10	−0.5174	0.7420	0.4420	0.085*
C11	−0.3253 (3)	0.8073 (3)	0.4160 (3)	0.0696 (10)
H11	−0.3032	0.7680	0.3543	0.084*
C12	−0.2292 (2)	0.87926 (19)	0.4528 (2)	0.0414 (5)
C13	−0.2709 (3)	0.9332 (3)	0.5439 (3)	0.0755 (11)
H13	−0.2104	0.9837	0.5730	0.091*
C14	−0.4017 (4)	0.9134 (3)	0.5928 (3)	0.0834 (12)
H14	−0.4257	0.9508	0.6556	0.100*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0278 (2)	0.0349 (2)	0.0275 (2)	0.00197 (17)	0.00304 (17)	0.00079 (18)
Ag1	0.04233 (11)	0.04225 (11)	0.03938 (11)	0.00550 (7)	−0.00247 (7)	0.00415 (8)
N1	0.0406 (10)	0.0512 (12)	0.0375 (11)	0.0005 (9)	−0.0021 (9)	0.0046 (9)
N2	0.0429 (11)	0.0405 (11)	0.0475 (12)	0.0059 (9)	0.0028 (9)	0.0021 (9)
N3	0.0334 (10)	0.0516 (12)	0.0380 (11)	−0.0005 (8)	0.0091 (8)	0.0052 (9)
N4	0.0445 (12)	0.0736 (16)	0.0563 (14)	−0.0162 (11)	0.0199 (11)	−0.0152 (12)
C1	0.0339 (11)	0.0454 (13)	0.0383 (13)	−0.0020 (9)	0.0035 (9)	0.0014 (10)
C2	0.0373 (12)	0.0409 (13)	0.0399 (13)	0.0033 (10)	0.0033 (9)	−0.0004 (10)
C3	0.0382 (13)	0.0735 (18)	0.0610 (18)	0.0115 (13)	0.0173 (12)	0.0196 (15)
C4	0.0537 (16)	0.0674 (19)	0.0641 (19)	0.0053 (13)	0.0227 (14)	0.0251 (15)
C5	0.0399 (13)	0.0551 (15)	0.0421 (14)	−0.0094 (11)	0.0136 (10)	−0.0062 (12)
C6	0.0351 (13)	0.077 (2)	0.077 (2)	0.0072 (13)	0.0167 (13)	0.0183 (17)
C7	0.0413 (14)	0.0680 (18)	0.071 (2)	0.0084 (12)	0.0161 (13)	0.0277 (16)
C8	0.0436 (14)	0.0595 (16)	0.0506 (15)	−0.0147 (11)	0.0152 (12)	−0.0066 (12)
C9	0.0343 (12)	0.0445 (13)	0.0461 (14)	−0.0037 (9)	0.0069 (10)	0.0000 (11)

C10	0.0570 (17)	0.077 (2)	0.080 (2)	−0.0310 (16)	0.0327 (16)	−0.0325 (18)
C11	0.0609 (18)	0.075 (2)	0.075 (2)	−0.0235 (15)	0.0359 (16)	−0.0332 (17)
C12	0.0339 (11)	0.0459 (13)	0.0447 (14)	−0.0035 (10)	0.0080 (10)	0.0000 (11)
C13	0.0533 (17)	0.102 (3)	0.073 (2)	−0.0369 (17)	0.0248 (15)	−0.0392 (19)
C14	0.0620 (19)	0.114 (3)	0.076 (2)	−0.0366 (19)	0.0344 (17)	−0.049 (2)

Geometric parameters (Å, °)

Ag1—C1	2.093 (2)	C9—H9A	0.9700
Ag1—C2	2.074 (2)	C9—H9B	0.9700
Ag1—N4^{i}	2.468 (2)	C10—N4	1.310 (4)
C1—N1	1.132 (3)	C10—C11	1.380 (4)
C2—N2	1.134 (3)	C10—H10	0.9300
C3—N3	1.314 (3)	C11—C12	1.364 (4)
C3—C4	1.380 (4)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.366 (4)
C4—C5	1.370 (4)	C13—C14	1.375 (4)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.370 (4)	C14—N4	1.321 (4)
C5—C8	1.505 (3)	C14—H14	0.9300
C6—C7	1.376 (4)	Mn1—N1^{ii}	2.212 (2)
C6—H6	0.9300	Mn1—N1^{iii}	2.212 (2)
C7—N3	1.329 (3)	Mn1—N2^{iv}	2.2417 (19)
C7—H7	0.9300	Mn1—N2	2.2418 (19)
C8—C9	1.510 (3)	Mn1—N3	2.3049 (18)
C8—H8A	0.9700	Mn1—N3^{iv}	2.3049 (18)
C8—H8B	0.9700	N1—Mn1^{v}	2.212 (2)
C9—C12	1.511 (3)	N4—Ag1^{vi}	2.468 (2)
C1—Ag1—C2	156.57 (9)	C12—C11—C10	120.7 (3)
C2—Ag1—N4^{i}	106.49 (8)	C12—C11—H11	119.6
C1—Ag1—N4^{i}	94.36 (8)	C10—C11—H11	119.6
N1—C1—Ag1	167.4 (2)	C11—C12—C13	115.5 (2)
N2—C2—Ag1	170.2 (2)	C11—C12—C9	124.3 (2)
N3—C3—C4	124.3 (3)	C13—C12—C9	120.2 (2)
N3—C3—H3	117.9	C12—C13—C14	120.4 (3)
C4—C3—H3	117.9	C12—C13—H13	119.8
C5—C4—C3	120.0 (3)	C14—C13—H13	119.8
C5—C4—H4	120.0	N4—C14—C13	124.0 (3)
C3—C4—H4	120.0	N4—C14—H14	118.0
C4—C5—C6	116.0 (2)	C13—C14—H14	118.0
C4—C5—C8	122.9 (3)	N1^{ii}—Mn1—N1^{iii}	180.0
C6—C5—C8	121.1 (2)	N1^{ii}—Mn1—N2^{iv}	87.98 (8)
C5—C6—C7	120.5 (3)	N1^{iii}—Mn1—N2^{iv}	92.02 (8)
C5—C6—H6	119.8	N1^{ii}—Mn1—N2	92.02 (8)
C7—C6—H6	119.8	N1^{iii}—Mn1—N2	87.98 (8)
N3—C7—C6	123.5 (3)	N2^{iv}—Mn1—N2	180.0

N3—C7—H7	118.3	$N1^{ii}$—Mn1—N3	90.47 (7)
C6—C7—H7	118.3	$N1^{iii}$—Mn1—N3	89.53 (7)
C5—C8—C9	112.8 (2)	$N2^{iv}$—Mn1—N3	89.88 (8)
C5—C8—H8A	109.0	N2—Mn1—N3	90.12 (8)
C9—C8—H8A	109.0	$N1^{ii}$—Mn1—$N3^{iv}$	89.53 (7)
C5—C8—H8B	109.0	$N1^{iii}$—Mn1—$N3^{iv}$	90.47 (7)
C9—C8—H8B	109.0	$N2^{iv}$—Mn1—$N3^{iv}$	90.12 (8)
H8A—C8—H8B	107.8	N2—Mn1—$N3^{iv}$	89.88 (8)
C8—C9—C12	115.2 (2)	N3—Mn1—$N3^{iv}$	180.00 (10)
C8—C9—H9A	108.5	C1—N1—$Mn1^{v}$	164.15 (19)
C12—C9—H9A	108.5	C2—N2—Mn1	158.1 (2)
C8—C9—H9B	108.5	C3—N3—C7	115.7 (2)
C12—C9—H9B	108.5	C3—N3—Mn1	124.14 (16)
H9A—C9—H9B	107.5	C7—N3—Mn1	119.99 (17)
N4—C10—C11	123.7 (3)	C10—N4—C14	115.7 (2)
N4—C10—H10	118.2	C10—N4—$Ag1^{vi}$	124.01 (19)
C11—C10—H10	118.2	C14—N4—$Ag1^{vi}$	119.78 (19)

Symmetry codes: (i) −*x*−1/2, *y*+1/2, −*z*+1/2; (ii) *x*+1/2, −*y*+5/2, *z*+1/2; (iii) −*x*+1/2, *y*−1/2, −*z*−1/2; (iv) −*x*+1, −*y*+2, −*z*; (v) −*x*+1/2, *y*+1/2, −*z*−1/2; (vi) −*x*−1/2, *y*−1/2, −*z*+1/2.

Fig. 1

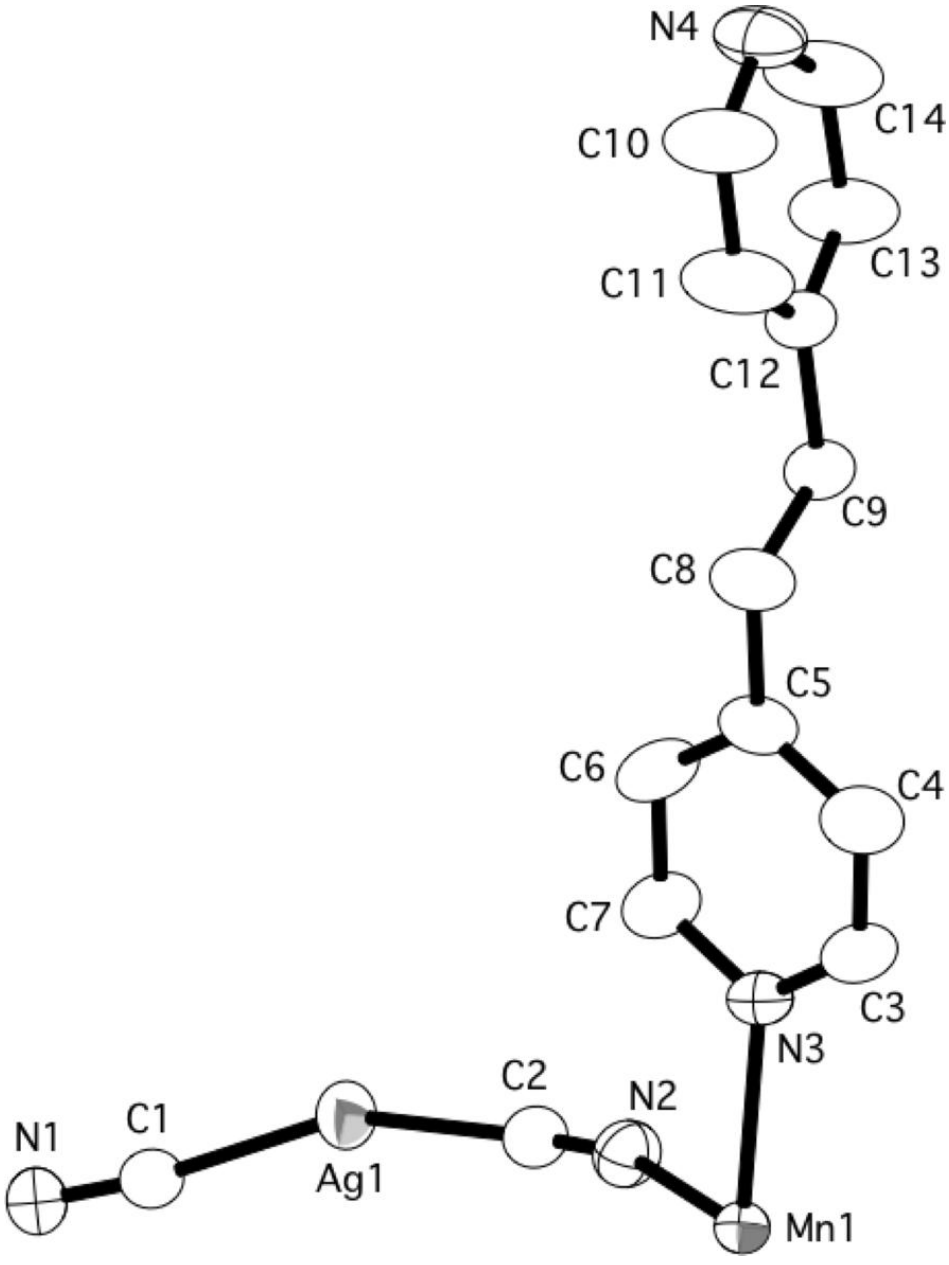

Fig. 2

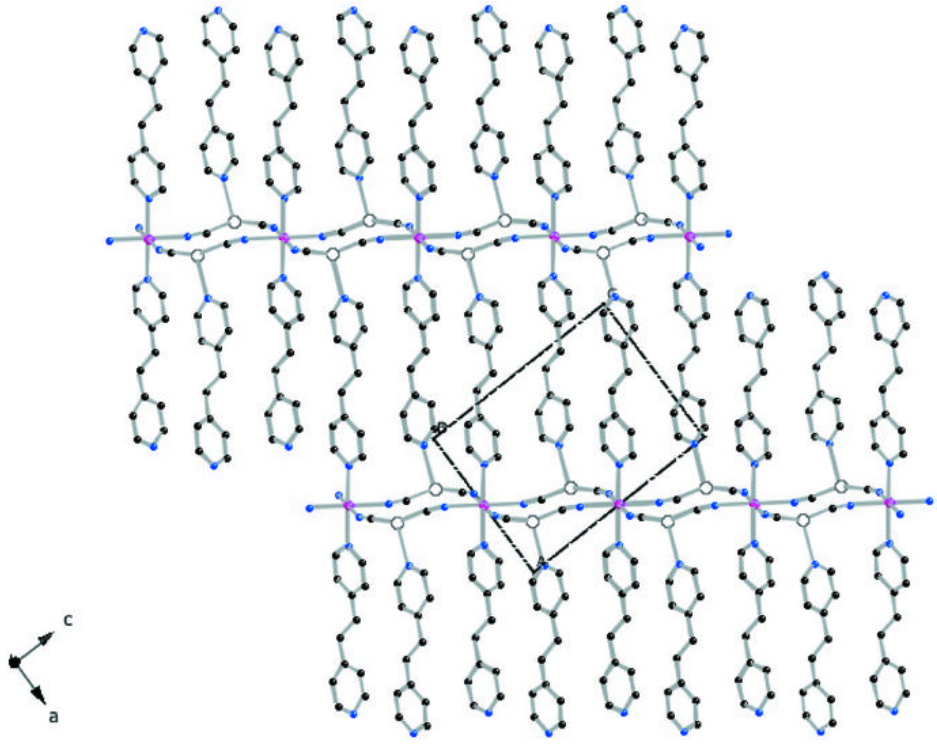

Fig. 3