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Chlorotris(3-methylpyridine-κ*N*)silver(I)

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Key indicators

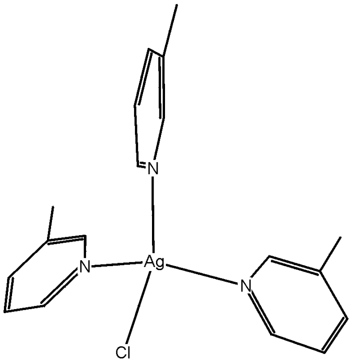
Single-crystal X-ray study
T = 170 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.029
wR factor = 0.068
Data-to-parameter ratio = 16.1

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

In the crystal structure of the title compound, $[AgCl(C_6H_7N)_3]$, each Ag atom is surrounded by one Cl atom and three N atoms of 4-methylpyridine ligands within a distorted tetrahedron. The Ag and Cl atom are located on special positions of site symmetry $3m$, whereas all atoms of the 3-methylpyridine ligand, except for the three disordered methyl H atoms, are located on a mirror plane.

Comment

The structure determination of the title compound was undertaken as a part of a project on the synthesis, structure and thermal reactivity of silver halide coordination polymers with nitrogen-donor ligands (Näther & Beck, 2004*a*,*b*). With 3-methylpyridine and silver halides only two compounds are known. These are *catena*[(μ_3-bromo)(3-methylpyridine-*N*)silver(I)] (Healy *et al.*, 1985) and *catena*[(μ_3-bromo)(3-methylpyridine-*N*)silver(I)] (Healy *et al.*, 1983).



(I)

In the crystal structure of the title compound, (I), the Ag atom is coordinated by one Cl atom and three symmetry-related 3-methylpyridine ligands within a distorted tetrahedron. The asymmetric unit contain 1/6 Ag and 1/6 Cl atom on special positions of site-symmetry $3m$, as well as half a 3-methylpyridine ligand which is located on a mirror plane. The C—N and Ag—Cl bond lengths are in the ranges of those observed in related structures retrieved from the Cambridge Structural Database (Allen, 2002; *ConQuest* Version 1.6 of 2003). In the crystal structure, the complexes are closely packed such that each 3-methylpyridine ligand of one complex points into an aperture of a neighbouring complex.

Experimental

AgCl (288.5 mg, 2.00 mmol) was reacted with 3-methylpyridine (2.0 ml, 20.0 mmol) in a glass container. After 7 d, colourless crystals suitable for X-ray structure analysis had grown. Pure crystalline

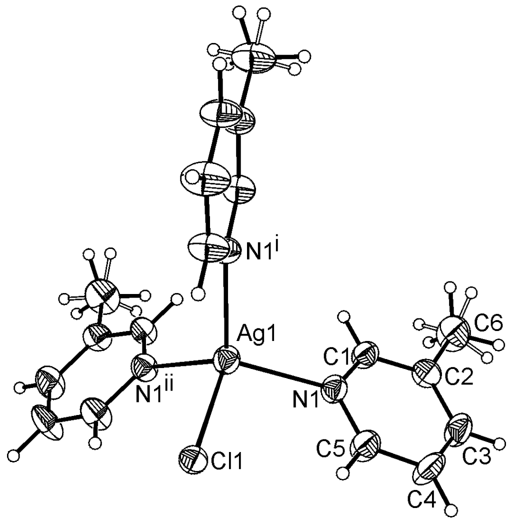


Figure 1
The molecular structure of the title compound, with labelling and displacement ellipsoids drawn at the 50% probability level. The disordered H atoms are displayed with solid and open bonds. [Symmetry codes: (i) $1-y, x-y-1, z$; (ii) $2-x+y, 1-x, z$.]

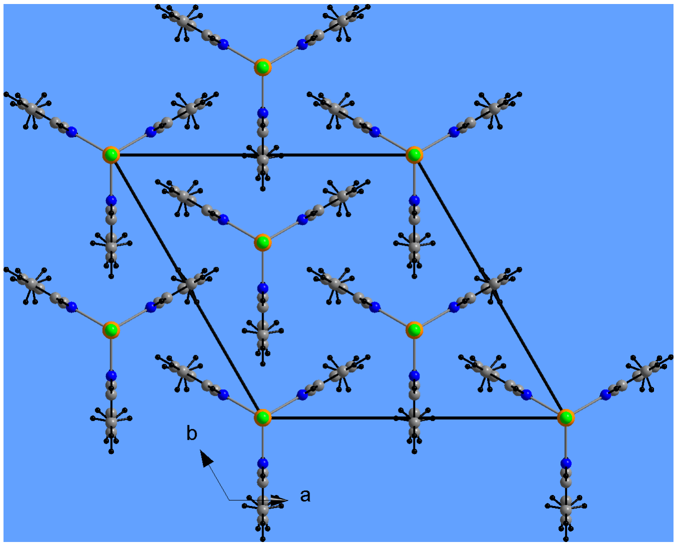


Figure 2
The crystal structure of the title compound viewed along the *c* axis. H atoms are disordered due to symmetry.

powder could be obtained if AgCl (289.4 mg, 2.01 mmol) was reacted with it under stirring in 3-methylpyridine (2.0 ml, 20.0 mmol) in a glass container for 3 d. The product was filtered off and washed with diethyl ether (yield: 212.03 mg, 44.4% based on AgCl). The homogeneity was checked by X-ray powder diffraction. All steps must be carried out in the dark.

Crystal data

$[AgCl(C_6H_7N)_3]$
$M_r = 422.70$
Trigonal, *R*3*m*
$a = 14.6795$ (10) Å
$c = 7.5114$ (4) Å
$V = 1401.76$ (15) Å^3
$Z = 3$
$D_x = 1.502$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 4495 reflections
$\theta = 3–28°$
$\mu = 1.22$ mm^{-1}
$T = 170$ (2) K
Block, colourless
0.10 × 0.09 × 0.09 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998)
$T_{min} = 0.879$, $T_{max} = 0.888$
3908 measured reflections
823 independent reflections

811 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 28.0°$
$h = -19 \rightarrow 19$
$k = -19 \rightarrow 17$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.068$
$S = 1.09$
823 reflections
51 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 1.7193P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0105 (11)
Absolute structure: Flack (1983), 397 Friedel reflections
Flack parameter = 0.01 (6)

Table 1
Selected geometric parameters (Å, °).

Ag1—N1	2.306 (4)	C1—C2	1.409 (7)
Ag1—Cl1	2.6066 (18)	C2—C3	1.373 (9)
N1—C5	1.327 (6)	C2—C6	1.499 (9)
N1—C1	1.327 (6)	C3—C4	1.389 (9)
N1—Ag1—N1^{i}	112.88 (8)	C3—C2—C1	117.1 (5)
N1—Ag1—Cl1	105.80 (10)	C3—C2—C6	123.3 (5)
C5—N1—C1	117.8 (4)	C1—C2—C6	119.6 (6)
C5—N1—Ag1	116.9 (3)	C2—C3—C4	119.6 (5)
C1—N1—Ag1	125.3 (3)	C3—C4—C5	118.5 (5)
N1—C1—C2	124.1 (5)	N1—C5—C4	122.8 (5)

Symmetry code: (i) $1-y, x-y-1, z$.

The aromatic H atoms were positioned with idealized geometry (C—H = 0.95 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. The absolute polarity was determined and is in agreement with the selected setting. In addition, refinement of the inverse structure leads to significantly poorer reliability factors [$R1$ for 811 $F_o > 4\sigma(F_o) = 0.0340$ and $wR2$ for all data = 0.0826]. Due to symmetry, the methyl H atoms are disordered over two orientations. In a space group of lower symmetry, *e.g.* *R*3, these H atoms do not necessarily have to be disordered. However, since all the other atoms could be successfully refined in space group *R*3*m*, refinement was performed in this space group.

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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