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Redetermination of *catena*-Poly[[(4-methylpyridine-κN)silver(I)]-μ_3-bromo]

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Key indicators

Single-crystal X-ray study
T = 170 K
Mean σ(C–C) = 0.006 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 21.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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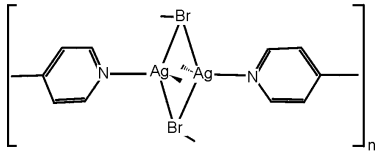
In the crystal structure of the title compound, $[AgBr(C_6H_7N)]_n$, the Ag and Br atoms form Ag–Br double chains in which each Ag atom is coordinated by three Br atoms and the N atom of a 4-methylpyridine ligand in a distorted tetrahedral geometry. The asymmetric unit contains two Ag atoms, two Br atoms, and two 4-methylpyridine ligands.

Comment

The structure determination of the title compound, (I), was undertaken as part of a project on the synthesis, structure and reactivity of coordination polymers based on silver(I) halides and nitrogen-donor ligands. The structure of (I) had been determined previously by Healy *et al.* (1985) at room temperature in space group $P2_1/n$ [a = 10.286 (5), b = 18.066 (9), c = 4.390 (3) Å, and β = 104.31 (5)°]. Healy *et al.* (1985) pointed out that the 4-methylpyridine ligand is disordered and they assumed that the ligand oscillated about the N—C—C axis. We have performed a low-temperature determination at 150 K and found a unit cell in which the c axis is doubled. Refinement has been carried out in space group $P2_1/n$ [a = 9.9601 (7) Å, b = 17.8849 (10) Å and c = 8.8523 (6) Å, and β = 99.550 (8)°].



(I)

In contrast to the room-temperature determination, at 150 K, the asymmetric unit consists of two crystallographically independent Ag and two Br atoms, as well as two crystallographically independent 4-methylpyridine ligands (Fig. 1). Each Ag atom is coordinated by three Br atoms and the N atom of a 4-methylpyridine ligand in a distorted tetrahedral

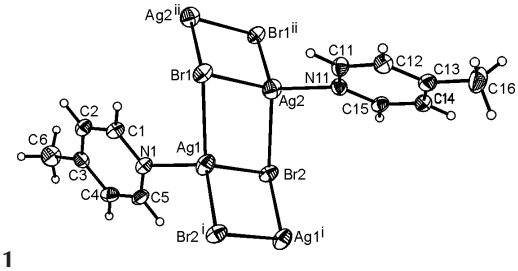


Figure 1
Part of the crystal structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level [symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$].

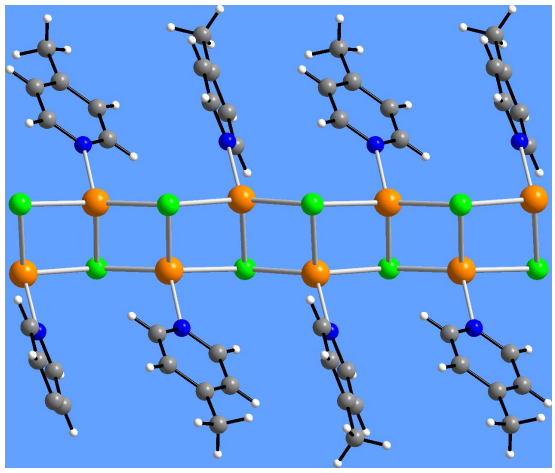


Figure 2
The crystal structure of the title compound, viewed along the *b* axis, showing the Ag–Br double chains.

geometry. The Ag and Br atoms are connected into Ag–Br double chains which are elongated in the direction of the *a* axis (Figs. 2 and 3). In contrast to the structure determined by Healy *et al.*, we found no disorder of the ligand. In addition, we have refined the structure using low-temperature data in the small unit cell with halved *c* axis. In this case, the asymmetric unit consists of one crystallographically independent Ag and Br atom and one independent ligand which is disordered. The structure can be refined successfully using a split model and assuming a disorder in which the phenyl ring exhibits two different orientations about the N—C—C axis. Because of the different temperatures of the previous and the present investigation, we cannot exclude that, for example, a low-temperature phase transition occurred. If our crystals are investigated at room temperature they immediately decompose completely. This was not the case for the previous determination because the crystals were sealed together with mother liquor into a capillary tube.

Experimental

AgBr (239.12 mg, 1.28 mmol) was reacted with 4-methylpyridine (2.0 ml, 20.0 mmol) in a glass container at room temperature in the dark. After 7 d, colourless crystals suitable for X-ray structure analysis were obtained. A large amount of a crystalline powder was obtained by the reaction of AgBr (240.70 mg, 1.28 mmol) in 4-methylpyridine (2.0 ml, 20.0 mmol) at room temperature with stirring for 3 d. The product was washed with diethyl ether and filtered off [yield (based on AgBr) 53.3%]. Elemental analysis calculated: C 25.7, H 2.5, N 5.0%; found: C 26.1, H 2.6, N 5.1%.

Crystal data

$[AgBr(C_6H_7N)]$	D_x = 2.400 Mg m^{-3}
M_r = 280.91	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 7718 reflections
a = 8.8523 (6) Å	θ = 2.5–28°
b = 17.8849 (10) Å	μ = 7.64 mm^{-1}
c = 9.9601 (7) Å	T = 170 (2) K
β = 99.550 (8)°	Block, colourless
V = 1555.05 (17) Å^3	0.11 × 0.09 × 0.09 mm
Z = 8	

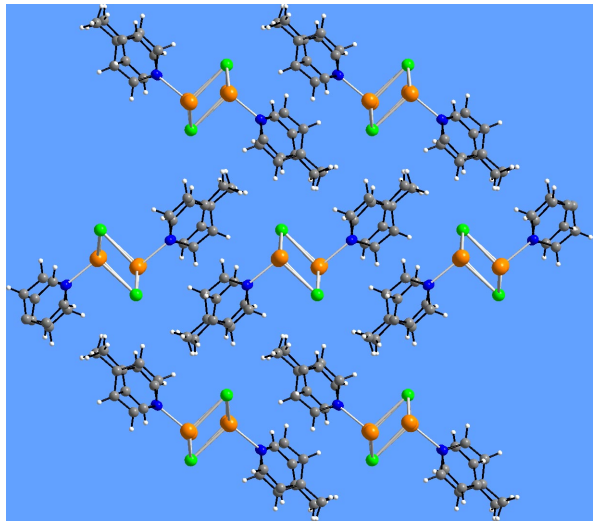


Figure 3
The crystal structure of the title compound, viewed along the the Ag–Br chains in the direction of the *a* axis.

Data collection

Stoe IPDS diffractometer	2208 reflections with $I > 2\sigma(I)$
φ scans	R_{int} = 0.046
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998)	θ_{max} = 28.0°
T_{min} = 0.444, T_{max} = 0.502	$h = -11 \rightarrow 11$
13360 measured reflections	$k = -23 \rightarrow 23$
3523 independent reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.078	$\Delta\rho_{max}$ = 0.61 e Å^{-3}
S = 0.93	$\Delta\rho_{min}$ = −0.69 e Å^{-3}
3523 reflections	Extinction correction: *SHELXL*97
166 parameters	Extinction coefficient: 0.00093 (17)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ag1—N1	2.307 (4)	Ag2—N11	2.302 (4)
Ag1—Br2^{i}	2.6460 (7)	Ag2—Br1^{ii}	2.6654 (7)
Ag1—Br1	2.7340 (7)	Ag2—Br2	2.7245 (7)
Ag1—Br2	2.8397 (6)	Ag2—Br1	2.8620 (6)
Ag1—Ag1^{i}	3.0792 (8)	Ag2—Ag2^{ii}	3.1982 (9)
Ag1—Ag2	3.1078 (6)		
N1—Ag1—Br2^{i}	120.68 (10)	N11—Ag2—Br1	98.36 (9)
N1—Ag1—Br1	102.63 (11)	Br1^{ii}—Ag2—Br1	109.397 (19)
Br2^{i}—Ag1—Br1	113.166 (18)	Br2—Ag2—Br1	112.05 (2)
N1—Ag1—Br2	94.61 (9)	N11—Ag2—Ag1	107.76 (10)
Br2^{i}—Ag1—Br2	111.811 (19)	Br1^{ii}—Ag2—Ag1	127.949 (17)
Br1—Ag1—Br2	112.45 (2)	Br2—Ag2—Ag1	57.825 (14)
N1—Ag1—Ag1^{i}	120.83 (10)	Br1—Ag2—Ag1	54.323 (15)
Br2^{i}—Ag1—Ag1^{i}	58.891 (18)	N11—Ag2—Ag2^{ii}	126.68 (10)
Br1—Ag1—Ag1^{i}	133.65 (2)	Br1^{ii}—Ag2—Ag2^{ii}	57.575 (17)
Br2—Ag1—Ag1^{i}	52.920 (16)	Br2—Ag2—Ag2^{ii}	126.43 (2)
N1—Ag1—Ag2	108.33 (10)	Br1—Ag2—Ag2^{ii}	51.822 (16)
Br2^{i}—Ag1—Ag2	130.481 (17)	Ag1—Ag2—Ag2^{ii}	89.461 (18)
Br1—Ag1—Ag2	58.250 (15)	Ag2^{ii}—Br1—Ag1	110.57 (2)
Br2—Ag1—Ag2	54.302 (15)	Ag2^{ii}—Br1—Ag2	70.603 (19)
Ag1^{i}—Ag1—Ag2	91.131 (18)	Ag1—Br1—Ag2	67.427 (16)
N11—Ag2—Br1^{ii}	124.10 (10)	Ag1^{i}—Br2—Ag2	110.69 (2)
N11—Ag2—Br2	104.56 (10)	Ag1^{i}—Br2—Ag1	68.189 (19)
Br1^{ii}—Ag2—Br2	108.030 (18)	Ag2—Br2—Ag1	67.873 (17)

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$.

The aromatic H atoms were positioned with idealized geometry (C—H) = 0.93 Å) and refined with fixed isotropic displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)] using the riding model. The positions of the methyl H atoms were idealized (C—H = 0.98 Å), then refined with fixed isotropic displacement parameters [U_{iso}(H) = 1.5U_{eq}(C)] as rigid groups allowed to rotate but not tip.

Data collection: *IPDS* Program Package (Stoe & Cie, 1998); cell refinement: *IPDS* Program Package; data reduction: *IPDS* Program Package; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *XCIF* in *SHELXTL*.

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